EXPERIMENTAL DETERMINATION OF TRANSMISSION PROBABILITIES FOR MOLECULAR FLOW THROUGH ORIFICES

By

ALVIN LEROY BALL, JR.

Bachelor of Science

Northwestern State College

Alva, Oklahoma

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Thesis Approved:

Robert D. Freeman
Thesis Adviser

Clarence M. Cunningham

Dean of the Graduate School

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CHAPTER I

INTRODUCTION

A Brief Review of the Knudsen Effusion Method

Vapor pressure data are extremely valuable in calculating thermodynamic functions for high-temperature vaporization and dissociation processes. The Knudsen effusion method (16) is one of the most useful and widely employed techniques for determining pressures from 10^{-1} to 10^{-6} Torr. In this method the vapor pressure is calculated from the weight of vapor effusing through an orifice of known length and cross-sectional area in unit time at a known temperature. For the method to be applicable, conditions of molecular flow of the gaseous species through the orifice must prevail. This condition exists when the molecular mean free path is so large in comparison to the dimensions of the orifice that molecular collisions within the orifice rarely occur. A discussion of the Knudsen method and some of its varied applications is given by Margrave (17).

Knudsen, in his effusion studies (16), derived what is often called the "ideal Knudsen equation" in which the vapor pressure is related to the aforementioned quantities by

$$P = (g/at)(2\pi RT/M)^{\frac{1}{2}} \qquad (1)$$

where g is the weight of vapor effusing in time t at an ab-

solute temperature T, and R is the gas constant, a the cross-sectional area of the orifice, and M the molecular weight of the effusing species. The assumption is made here that the orifice is "ideal", or has infinitesimal length.

A major difficulty encountered in the application of Knudsen's ideal effusion equation to experimental systems arises from the necessity of using orifices of finite length rather than ideal orifices for which the equation is valid. Theoretical corrections factors for non-ideal cylindrical orifices have been derived by Clausing (6) through application of the classical kinetic theory of gases. These Clausing factors (more descriptively referred to as transmission probabilities or transmission coefficients) depend only on the ratio of the length (L) of the orifice to its diameter (D), and express the probability that a particle, having entered one end of the cylindrical orifice, will leave through the other end. Demarcus (7) has also discussed the derivation of transmission probabilities for cylindrical orifices; his initial assumptions are essentially those of Clausing but he obtains a more rigorous mathematical solution for the integral equations which arise.

It has sometimes been assumed (3,18) that the ideal orifice can be closely approximated in actual practice by certain conical, or tapered, orifices. The theoretical derivation of transmission probabilities for conical orifices, of which cylindrical orifices are a special case, has been treated by Balson (2), and more recently and more rigorously by Edwards (8) and by Iczkowsky, Margrave and Robinson (15).

When these correction factors are applied to Equation (1), it becomes

$$P = (g/Wat)(2\pi RT/M)^{\frac{1}{2}} \qquad (2)$$

where $\underline{W}$ is the transmission probability and the other symbols retain their previous meaning. Freeman (10) has given an elucidation of Equation (2) and other equations relevant to the measurement of vapor pressures by effusion techniques.

The preceding discussion of molecular flow through non-ideal orifices and its application to vapor pressure measurements is not intended to be a complete review of available information on the subject. For a more thorough summary of pertinent theories and experiments the reader is referred to the discussion of Adams (1) and Carlson (4), in addition to that by Freeman (10).

The Problem

Though the theoretical transmission probabilities which have been calculated from the model of Clausing are widely used to correct for the non-ideality of orifices used in effusion experiments, their accuracy has never been demonstrated sufficiently well by experiment. Experimental verification or refutation of these correction factors is desirable since it is not known how well they describe the actual behavior of the effusing species nor how large an error they introduce into effusion measurements.

This research was originally designed to study several associated problems:

(1) to investigate the agreement between theoretical and experimental transmission probabilities over a wide range of length to diameter (L/D) ratios, first for cylindrical orifices and then for conical orifices.

(2) to determine the upper pressure limit for the existence of molecular flow, and hence, the source pressure above which the Knudsen method is not applicable with the desired accuracy.

(3) to determine whether the transmission probability is dependent on the source pressure in the region of molecular flow as has been suggested by Adams (1).

As will be discussed in Chapter III, only the first of these problems was investigated, and this one only for cylindrical orifices.

The Multicell Approach

The experimental approach in this test of Clausing's theory will be referred to as the "multicell approach". A delineation of this approach follows.

Suppose we have a number of effusion cells, all made of the same material and constructed to be identical in every respect except that the orifice lengths differ from each other. These cells are then charged with equal amounts of the same substance and simultaneously heated for the same length of time at the same temperature. The weight of vapor which effuses from the ith cell during a run, as determined by weighing the cell before and after heating, is given by Equation (2) as

$$g_i = P_i W_i a_i t_i (M_i/2\pi RT_i)^{\frac{1}{2}}. \quad (3)$$

The weight loss for any other cell, say the kth cell, will be

$$g_k = P_k W_k a_k t_k (M_k/2\pi RT_k)^{\frac{1}{2}}. \quad (4)$$

But under the conditions dictated by the experiment, $P_i = P_k$, $a_i = a_k$, $t_i = t_k$, $M_i = M_k$, and $T_i = T_k$; Equations (3) and (4) therefore reduce to

$$W_i/W_k = g_i/g_k. \quad (5)$$

Now if one of the cells is arbitrarily chosen to be the reference cell, designated by subscript r, the ratios of the weight loss of each cell to that of the reference cell, g_i/g_r, can be calculated, and from Equation (5)

$$g_i/g_r = W_i/W_r. \quad (6)$$

For a given reference cell, the values of the ratio (g_i/g_r) for the various cells depend only on W_i, and hence only on $(L/D)_i$, for the various orifices. Therefore, if the ratios (g_i/g_r) are plotted against some suitable function of $(L/D)_i$, designated for the moment by $f(L/D)_i$, and extrapolated to $L/D = 0$, one may write, from Equation (6),

$$(g_i/g_r)_o = (W_i/W_r)_o \quad (7)$$

where the subscript zero designates the value of the quantity when $L/D = 0$. But $L/D = 0$ corresponds to the ideal orifice for which $W_i = 1$; therefore,

$$(g_i/g_r)_o = 1/W_r. \quad (8)$$

In other words, the value obtained by extrapolating a plot of (g_i/g_r) versus $f(L/D)_i$ to $L/D = 0$ is just the reciprocal of the transmission probability of the orifice in the reference cell.

With this experimental value of W_r, the experimental transmission probability for each of the other cells is calculated from Equation (6) by

$$W_i = W_r(g_i/g_r).$$

The function of $(L/D)_i$ deemed most suitable to use in this plot of (g_i/g_r) versus $f(L/D)_i$ is $W_{i(th)}$, the theoretical transmission probability calculated from the orifice dimensions. The choice of $f(L/D)_i = W_{i(th)}$ results in a bounded plot with abscissa values ranging from zero to one; furthermore, if the theoretical transmission probabilities predicted by Clausing's theory are correct, a plot of (g_i/g_r) versus $W_{i(th)}$ would give a straight line, which may be precisely extrapolated to $W_{i(th)} = 1$ (i.e., $L/D = 0$).

It may also be noted that the slope of the straight line resulting from such a plot should equal $1/W_r$. Thus, if the theoretical transmission probabilities are correct, the slope of the line which results from plotting (g_i/g_r) versus $W_{i(th)}$ will have the same value as the intercept of that line at $W_{i(th)} = 1$. However, should this slope and intercept differ, or should either or both of them differ from the theoretical value of $1/W_r$, an explanation of the observed behavior could give added insight into the limitations of Clausing's theory.

An alternate treatment of the data, essentially identical to that just described, is employed in this thesis. Here we consider the constancy, in a given run, of the ratios of the weight loss of each cell to the transmission probability for

that cell as seen by Equation (5):

$$g_i/W_i = g_k/W_k = \text{constant} = c. \tag{9}$$

When the weight losses (g_i) are plotted against a suitable function of $(L/D)_i$, again chosen to be $W_{i(th)}$, a straight line should be obtained. If this line is extrapolated to $W_{i(th)} = 1$, the extrapolated value is $(g_i)_o$, the weight loss corresponding to $L/D = 0$. We see from Equation (9) that

$$(g_i/W_i)_o = c,$$

but since $L/D = 0$ corresponds to the ideal orifice for which $W_i = 1$, it is evident that

$$(g_i)_o = c. \tag{10}$$

In other words, a plot of g_i versus $f(L/D)_i$ gives, upon extrapolation to $L/D = 0$, a value, $(g_i)_o$, which is the weight loss from a cell with an ideal orifice. This "ideal" weight loss is in turn the proportionality factor between the weight loss of any given cell and the transmission probability of the orifice in that cell.

The experimental transmission probability of each orifice is then calculated from Equations (9) and (10) by

$$W_i = g_i/(g_i)_o. \tag{11}$$

Previous Work on the Multicell Approach

Initial development of the multicell approach was done in this laboratory by Jimmie G. Edwards. The work discussed in this thesis is, in essence, a continuation of Edwards' efforts.

A brief résumé of his work will be given here as a prelude to a more detailed discussion of this author's research.

In Edwards' first experiments eight nickel-plated steel effusion cells were heated simultaneously under vacuum. The cells, which contained anthracene as the evaporant, rested in holes in an aluminum block; heating was accomplished with four cartridge heaters strategically placed in the block. Unsatisfactory results were attributed to temperature gradients in the aluminum block and in the anthracene itself. When cadmium metal was then used as the sample material, the results remained unsatisfactory. To eliminate the thermal gradients which were still present, a new vacuum system was designed and constructed, and another method of heating was employed. The cells were still held in the upper half of the same aluminum block; the lower portion which contained the cartridge heaters had been removed. The block and attached flange formed the bottom of a vacuum chamber which was placed inside a forced-air, constant-temperature oven. This method of heating produced no detected thermal gradients. However, a new problem —— oxidation of the cadmium metal samples —— was encountered and it proved to be a very persistent one. Consistent results were never obtained.

This is the system which existed when this author began work on the problem; except for relatively minor changes made at the outset, it is the system used in the present research and is described in detail in Chapter II.

CHAPTER II

APPARATUS

The Vacuum System

The all-metal vacuum system (Fig. 1) used in this work consists of the stainless steel vacuum chamber (A) connected to the "tee" (C) and a water-cooled baffle (D) by a long stainless steel arm (B). Evacuation of the system is achieved by use of a Consolidated Vacuum Corporation type MCF-60 oil diffusion pump (E) backed by a Welch Duo-Seal mechanical pump (F).

Soldered into a section of 0.875-in. diameter copper pipe between the two pumps is a thermistor (G) which is used in leak detection. The leak detector was developed in this laboratory and is discussed by Edwards (8).

A more detailed description of the components of the vacuum system follows.

The Baffle. The basic design of the Veeco type BAF-200 baffle was adapted to the existing apparatus. The modified design is shown in Figure 2. The body, which is 3 in. high and 5 in. in diameter, and the 2.25-in. diameter baffle disk in the center are made of brass, while the cooling coils are 0.25-in. copper tubing. In the top plate of the baffle are located an O-ring groove and six tapped holes which match the lower flange

