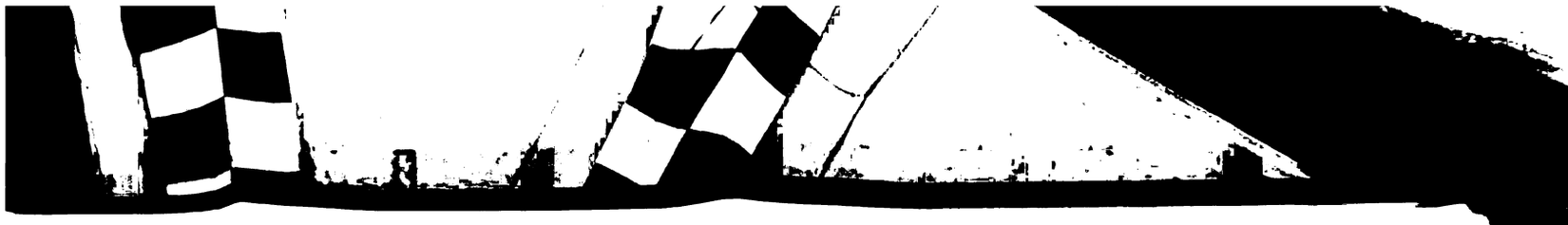
GASEOUS DIFFUSION OF DIBORANE, HYDROGEN, AND NITROGEN

THESIS FOR THE DEGREE OF PH. D.
MICHIGAN STATE COLLEGE

RALPH E. JOHNSON
1955



GASEOUS DIFFUSION OF DIBORANE, HYDROGEN, AND NITROGEN

By

RALPH E. JOHNSON

A THESIS

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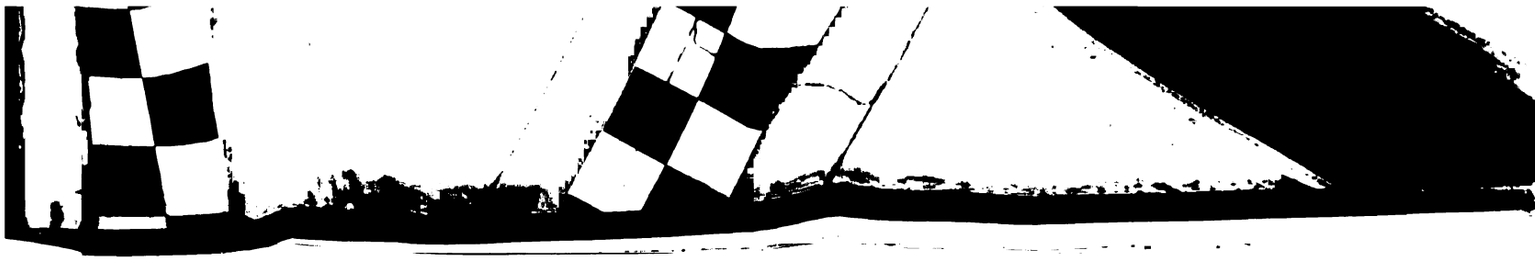
ABSTRACT

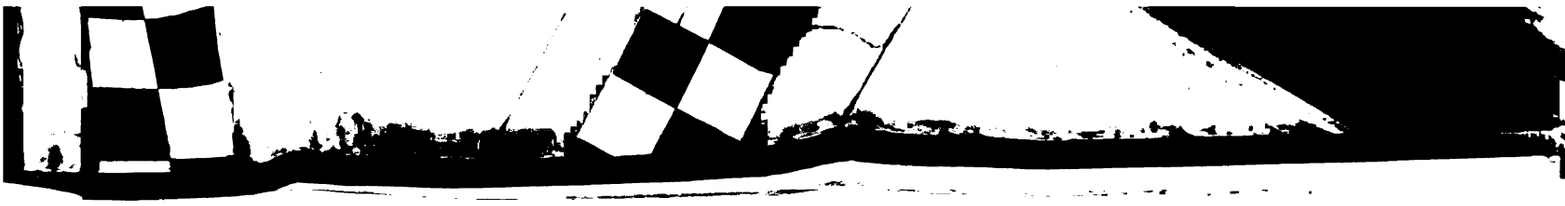
The problem consisted in effecting a separation of diborane from hydrogen and nitrogen. The method used was one involving gaseous diffusion. The system which was adapted to this problem was one that was used by Thon (19). The system was altered slightly in order to render it suitable for the study of binary mixtures. The gases were diffused through a solid barrier from the right side of the system which was at a fixed overpressure into the left side of the system which had been evacuated to a pressure of 10^{-5} mm. of Hg. The barriers used were electrodeposited nickel foils with thicknesses which varied down to 0.0001 inch, commercially obtained plastic films, and brass shim stock from which the zinc had been distilled. The thickness of the brass varied down to 0.001 inch.

The nickel foils were