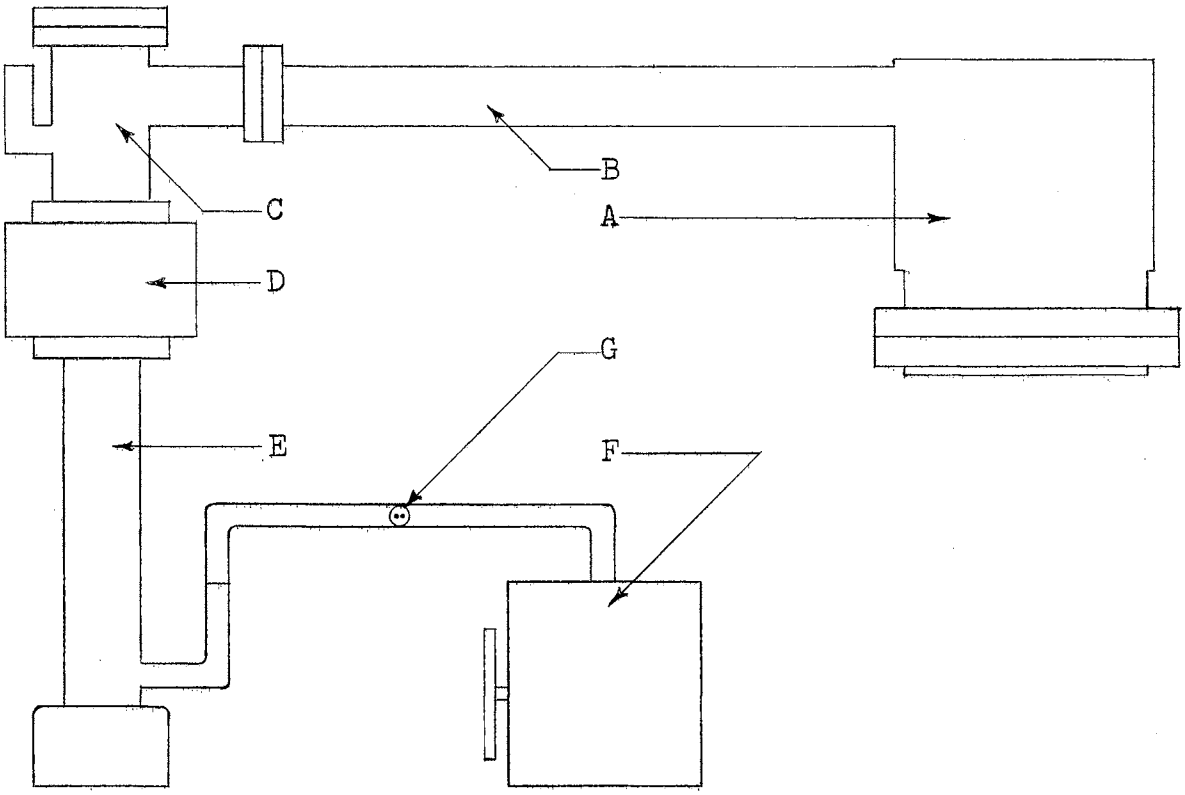


Figure 1. Vacuum System

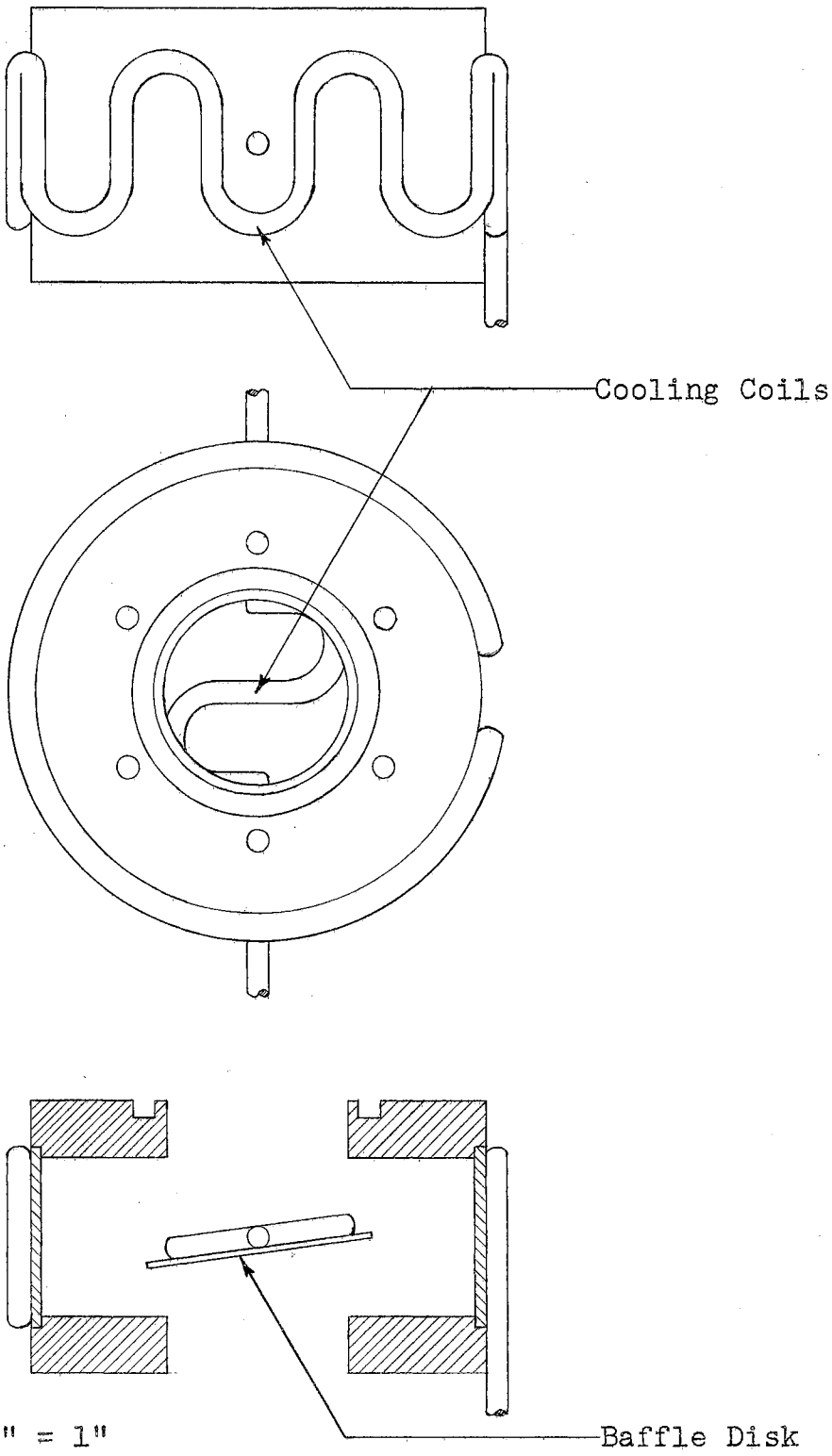


Figure 2. Water-Cooled Baffle

of the "tee". The bottom plate, which matches the flange of the diffusion pump, also contains six tapped holes but it has no O-ring groove.

The "Tee" Assembly. The "tee" assembly (Fig. 3), also made of brass, consists of a 4-in. length of 2.5-in. diameter pipe (A) joined at a right angle to a 2.5-in. length of 1.5-in. diameter pipe (B). Water circulating through 0.25-in. copper coils cools the latter arm. The flanges (C), (D) and (E) are 0.5-in. brass plate; flanges (C) and (D) each have an O-ring groove, while flange (E) is flat and seats on the O-ring in the baffle.

Two type A434 Hoke valves (F), one serving as the helium inlet to the system and the other as an opening to the atmosphere, are joined to the side of the "tee" by short lengths of copper tubing.

Initially, two holes, also on the side, were fitted with type CGB192 "Condulet" connectors (G) (Crouse-Hinds Company, Syracuse, New York) and were used to introduce the vacuum gauge sensing tubes into the system. In an attempt to eliminate their contamination by the effusing cadmium vapor, the sensing tubes were later positioned in new, similarly fitted holes (H) cut in the 0.25-in. top plate. Finally it was found necessary to move the cold cathode discharge tube to the end of an "ell"-shaped arm (I) made from 0.75-in. diameter brass tubing and fitted with a "Condulet" connector. This multiplicity of holes, though neither planned nor needed for the operation of the apparatus,

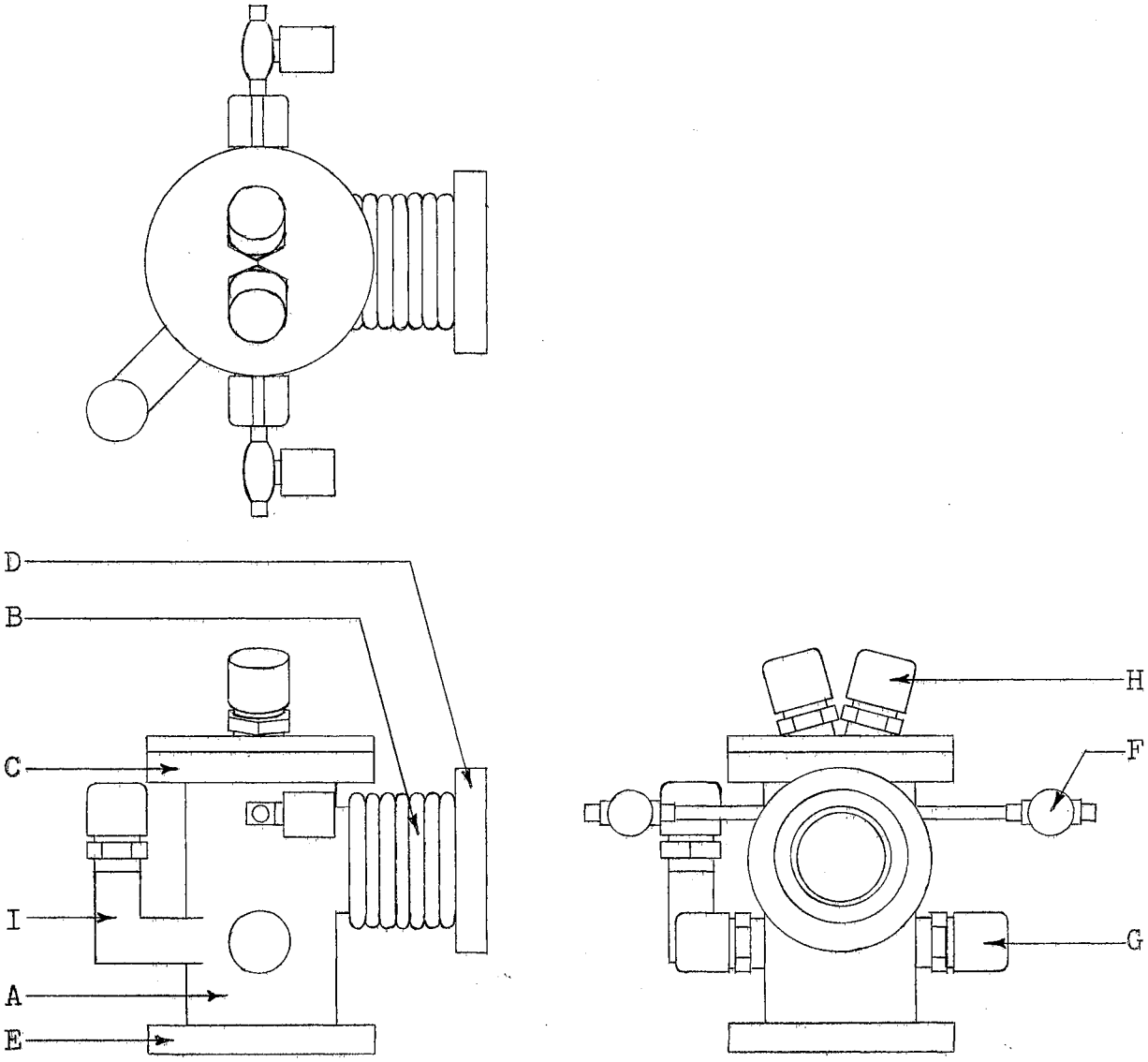


Figure 3. "Tee" Assembly

proved somewhat fortuitous in that it made simultaneous calibration of several vacuum gauges quite convenient.

One of the original purposes of the "tee" was to house a liquid nitrogen trap, but it was discovered that the decreased pumping speed resulting from the trap's geometry more than offset the advantages gained by its presence. Since adequate pressures were obtained without it, the trap was not used.

The Vacuum Chamber and Arm. The long arm (B in Fig. 1) is a 17-in. length of 1.38-in. inside diameter type 304 stainless steel pipe (schedule 40); it is heli-arc welded at a right angle to the top of the vacuum chamber (A). It allows the introduction of the chamber into the center of the oven (described subsequently) through a slot in the oven door. It should be noted here that all joints of the system which were subjected to the temperature of the oven are heli-arc welded.

The chamber itself is constructed from a 6-in. section of 6.065 inside diameter type 304 stainless steel pipe (schedule 40), the top being cut from 0.25-in. plate of the same type of steel.

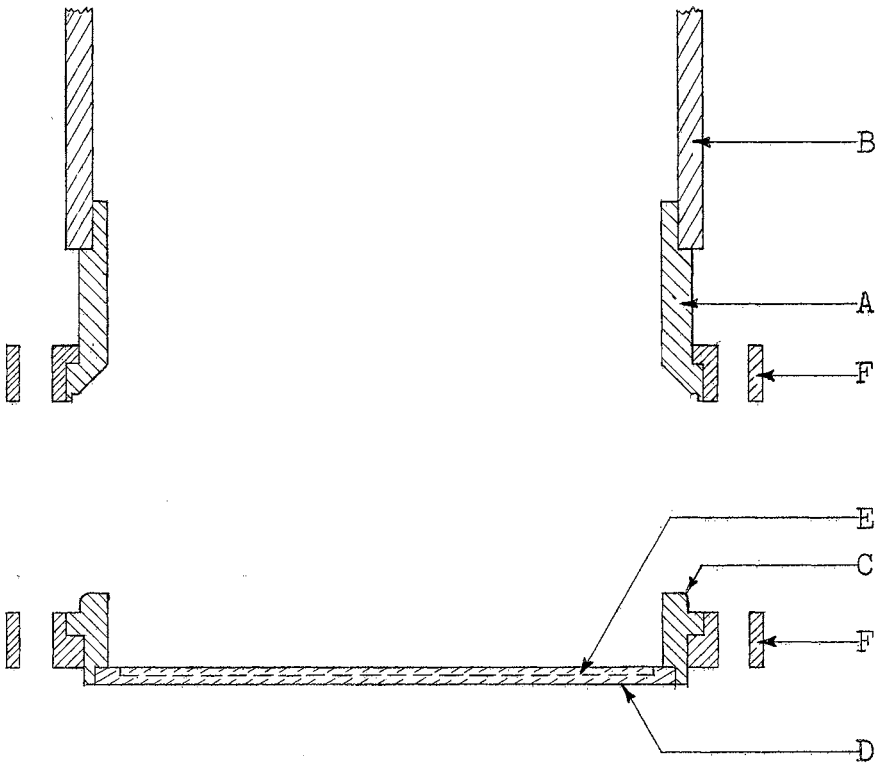
In Figure 1, the vacuum chamber (A) is shown with its bottom plate and demountable seal in their final development stage. Before this, however, when O-ring seals were attempted, there was a 0.25-in. type 304 stainless steel flange around the bottom of the chamber. A 0.375-in. aluminum flange, containing an O-ring groove and matching the steel flange on the chamber, was welded to the top of the cylindrical aluminum block which held

the eight effusion cells; the aluminum block then served as the bottom of the chamber. With this assembly the seal was made using either a silicone-rubber or "Teflon" O-ring.

When the O-ring seal was found to be unsatisfactory, a recent inovation in all-metal demountable vacuum couplings (12) was employed. The construction of this seal was aided by a number of helpful comments from Hall (13) who originated this sealing technique; henceforth in this thesis it will be referred to as the "Hall seal".

As shown cross-sectionally in Figure 4, the upper, or female, member (A) of the Hall seal is welded to the wall (B) of the vacuum chamber; the male member (C) is removable. Both members are cut from 5.761-in. inside diameter type 304 stainless steel pipe (schedule 80). Made from 0.25-in. type 304 stainless steel sheet, the bottom plate (D) has centered in it a cylindrical depression (E), 5.520 in. in diameter and 0.062 in. deep, in which is placed the aluminum cell holder. The flanges (F), made of hot rolled steel, are 0.56 in. thick with an outside diameter of 7.88 in., and are rotatable and completely separate from the rest of the seal; their inside diameters were machined to give a 0.035-in. clearance with the seal members. This clearance compensates for the difference in thermal expansion of the hot rolled and stainless steel.

The metal-to-metal seal is made between two highly polished surfaces: the conical surface of the female member and the quarter toroidal surface of the male member. An enlarged



SCALE: 1/2" = 1"

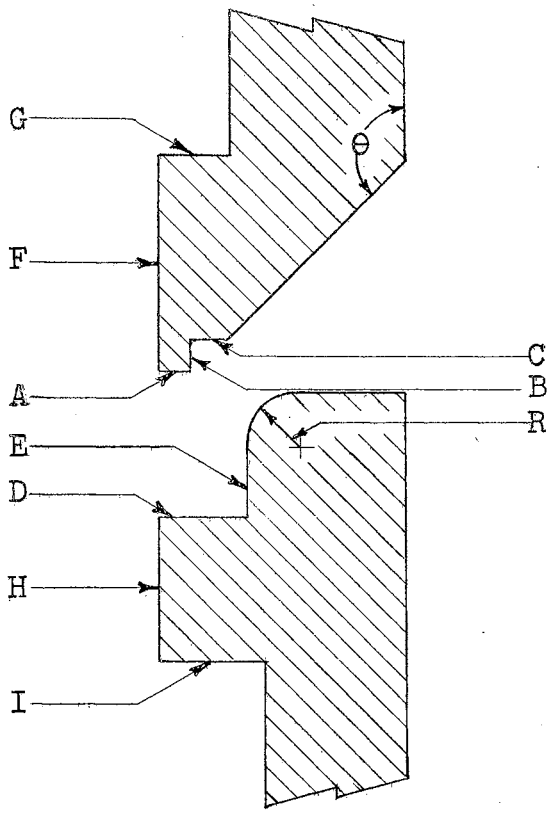
Figure 4. Hall Seal

drawing of the sealing surfaces and shoulders (Fig. 5) illustrates more clearly their geometry and dimensions, which are quite critical.

Though the Hall seal was originally designed to be used without gaskets, it was found necessary to employ them. These are flat gaskets, 6.430 ± 0.005-in. outside diameter and 5.770 ± 0.005-in. inside diameter, cut to the indicated tolerances by Industrial Gasket and Packing Co., Inc., Oklahoma City, Oklahoma, from 0.032-in. OFHC copper sheet supplied by A. D. Mackay, Inc., New York, New York.

In the top of the aluminum cell holder, which is 5.50 in. in diameter and 1.75 in. high, are cut eight cylindrical wells as shown in Figure 6. These wells, 1.0 in. deep and 1.0 in. in diameter, are arranged with their centers equidistantly spaced on a circle 3.0 in. in diameter and concentric with the top of the holder. Eight 0.125-in. diameter holes form a passage from the bottom of each well to the top surface of the holder; the cells can be conveniently removed from the wells by simply forcing air into these small holes. Four holes, which serve as thermocouple wells, were drilled 1.0 in. deep into the side of the holder, 0.5 in. from the top.

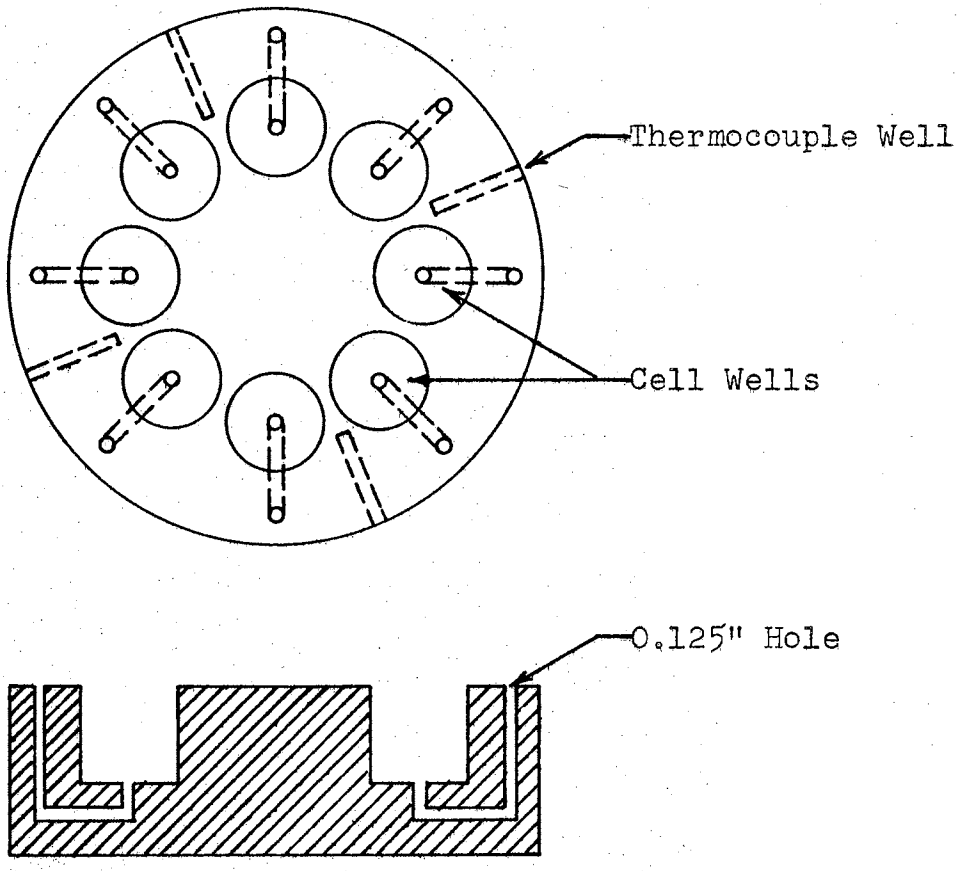
The pressure in the system is monitored with a model GV-3RS Hastings gauge in the range 760 to 10^{-3} Torr. A CVC cold cathode discharge gauge, type GPH-100A, which has been calibrated against a type RG-2A Veeco hot-filament ionization gauge is employed to monitor pressures from 10^{-3} to 10^{-6} Torr.



SCALE: 3" = 1"

A = 0.060"
B = 0.060"
C = 0.060"
D = 0.159"
E = 0.120"
F = 0.375"
G = 0.125"
H = 0.250"
I = 0.188"
R = 0.094"
θ = 135°

Figure 5. Sealing Surfaces



SCALE: 1" = 2"

Figure 6. Cell Holder

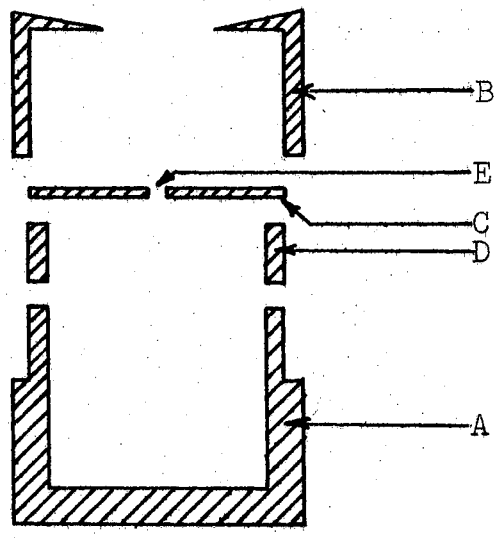
The Effusion Cells

Figure 7 is an exploded, cross-sectional view of a Knudsen effusion cell typical of those used in this experiment. These cells were fabricated from leaded steel; all components other than the orifice plates were nickel-plated to prevent corrosion.

The body (A) of the cell is 0.75 in. high and has an outside diameter of 1.0 in. at the base. This diameter is slightly smaller than those of the wells in the aluminum cell holder, so that the cell fits into the well with a clearance of about 0.001 in. The concentric cavity in the cell body is 0.625 in. deep and 0.75 in. in diameter.

The lid (B), which slips over the neck of the body and seats on the shoulder, is 0.50 in. high, making the over-all height of the cell 1.0 in. A set-screw in the side of the lid holds it firmly in position. The hole in the center of the lid is tapered and of sufficient diameter to prevent significant interaction of effusing vapor with the lid. In addition to serving as a clamp for securing the orifice plate (C), the lid increases the mass surrounding the orifice (E), thus helping to prevent the relatively thin orifice plate from becoming "cold" with respect to the remainder of the cell.

When the lid is in place, the orifice plate (C) is pressed tightly against it by the spacer ring (D). The thickness of the plates, and consequently the length of the orifices, vary from 0.0123 in. to 0.0504 in.; all orifices have 0.0630-in. diameters.



SCALE: 1 1/2" = 1"

Figure 7. Knudsen Effusion Cell

The heights of the spacer rings for the various cells vary also; the height of any given ring is just that necessary to keep the orifice plate pressed firmly against the lid.

The theoretical values of the transmission probabilities used in this research were obtained by interpolation of values computed by Heydman (14) from Clausing's angular distribution equations (5,10). These values and those calculated by Demarcus (7), by Edwards (8) and by Iczkowski, Margrave and Robinson (15) differ by less than 0.1% within the range of L/D values studied.

Table I lists the orifice dimensions and corresponding theoretical transmission probabilities for all the cells used; D is the orifice diameter, L is the length of the orifice, and W is the transmission probability. The diameters of the orifices were measured to the nearest 0.0001 in. with a traveling microscope (serial number 28327) manufactured by David W. Mann Precision Instruments, Lincoln, Massachusetts. A Brown and Sharp type 13RS micrometer was used to determine the orifice lengths to the nearest 0.0001 in. These measurements result in a maximum error in L/D ranging from 0.36% for L/D = 0.8000 to 0.97% for L/D = 0.1952. This in turn causes a maximum possible error of 0.16% in the values of the transmission probabilities.

Changes in the orifice dimensions with temperature need not be considered in this experiment. For a given change of temperature, the fractional changes in both the length and the diameter are identical; consequently, L/D is independent of the temperature.

TABLE I

EFFUSION CELL CONSTANTS

Cell	L(in.)	L/D[a]	W
A	0.0123±0.0001	0.1952	0.8374±0.0013
P	0.0183±0.0001	0.2905	0.7767±0.0011
B	0.0194±0.0001	0.3079	0.7666±0.0012
U	0.0261±0.0001	0.4143	0.7109±0.0010
I	0.0282±0.0001	0.4476	0.6953±0.0011
E	0.0328±0.0001	0.5206	0.6635±0.0010
M	0.0329±0.0001	0.5222	0.6628±0.0010
S	0.0342±0.0001	0.5429	0.6544±0.0010
N	0.0385±0.0001	0.6111	0.6284±0.0010
F	0.0420±0.0001	0.6667	0.6087±0.0009
X	0.0504±0.0001	0.8000	0.5669±0.0009

a. D = 0.0630±0.0001 in. for all cells.

The Helium Inlet Assembly

The purpose of introducing helium into the system is threefold: (1) to prevent effusion of the sample while heating the Knudsen cells to the desired operating temperature; (2) to insure thermal equilibrium (and to facilitate its attainment) in the vacuum chamber before effusion begins; (3) to stop effusion from the Knudsen cells at the termination of the heating period.

Initially the helium inlet assembly consisted of a tank supplying Matheson research grade helium through a cold trap into the "tee"; an open-end mercury manometer, used to indicate near-atmospheric pressures, was connected to the helium line at the cold trap.

The first modification of this assembly was the addition of a "pressure regulator" in which the helium is forced to bubble through a column of mercury in escaping to the atmosphere. The height of the mercury column is such that the maximum pressure attainable in any part of the system open to the regulator is about 35 Torr above atmospheric pressure. The primary function of the regulator is to allow a slight positive pressure of helium to be maintained in the vacuum chamber while the effusion cells are cooling after the completion of a run. The regulator also serves to prevent an excessive helium pressure in the chamber when the cells are being brought to temperature in preparation for a run. This modification was made in an attempt to solve the problem of sample oxidation, which is discussed in

Chapter III. O-ring seals were being used on the chamber at the time. Prior to the introduction of the regulator, it was thought that excessively high pressures within the chamber at temperature might have been distorting the O-ring. As the chamber cooled, this distortion could have allowed seepage of air into the system with subsequent oxidation of the warm sample. Use of the pressure regulator did decrease sample oxidation, but only slightly; the problem persisted. However, because of its convenience, the regulator remained a part of the assembly.

Another possible source of the contaminating oxygen in the vacuum chamber was the helium, despite its extremely high purity specifications. To eliminate contamination from this source, a 16-in. long column of cadmium turnings was packed into a "Pyrex" tube which could be heating to 260°C in a tube furnace. It was introduced into the helium line upstream from the cold trap and manometer. This, too, failed to eliminate the oxidation; nevertheless, use of the hot cadmium turnings was continued as a worth-while precaution.

Also installed was a "flow rate indicator" which serves additionally as a convenient outlet to the atmosphere when the helium line is being flushed. To get an indication of its flow rate, the escaping gas is bubbled through used Octoil pump fluid.

A schematic illustration of the entire helium inlet assembly as it ultimately existed is shown in Figure 8.

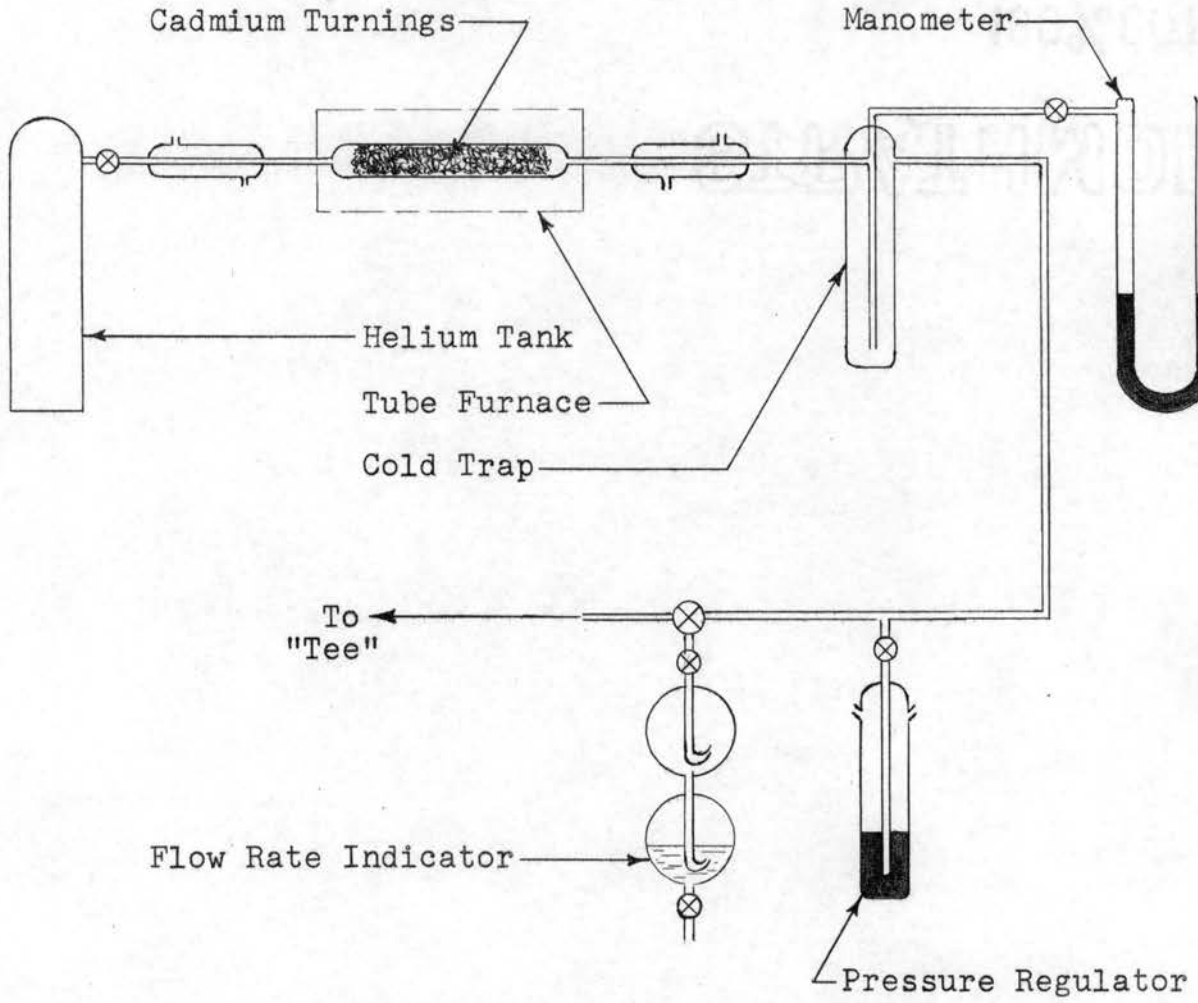


Figure 8. Helium Inlet Assembly

Temperature Control and Measurement

During a run, the vacuum chamber and effusion cells are enclosed by a circulating-air, constant-temperature oven. This is a model OV-490 Stabil-Therm oven produced by Blue M Electric Company, Blue Island, Illinois. As was mentioned earlier, the vacuum chamber is introduced into the center of the oven through a slot cut in the door; the slot fits around the stainless steel arm attached to the chamber. An asbestos plug then covers the portion of the slot not occupied by the arm.

The temperature of the vacuum chamber, and consequently the approximate temperature of the cells, is indicated by a Fisher mercury thermometer (No. 14-985) which is introduced into the oven through a hole provided in the top. This temperature measurement is sufficient since it is not required that the temperature be accurately known — only that the cells be kept at a uniform and fairly constant temperature. Had the study of the effects of source pressure on the transmission probability been made, it would have been necessary to know the temperature of the cells more exactly. This would necessitate the insertion of one or more thermocouples in the wells provided in the cell holder.

CHAPTER III

EXPERIMENTAL

Only the first of the three problems mentioned in Chapter I, that of the agreement between theoretical and experimental transmission probabilities for cylindrical orifices, was studied in this research. The major difficulty encountered was oxidation of the cadmium samples during the heating period; the persistence of this problem prevented further progress in this work. When silicone-rubber and "Teflon" O-rings were employed in the demountable seal, the oxidation was usually quite extensive. Conversion to the metal-to-metal seal decreased the extent of the oxidation to a very slight discoloration on the cadmium surface; however, it remained too great to be considered insignificant.

That the yellow coating on the surface was CdO was verified by x-ray diffraction. For this analysis, a 114.59 mm. diameter Philips Debye Scherrer Type 52056-B Powder Camera was used in conjunction with a copper x-ray source and a nickel filter.

Experimental Procedure

The procedure followed in all the runs for which the data are evaluated in this thesis was as follows.

(1) The effusion cells were cleaned by washing them first in 4M acetic acid to dissolve any CdO, then in distilled water, and finally in reagent grade acetone. When the cadmium samples were used more than once, they, too, were cleaned the same way.

(2) After being dried at about 50°C for approximately ten minutes, the cells were charged with sample, which consisted of small chunks of cadmium; eight to ten chunks were used in each cell. The cadmium used as the evaporant was supplied by A. D. Mackay, Inc., and had a stated purity of 99.95 percent.

(3) After cooling in a desiccator, the charged cells were weighed to $\pm$0.02 mg on a Model SM 10/100 g. Sartorius-Werke semi-micro balance.

(4) When the Hall seal was used, the aluminum cell holder with the charged cells in place was then positioned on the bottom plate of the vacuum chamber and the seal was made; with the O-ring seal, the aluminum block itself served as the bottom of the chamber as described in Chapter II.

(5) The vacuum chamber was evacuated to 10^{-5} Torr or less. At this pressure the pumps were turned off and highly purified helium was introduced into the chamber in which a slight positive pressure of about 35 Torr was maintained by the "pressure regulator" mentioned in Chapter II.

(6) The vacuum chamber was next introduced into the constant-temperature oven, and the effusion cells were brought to the desired temperature under the helium atmosphere.

(7) After thermal equilibrium was attained in the chamber, the pumps were turned on to evacuate the system. The lengths of the runs varied; when O-rings were used in the bakeable seal, effusion usually lasted between three and four hours, but with the metal-to-metal seal it was usually necessary to permit effusion for about six hours to obtain comparable weight losses. The author does not fully understand this peculiar behavior. It may be caused by some phenomenon associated with the oxidation of the cadmium.

(8) To stop the effusion, the pumps were turned off and helium was again introduced until a positive pressure of 35 Torr existed in the chamber.

(9) The oven was removed and the cells were allowed to cool under the helium atmosphere.

(10) After the cells had cooled, the chamber was evacuated with the mechanical pump to permit the effusion of helium from inside the cells. This procedure prevented drift, caused by loss of residual helium from the cell, during their subsequent weighing.

(11) At this point, the pump was turned off, the system opened to the atmosphere, and the cells removed and placed in the desiccator.

(12) The cells were weighed again and the respective weight losses were calculated. These weight losses usually ranged from twenty to forty milligrams, but occasionally were as high as seventy milligrams. That none of this weight loss was attributable to any change in weight of the cell itself was previously established by repeated heating of the empty cell to 270°C; the cell weight after each heating cycle remained constant to within 0.02 mg.

Performance of the Apparatus

The Oven. All the runs were made at a temperature between 250° and 255°C. In this range, the temperature of the circulating-air oven was maintained constant to ±2°C.

The Vacuum System. The pressures attained in the vacuum system were quite adequate; the performance of the system with each of the three types of demountable seals used on the chamber will be discussed in turn.

(1) The system with a silicone-rubber O-ring:

a. At room temperature the chamber pressure could be reduced from atmospheric pressure to 5×10^{-5} Torr in fifteen minutes and would ultimately reach about 1×10^{-5} Torr after an hour of pumping.

b. During a run, after thermal equilibrium was established and the pumps were turned on, the pressure would drop from 800 Torr to 5×10^{-5} Torr within

thirty minutes, and would be about 2×10^{-5} Torr at the completion of the run.

(2) The system with a "Teflon" O-ring:

a. At room temperature the chamber was evacuated to 1×10^{-5} Torr within twenty minutes, and ultimately pumped down to about 5×10^{-6} Torr in several hours.

b. During a run, after thermal equilibrium was established, the pressure remained rather high initially, but then dropped rapidly to 5×10^{-5} Torr within thirty minutes after the pumps were turned on. At the termination of the run, the chamber pressure was less than 7×10^{-6} Torr.

The major difficulty encountered with the "Teflon" O-rings seemed to be their tendency to leak as the seal cooled after the oven was removed.

(3) The system with the Hall Seal:

a. At room temperature the pressures attained were comparable to those attained with "Teflon" O-rings; ultimately, the pressure would decrease to about 7×10^{-6} Torr, but a longer pumping time was usually required to achieve this pressure with the Hall seal than with the "Teflon" O-rings.

b. During a run, after thermal equilibrium was established and the pumps were turned on, the pressure dropped to 5×10^{-5} Torr within fifteen minutes and to 1×10^{-5} Torr within an hour, finally reaching

5×10^{-6} Torr and lower at the end of the run. The best performance of the vacuum system at the temperature of the runs was obtained with the Hall seal; this might be expected since the expansion of the copper gasket at elevated temperatures would tend to give a tighter seal.

Results

The data analyzed subsequently are from seven "acceptable" runs: two runs with a silicone-rubber O-ring (runs 1 and 2); one with a "Teflon" O-ring (run 3); and four with the Hall seal and copper gasket (runs 4, 5, 6 and 7). A run was considered acceptable if (1) the experimental procedure followed was that given earlier in this chapter, (2) the apparatus performed satisfactorily, (3) sample oxidation was comparatively slight for the type of seal used, and (4) sufficiently high weight losses resulted.

Of the three runs in which O-rings were used, runs 1 and 2 yielded the best data. The results of run 3 are the poorest of the seven runs. The most consistent data were obtained from the four runs employing the Hall seal.

The data are presented according to the alternate treatment discussed in Chapter I, i.e., the weight loss, g_i, is plotted versus the theoretical transmission probability, $W_{i(th)}$, and extrapolated to $W_{i(th)} = 1$. The least squares straight line is assumed to be the "best" line through the points.

Data for three of the runs are represented graphically in Figures 9, 10 and 11 to illustrate the scatter of experimental points about the least squares line; the first two represent the smallest scatter obtained, while that shown in Figure 11 is the greatest.

The weight losses, g_i, for the seven runs are given in Table II. Included in the Table with each set of weight losses are the slope, $\underline{m}$, and the intercept, $\underline{b}$, of the least squares line of the form

$$g_i = mW_{i(th)} + b,$$

and $\underline{(g_i)_o}$, the "extrapolated" value at $W_{i(th)} = 1$ (actually, $\underline{(g_i)_o}$ is calculated as the algebraic sum of $\underline{m}$ and $\underline{b}$).

These data were used to calculate the experimental transmission probabilities in two different ways: (1) the ordinate value of each experimental point was divided by $\underline{(g_i)_o}$ — Equation (11) of Chapter I; (2) the corresponding ordinate values on the least squares line were divided by $(g_i)_o$. In Table III, the transmission probabilities calculated in the two ways from the data of run 7 are compared to the theoretical transmission probabilities. In the table, W_p and W_{ls} are the experimental transmission probabilities calculated from the experimental points and from the least squares line respectively. The fifth and seventh columns of the table give the percentage deviation of each of the two experimental quantities from the theoretical transmission probability of each orifice. The agreement between theory and experiment in run 7 is about average for the seven

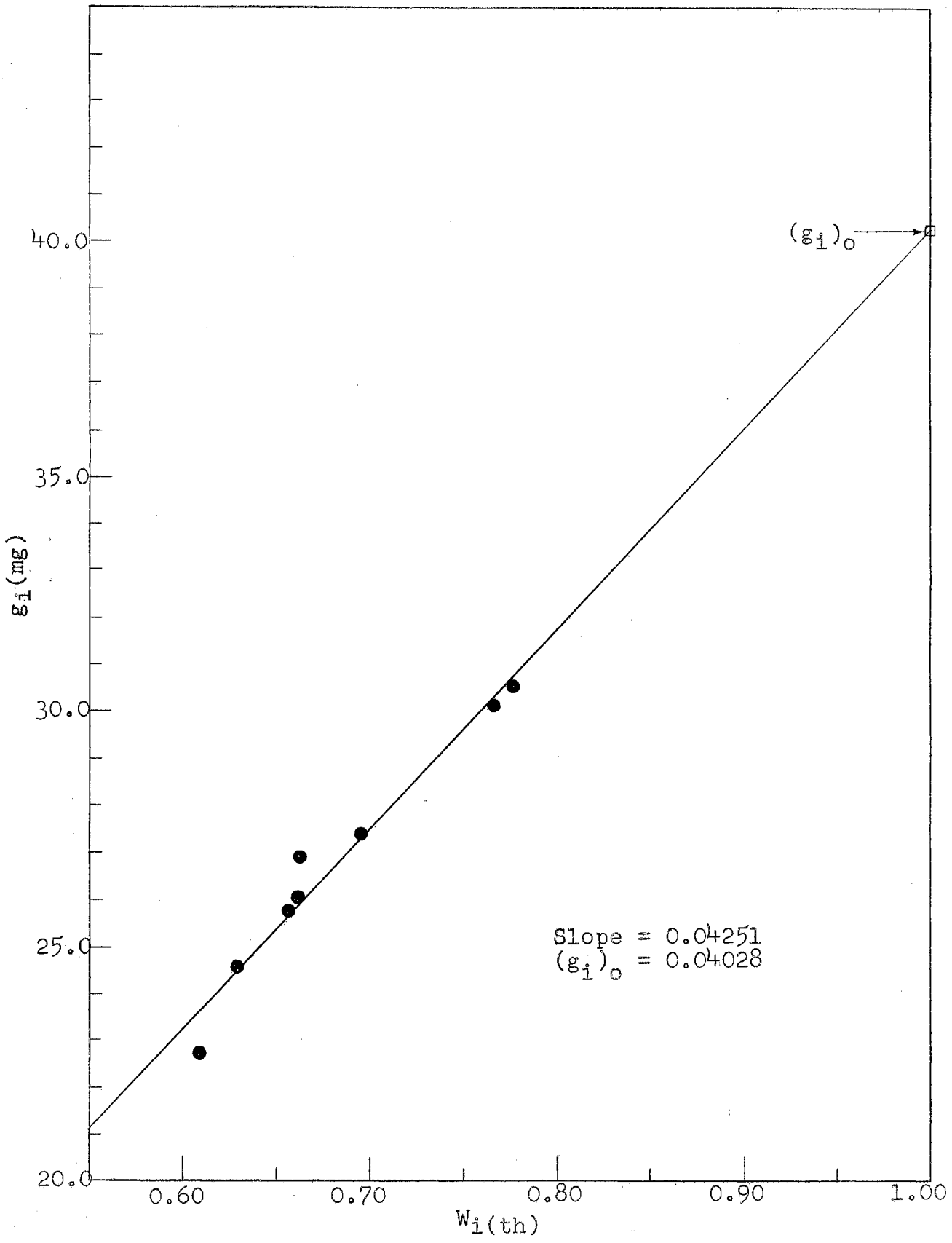


Figure 9. Run 2

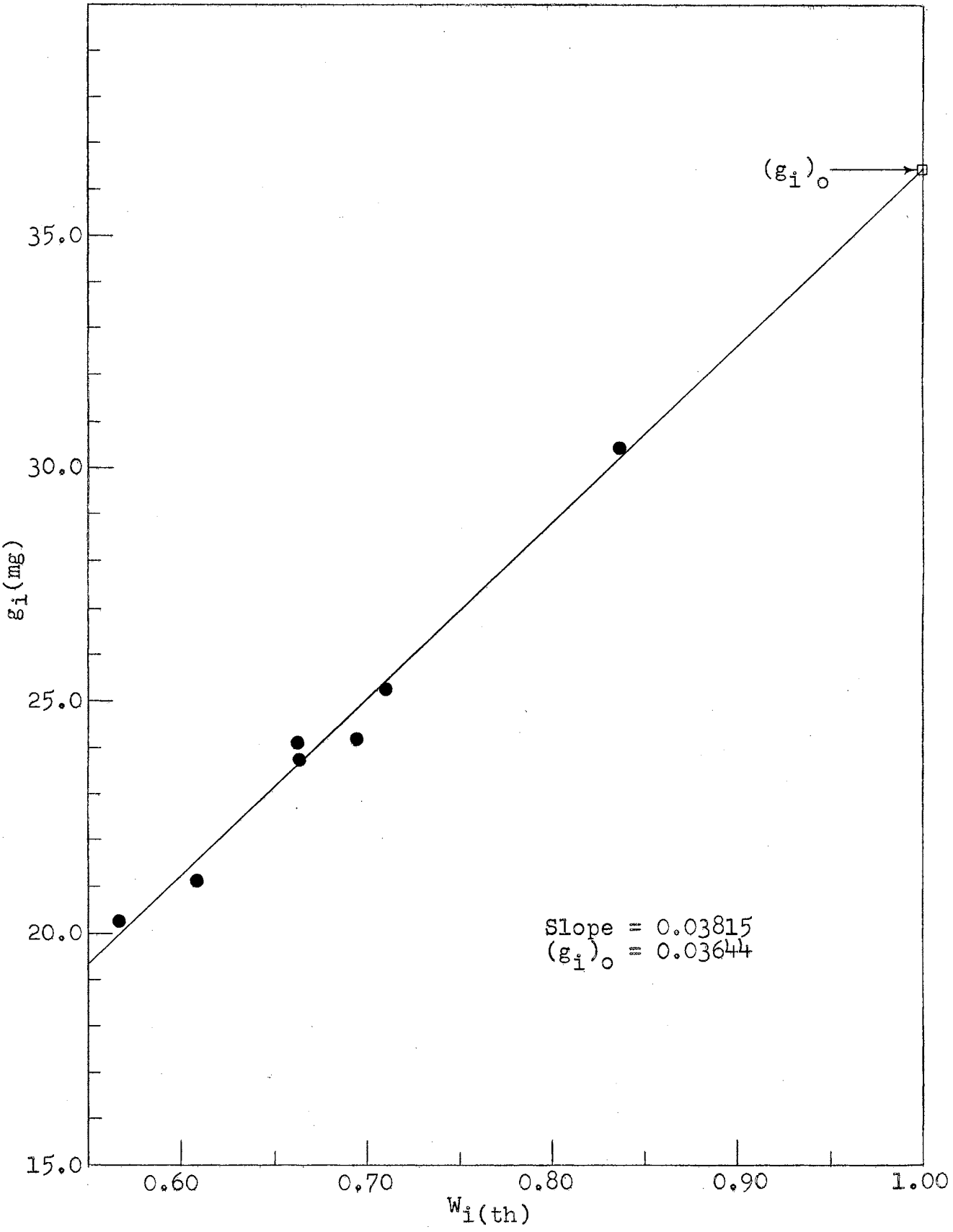


Figure 10. Run 4

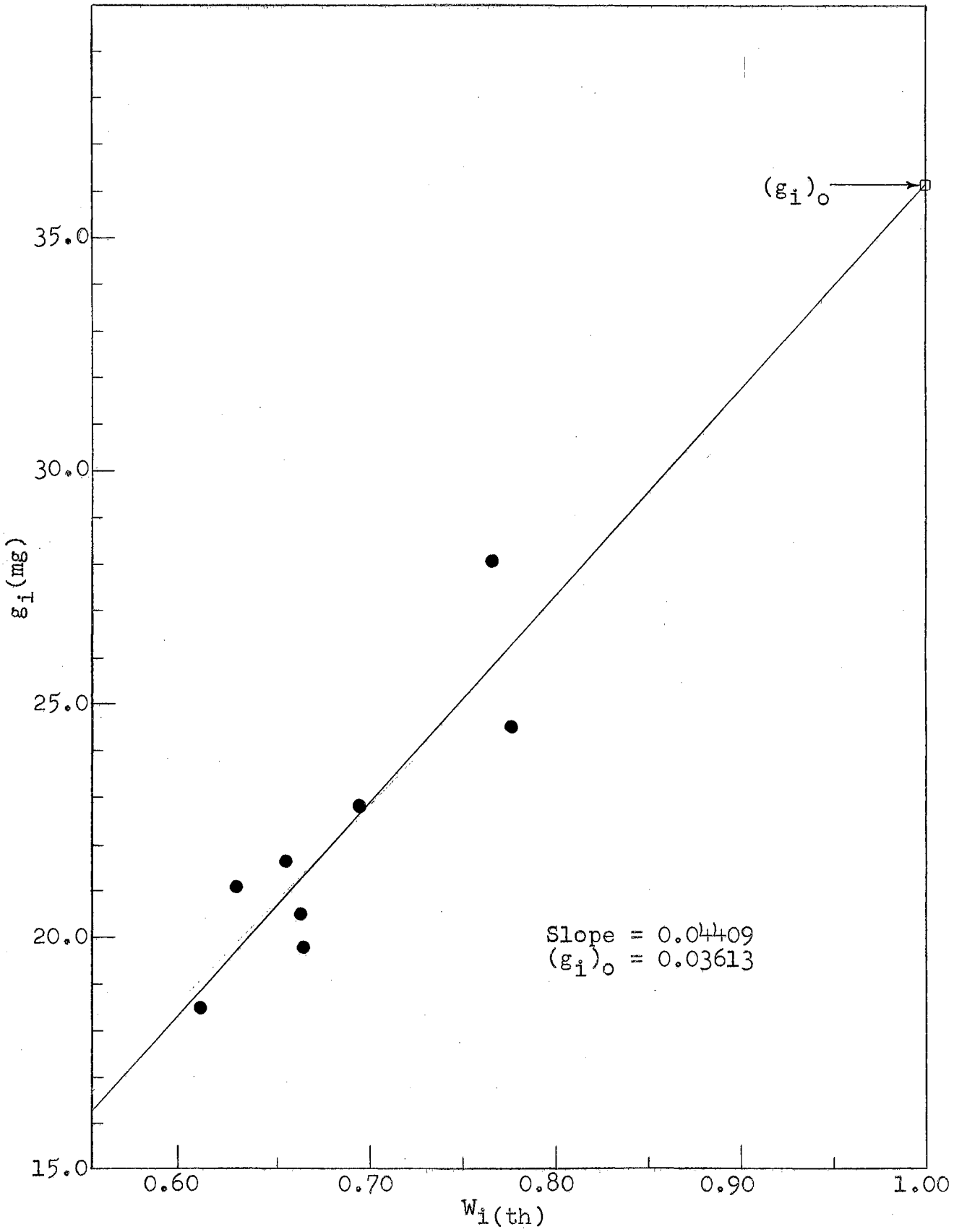


Figure 11. Run 3

TABLE II

WEIGHT LOSSES AND LEAST SQUARES DATA

Run 1

Cell	g_i (mg)	Cell	g_i (mg)	Cell	g_i (mg)	Cell	g_i (mg)
B	22.83	F	19.43	M	21.09	P	25.51
E	20.17	I	23.04	N	20.93	S	20.73

$m=+0.02877$, $b=+0.00209$, $(g_i)_o=+0.03086$

Run 2

Cell	g_i (mg)	Cell	g_i (mg)	Cell	g_i (mg)	Cell	g_i (mg)
B	30.14	F	22.72	M	26.08	P	30.56
E	26.91	I	27.39	N	24.57	S	25.74

$m=+0.04251$, $b=-0.00223$, $(g_i)_o=+0.04028$

Run 3

Cell	g_i (mg)	Cell	g_i (mg)	Cell	g_i (mg)	Cell	g_i (mg)
B	28.08	F	18.48	M	20.49	P	24.49
E	19.76	I	22.80	N	21.08	S	21.68

$m=+0.04409$, $b=-0.00796$, $(g_i)_o=+0.03613$

Run 4

Cell	g_i (mg)	Cell	g_i (mg)	Cell	g_i (mg)	Cell	g_i (mg)
A	30.44	F	21.12	M	24.11	U	25.26
E	23.72	I	24.18	P	cell not used	X	20.22

$m=+0.03815$, $b=-0.00171$, $(g_i)_o=+0.03644$

TABLE II (Continued)

Run 5

Cell	g_i (mg)	Cell	g_i (mg)	Cell	g_i (mg)	Cell	g_i (mg)
A	29.75	F	21.39	M	22.97	U	25.84
E	25.91	I	24.18	P	25.19	X	18.72

$m=+0.03457$, $b=+0.00038$, $(g_i)_o=+0.03495$

Run 6

Cell	g_i (mg)	Cell	g_i (mg)	Cell	g_i (mg)	Cell	g_i (mg)
A	69.06	F	47.71	M	55.28	U	55.76
E	55.90	I	56.60	P	61.90	X	46.15

$m=+0.08076$, $b=+0.00020$, $(g_i)_o=+0.08096$

Run 7

Cell	g_i (mg)	Cell	g_i (mg)	Cell	g_i (mg)	Cell	g_i (mg)
A	42.36	F	28.85	M	31.91	U	35.62
E	33.83	I	32.94	P	38.16	X	28.55

$m=+0.05239$, $b=-0.00213$, $(g_i)_o=+0.05025$

TABLE III

COMPARISON OF EXPERIMENTAL AND THEORETICAL TRANSMISSION PROBABILITIES: RUN 7

Cell	L/D	W_{th}	W_p	$\frac{W_p - W_{th}}{W_{th}} \times 100$	W_{ls}	$\frac{W_{ls} - W_{th}}{W_{th}} \times 100$
A	0.195	0.837	0.8429	+0.7	0.8305	-0.8
P	0.290	0.777	0.7593	-2.3	0.7672	-1.3
U	0.414	0.711	0.7088	-0.3	0.6986	-1.8
I	0.448	0.695	0.6555	-6.0	0.6824	-1.8
E	0.521	0.664	0.6732	+1.4	0.6492	-2.2
M	0.522	0.663	0.6350	-4.4	0.6485	-2.2
F	0.667	0.609	0.5741	-6.1	0.5921	-2.9
X	0.800	0.567	0.5681	+0.2	0.5485	-3.4

runs, it is, however, poorer than average for the four runs with the Hall seal. The results of this run are presented here to give the reader some quantitative feeling for the agreement between experimental and theoretical values in individual runs.

Because of the scatter of points about the least squares line and the internal inconsistency in the results obtained for the individual runs, the author feels that averaging the transmission probabilities calculated for each orifice gives more reliable values than those obtained from any single, arbitrarily chosen run. These "average" transmission probabilities have been calculated, and the results are summarized in Table IV. Columns four through ten of this table give the transmission probabilities calculated from the individual runs. It will be noticed that two values are given for each orifice: the first of these values is the transmission probability calculated from the experimental points (comparable to $\underline{W_p}$ in Table III), and the second is the value calculated from the least squares line (comparable to $\underline{W_{ls}}$ in Table III). In Table IV, $\underline{W_{13}}$ is the average of the transmission probabilities calculated for runs 1, 2 and 3; $\underline{W_{47}}$ is the average of the transmission probabilities calculated for runs 4 through 7; and $\underline{W_{17}}$ is the average of the values for all seven runs. In columns twelve, fourteen and sixteen, these "average" experimental transmission probabilities are compared to the theoretical value for each orifice.

Information similiar to that summarized in Table IV is also presented in Table V; the latter covers L/D values ranging, in

TABLE IV

EXPERIMENTAL TRANSMISSION PROBABILITIES FOR ELEVEN CYLINDRICAL ORIFICES

Cell	L/D	W_{th}	$W_{(experimental)}$ Silicone-Rubber O-Ring Run 1	Silicone-Rubber O-Ring Run 2	"Teflon" O-Ring Run 3	Hell Seal Run 4	Hell Seal Run 5	Hell Seal Run 6	Hell Seal Run 7	W_{13}	$\frac{W_{13}-W_{th}}{W_{th}}\times 100$	W_{47}	$\frac{W_{47}-W_{th}}{W_{th}}\times 100$	W_{17}	$\frac{W_{17}-W_{th}}{W_{th}}\times 100$
A	0.195	0.837	—	—	—	0.8354[a]	0.8512	0.8530	0.8429	—	—	0.846	+1.1	—	—
			—	—	—	0.8297[b]	0.8392	0.8378	0.8305	—	—	0.834	-0.4	—	—
P	0.290	0.777	0.8265[a]	0.7587	0.6779	—	0.7207	0.7546	0.7593	0.754	-3.0	0.745[c]	-4.1	0.750[d]	-3.5
			0.7918[b]	0.7643	0.7275	—	0.7791	0.7772	0.7672	0.761	-2.1	0.775[c]	-0.3	0.768[d]	-1.2
B	0.308	0.767	0.7397[a]	0.7483	0.7773	—	—	—	—	0.755	-1.6	—	—	—	—
			0.7824[b]	0.7537	0.7151	—	—	—	—	0.750	-2.2	—	—	—	—
U	0.414	0.711	—	—	—	0.6932[a]	0.7393	0.6888	0.7088	—	—	0.708	-0.4	—	—
			—	—	—	0.6973[b]	0.7141	0.7116	0.6986	—	—	0.706	-0.7	—	—
I	0.448	0.695	0.7465[a]	0.6780	0.6311	0.6636	0.6918	0.6991	0.6555	0.685	-1.4	0.678	-2.4	0.681	-2.0
			0.7160[b]	0.6784	0.6281	0.6810	0.6986	0.6960	0.6824	0.674	-3.0	0.690	-0.7	0.683	-1.7
E	0.521	0.664	0.6535[a]	0.6681	0.5470	0.6510	0.7413	0.6905	0.6732	0.623	-6.2	0.689	+3.8	0.661	-0.5
			0.6863[b]	0.6449	0.5893	0.6477	0.6672	0.6643	0.6492	0.640	-3.6	0.657	-1.1	0.650	-2.1
M	0.522	0.663	0.6833[a]	0.6475	0.5672	0.6617	0.6572	0.6828	0.6350	0.633	-4.5	0.659	-0.6	0.648	-2.3
			0.6857[b]	0.6441	0.5884	0.6469	0.6665	0.6636	0.6485	0.639	-3.6	0.656	-1.1	0.649	-2.1
S	0.543	0.654	0.6716[a]	0.6390	0.6001	—	—	—	—	0.637	-2.7	—	—	—	—
			0.6778[b]	0.6353	0.5782	—	—	—	—	0.630	-3.7	—	—	—	—
N	0.611	0.628	0.6781[a]	0.6100	0.5835	—	—	—	—	0.624	-0.6	—	—	—	—
			0.6535[b]	0.6078	0.5465	—	—	—	—	0.603	-4.0	—	—	—	—
F	0.667	0.609	0.6295[a]	0.5641	0.5115	0.5796	0.6120	0.5893	0.5741	0.568	-6.7	0.589	-3.3	0.580	-4.8
			0.6352[b]	0.5870	0.5224	0.5903	0.6130	0.6096	0.5921	0.582	-4.4	0.601	-1.3	0.593	-2.6
X	0.800	0.567	—	—	—	0.5549[a]	0.5356	0.5701	0.5681	—	—	0.557	-1.8	—	—
			—	—	—	0.5465[b]	0.5716	0.5679	0.5485	—	—	0.559	-1.4	—	—

a. The upper number of each pair is the value obtained from the experimental points.
b. The lower number of each pair is the value obtained from the least squares line.
c. This value is the average of three numbers instead of four.
d. This value is the average of six numbers instead of seven.

TABLE V

EXPERIMENTAL TRANSMISSION PROBABILITIES FOR L/D VALUES RANGING FROM 0.100 TO 1.000

		$W_{(experimental)}$												
		Silicone Rubber O-Ring		"Teflon" O-Ring	Hall Seal									
L/D	W_{th}	Run 1	Run 2	Run 3	Run 4	Run 5	Run 6	Run 7	W_{13}	$\frac{W_{13}-W_{th}}{W_{th}}\times 100$	W_{47}	$\frac{W_{47}-W_{th}}{W_{th}}\times 100$	W_{17}	$\frac{W_{17}-W_{th}}{W_{th}}\times 100$
0.100	0.909	0.9154	0.9042	0.8892	0.9049	0.9102	0.9094	0.9053	0.903	-0.6	0.908	-0.1	0.906	-0.3
0.200	0.834	0.8454	0.8249	0.7975	0.8263	0.8359	0.8345	0.8271	0.823	-1.3	0.831	-0.4	0.827	-0.8
0.300	0.771	0.7867	0.7585	0.7207	0.7604	0.7737	0.7718	0.7615	0.755	-2.1	0.767	-0.5	0.762	-1.2
0.400	0.718	0.7369	0.7022	0.6556	0.7045	0.7209	0.7185	0.7058	0.698	-2.8	0.712	-0.8	0.706	-1.7
0.500	0.672	0.6953	0.6539	0.5998	0.6567	0.6757	0.6729	0.6582	0.650	-3.3	0.666	-0.9	0.659	-1.9
0.600	0.632	0.6573	0.6120	0.5513	0.6151	0.6364	0.6333	0.6168	0.607	-4.0	0.625	-1.1	0.618	-2.2
0.700	0.598	0.6249	0.5753	0.5089	0.5787	0.6020	0.5986	0.5805	0.570	-4.7	0.590	-1.3	0.581	-2.8
0.800	0.567	0.5963	0.5429	0.4714	0.5465	0.5716	0.5679	0.5485	0.537	-5.3	0.559	-1.4	0.550	-3.0
0.900	0.539	0.5706	0.5139	0.4378	0.5177	0.5444	0.5405	0.5198	0.507	-5.9	0.531	-1.5	0.521	-3.3
1.000	0.515	0.5476	0.4878	0.4077	0.4919	0.5200	0.5159	0.4941	0.481	-6.6	0.506	-1.7	0.495	-3.9

even intervals, from 0.1 to 1.0. The experimental transmission probabilities given for each run are calculated from the least squares line.

Discussion

As was pointed out earlier in this chapter, the non-reproducibility of the data from one run to the next prevents one from placing much confidence in the results of any single run; consequently, the transmission probabilities were averaged as shown in Tables IV and V. It is felt that these average values are the best that can be calculated from the scattered experimental points obtained in the individual runs.

The question arises as to which of the two experimental transmission probabilities calculated for each orifice is the more accurate, W_p from the experimental points or W_{ls} from the least squares line. The following considerations cause the author to conclude that W_{ls} represents the better value. It is believed that the greatest, single contributing factor to the scatter of the experimental points is oxidation of the cadmium samples. If this is so and if the oxidation were eliminated, one would expect the points to fall very nearly on a straight line (the experimental error, excluding oxidation effects, is estimated to be less than 0.2%). Since the experimental points of these seven runs do not fall on straight lines, as would be expected of <u>good</u> data, the transmission probabilities calculated from the "best" straight line drawn through the points should be

more accurate than those calculated from the individual points themselves.

The author gives more credence to the results of the four runs using the Hall seal than to the results of the first three runs. Very little cadmium oxide was visible on the sample surfaces after runs 4, 5, 6 and 7 — much less than after the other runs. It will be noted, too, (Tables IV and V) that the individual transmission probabilities calculated from the least squares lines for the last four runs show greater consistency than the corresponding transmission probabilities obtained from runs 1, 2 and 3. For these reasons, the author feels that W_{47} and W_{17}, but not W_{13}, are the experimental values which should be considered: W_{17}, because it is the average resulting from all the acceptable runs; W_{47}, because of the greatly reduced sample oxidation and more consistent data in runs 4 and 7. However, if it were necessary to choose between the two, this author would favor, slightly, W_{47}, thereby disregarding completely the less reliable data of runs 1, 2 and 3.

The lack of reproducible data in this research makes it difficult to arrive at any definite conclusions concerning the validity of Clausing's theory. The data presented here indicate that Clausing's predictions agree with experiment to within two percent (if W_{47} calculated from the least squares line is accepted as the most accurate value) for L/D values up to 1.0. This most certainly is not a refutation of the Clausing Theory; neither is it sufficient confirmation.

The results of this work, inconclusive as they may be, are promising enough to warrant further study of this and associated problems by the multicell approach. But first, certain modifications of the vacuum system should be made; a number of such modifications are discussed in the next chapter. If the problem of sample surface contamination is eliminated, the multicell technique should give good data on transmission probabilities.

CHAPTER IV

SUGGESTED MODIFICATIONS

It is apparent from the preceding discussion that the results from this experiment lack sufficient consistency. These results, though encouraging, indicate that redesign of the apparatus is in order. This chapter is devoted to modifications of the present design which this author feels would eliminate some of the existing problems and improve the over-all performance of the system.

It is not intended that these suggestions should comprise a detailed description of a refined vacuum system. Certainly, a more careful consideration of the specific dimensions and the details of the design would be necessary before these suggestions could be put into practice.

The Effusion Chamber and "Tee"

It appears from all indications that the major source of trouble in the present experiment is sample oxidation; it is believed that the contaminating oxygen enters the system at operating temperatures primarily, if not solely, through the demountable seal on the chamber. Keeping all seals outside the oven should eliminate this problem. The author feels that this is the single most important change to be made; it could be con-

veniently accomplished with a design similar to that illustrated in Figure 12. The cells could be heated to much higher temperatures if desired; consequently, a higher temperature, i.e., source pressure, range might be investigated, or another substance with a lower vapor pressure might be used as the evaporant.

To obtain a greater pumping speed, the effusion chamber section and the horizontal arm of the "tee" could be made of six- or eight-inch inside diameter stainless steel pipe. A baffle, diffusion pump, and mechanical pump would complete the system. This system would require a larger diffusion pump, say a CVC type MCF-300. The end portion of the effusion chamber section could have a flat bottom upon which the cell holder would rest; this will be discussed further in the next section. Nickel-plating both the inside and outside of the effusion chamber and "tee" would enhance their degassing characteristics and help to prevent corrosion. Use of the baffle would be optional; experience by others in this laboratory indicate sufficiently improved performance of similar systems when the baffle is removed to warrant its omission.

A constant temperature oven similar to one described in Chapter II could be used; introduction of the effusion chamber into the oven could be accomplished through an appropriate hole cut in the oven door. The neoprene O-ring seal between the "tee" and the effusion chamber section should be protected by cooling coils.

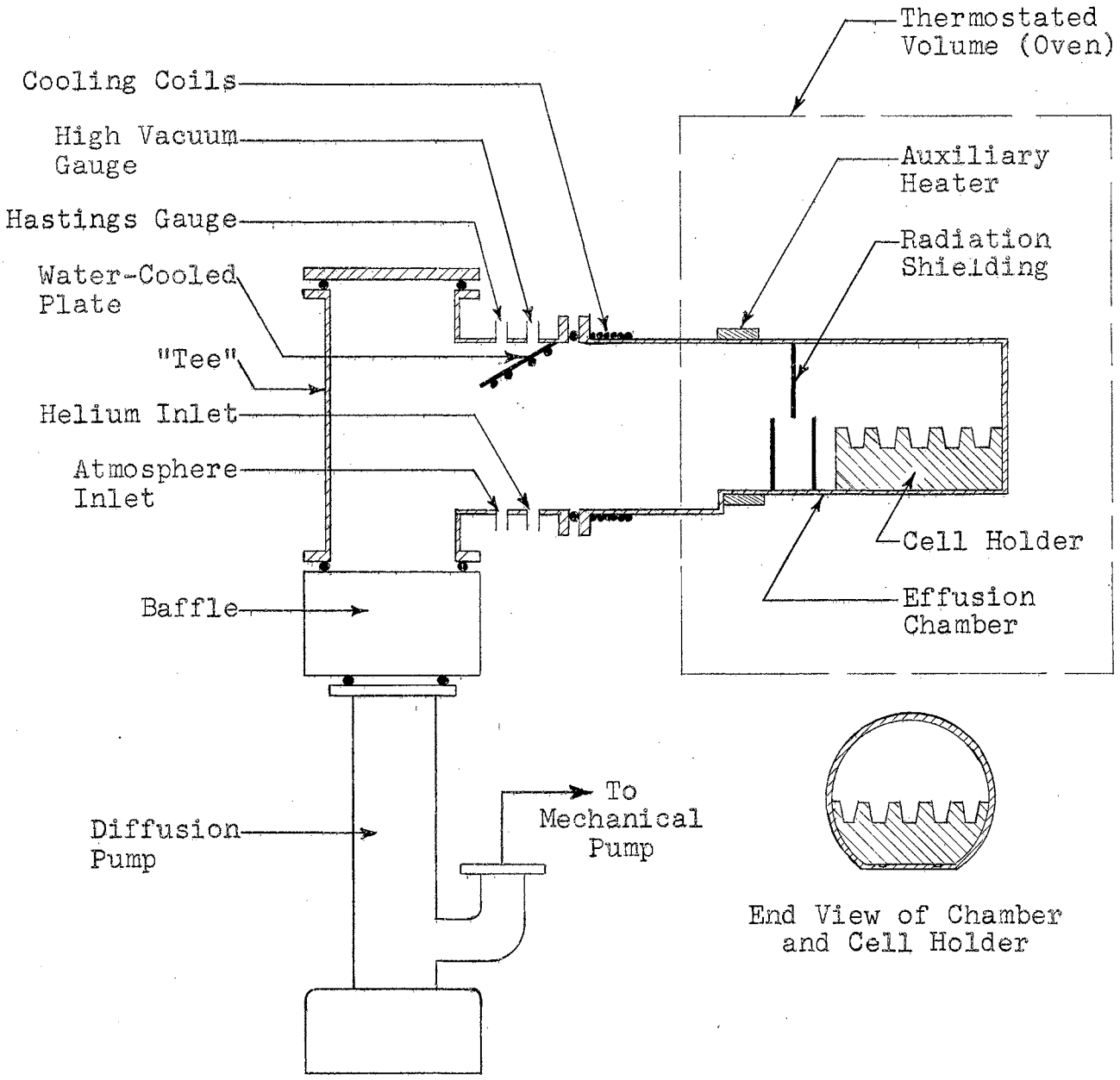


Figure 12. Modified Vacuum System

A liquid nitrogen trap could be introduced through the top plate of the "tee" if it is desired. Such a trap should probably be used if the baffle is omitted; some precaution should be taken to condense as much of the effusing material as possible before it reaches the diffusion pump.

Freeman (11) has suggested that provision should be made for radiation shielding between the cells and the oven door. Of course, this shielding must be removable to permit the insertion and removal of the cell holder. An auxiliary heater of some type should be placed between the radiation shielding and the oven door to prevent heat loss from the chamber area by conduction down the pipe; the heater could be automatically controlled by a differential thermocouple with one junction near the closed end of the chamber and the other junction in the vicinity of the radiation shielding.

The vacuum gauge sensing tubes could be installed at any convenient place on the horizontal arm of the "tee". Locating them there, rather than on the vertical arm, would provide better protection from contamination by any pump fluid vapor which might find its way past the baffle. It might be necessary to move the high vacuum gauge tube to an "ell"-shaped arm as described on page 12 to protect it from the effusing species. Sufficient protection against this contamination might be achieved with a water-cooled plate in a position similar to that indicated in Figure 12; such a plate could likely be designed so that advantages gained by condensing the effusing

vapor would outweigh the adverse effect on the pumping speed.

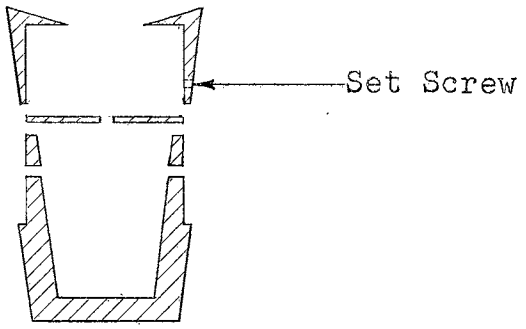
The Cells and Cell Holder

It is felt that no major modification of the effusion cells is necessary; however, several improvements could be made.

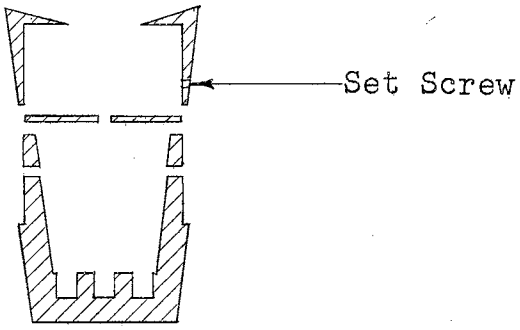
Edwards (9) has suggested that thermal contact between the cells and the cell holder could be improved if the outside of the cells were slightly tapered as shown in Figure 13-a. If the inside wall of the cell were tapered at the same angle as the outside, the resulting uniform wall thickness might effect better thermal equilibrium inside the cell. A thermocouple well in the bottom of each cell would permit an independent determination of each cell's temperature; this point will be discussed subsequently.

It has also been suggested (9) that if a substance with low heat conductivity is to be used as the evaporant, precaution should be taken to minimize thermal gradients within the sample. One way to do this would be to drill small holes in the bottom of the cell cavity as illustrated in Figure 13-b; when the cell was charged, there would be small "pillars" of the cell material protruding into the sample mass.

The cell holder should be modified, too. This author feels that it should be fabricated of copper rather than aluminum because the thermal expansivity of copper is less than that of aluminum and its thermal conductivity is greater than that of aluminum. This change would result in improved thermal contact



(a)



(b)

SCALE: 1" = 1"

Figure 13. Modified Knudsen Effusion Cells

between the cells and the cell holder, and would reduce the possibility of thermal gradients in the holder.

Another suggestion is to modify the cell holder to accommodate more cells. This could be done conveniently with a design similar to the one shown in Figure 14. The holder could have any convenient length, and the width of its top surface would be either six or eight inches, depending on the diameter of the effusion chamber. A cell holder with six-inch-square top surface would accommodate sixteen 1.0-in. diameter cells; one with an eight-inch-square top surface could easily hold twenty-five such cells. Having a greater number of effusion cells would afford several advantages: (1) more experimental points to which to fit a curve would be obtained; (2) a greater range of L/D ratios could be covered adequately in each run; (3) a number of experimental points could be grouped at both ends of the range of L/D ratios examined on a given run, thus eliminating, somewhat, the uncertainty associated with the experimental "end-points".

The author feels that it would be advantageous to have a thermocouple at each cell site if this could be accomplished without allowing thermal gradients to develop in the holder at operating temperatures. Monitoring the temperature of each cell individually would enable one to be more certain of the cell temperature than if the temperature of only the cell holder were measured; the temperature of the cells must be accurately known in an investigation of the dependency of the

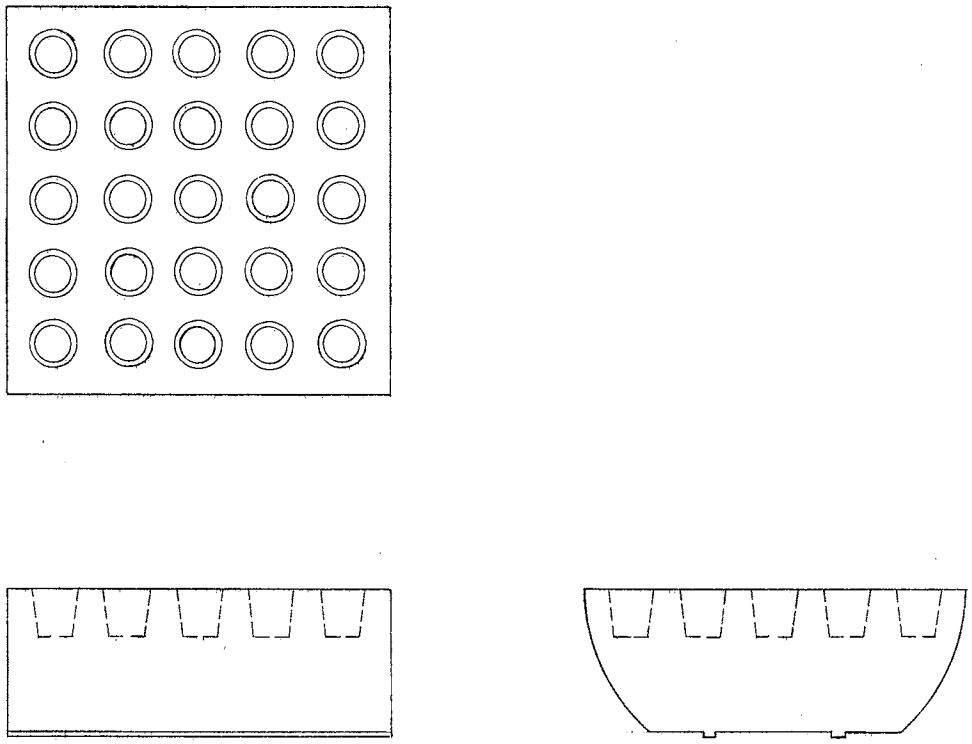


Figure 14. Modified Cell Holder

transmission probability on source pressure. Also, the anomalous behavior of any cell might be explained if its temperature, relative to the temperature of the other cells, were known. At least two, preferably four, thermocouple wells should be provided in the body of the cell holder to test for thermal gradients in the holder itself.

Openings from the bottom of each cell well to the top surface of the cell holder should be provided, as described on page 17, to facilitate removal of the cells from the holder.

Figure 12 shows how the cell holder would fit into the effusion chamber. Shallow slots in the flat bottom of the chamber would locate the holder. The flat bottom would provide good thermal contact with the cell holder even if the difference in thermal expansion of the metals causes loss of contact on the sides.

Alternate Modifications

There are a number of other modifications which could be made in place of, or in addition to, those suggested in the preceding sections of this chapter. Several of these changes which the author feels should be considered are discussed in this section.

One modification particularly worth considering is to provide valves in the vacuum system so that the diffusion pump can be kept in continuous operation, but can be isolated from the remainder of the system when so desired. This valving arrangement, patterned after that described by Heydman (14), would

involve no change in the basic design of the apparatus suggested in Figure 12. With this arrangement, illustrated schematically in Figure 15, the system could be evacuated much more rapidly at the beginning of the effusion period; the present arrangement, and that in Figure 12, requires appreciable time for the diffusion pump to "warm up" before it becomes effective.

Serious consideration should also be given to using another substance as the evaporant. With the problem of obtaining a satisfactory demountable vacuum seal inside the oven eliminated, a sample which requires a higher operating temperature might be used. Also, another search should be made for a material which could be used at lower temperatures; mercury would appear to be quite satisfactory if it could be kept free of contamination, and if the vacuum gauge sensing tubes and other parts of the system could be sufficiently protected from the mercury vapor.

Operating at a lower temperature, as just suggested, would offer at least one important advantage: if the temperature were low enough to permit a copper-to-stainless steel joint to be made, the portion of the effusion chamber in contact with the cell holder could be made of copper; this should afford optimum thermal contact between the chamber walls and the copper cell holder. For that matter, the entire length of pipe which comprises the effusion chamber section could be made of copper if thermal conduction away from the chamber area could be ade-

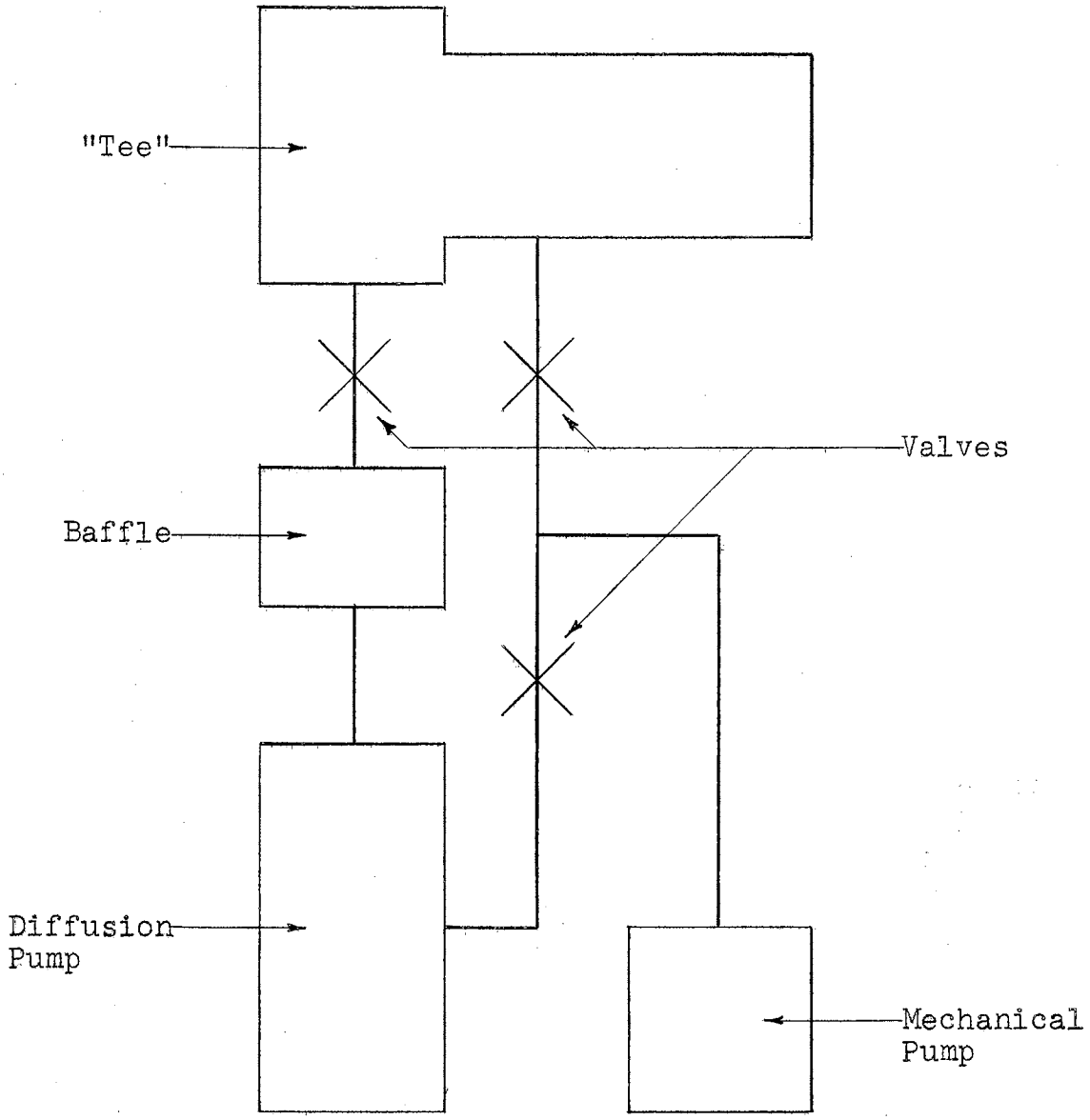


Figure 15: System with Valves to Isolate Diffusion Pump

quately overcome with the auxiliary heater; the necessity of making a copper-to-stainless steel joint would thus be eliminated.

An alternate design of the modified effusion chamber is shown, without regard to detail, in Figure 16; the change here is the provision for a depression in which a cylindrical cell holder would rest with a tight fit. The advantage of this design, as opposed to that in Figure 12, is that the bottom and all sides of the holder are heated by conduction through the chamber walls; the more uniform heating might help to eliminate thermal gradients in the holder. This author believes, however, that no thermal gradients of any consequence will develop in the cell holder with a chamber designed as in Figure 12 if the holder is constructed of copper. Also, the design in Figure 16 would make it difficult to provide for all the thermocouples suggested earlier in this chapter. These two reasons and the greater ease with which the holder may be introduced into a chamber of the former design influence the author to favor, slightly, that design.

A change of comparatively minor importance, but one which might be helpful, would be to employ smaller effusion cells of the modified design suggested earlier. This would allow the use of more cells in the cell holder or the reduction in size of the holder and effusion chamber, whichever was preferred.

Also, another method of heating might be investigated. Perhaps some type of resistance heating of the effusion cham-

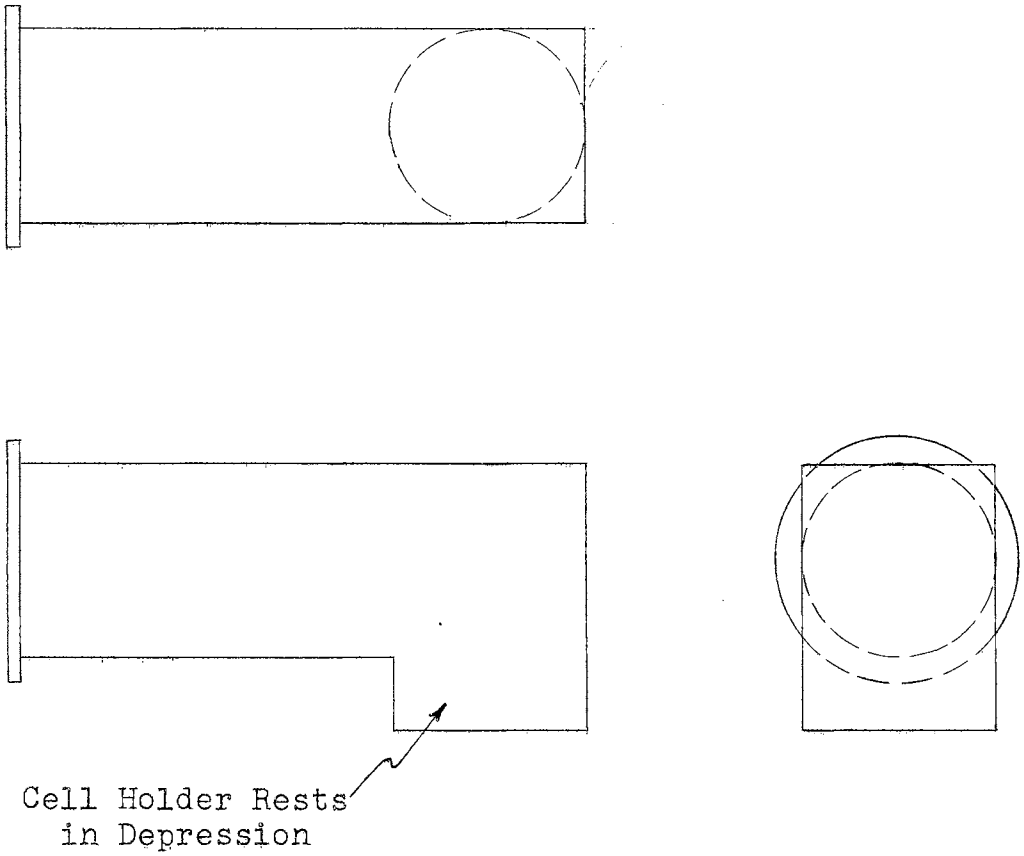


Figure 16. Alternate Modified Effusion Chamber

ber would provide more precise temperature control than a "constant-temperature" air bath.

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VITA

Alvin Leroy Ball, Jr.

Candidate for the Degree of

Master of Science

Thesis: EXPERIMENTAL DETERMINATION OF TRANSMISSION PROBABILITIES FOR MOLECULAR FLOW THROUGH ORIFICES

Major Field: Chemistry

Biographical:

Personal Data: Born at Alva, Oklahoma, August 20, 1937, the son of Alvin L. and Martha A. Ball, Sr.; married Ruth Ann Halacka in El Reno, Oklahoma, August 5, 1962.

Education: Attended the public schools of Alva, Oklahoma, and Mesa, Arizona; graduated from Alva High School in May, 1955; received the Bachelor of Science degree from Northwestern State College, Alva, Oklahoma, in May, 1959, with a major in chemistry and physics; completed the requirements for the Master of Science degree in May, 1963.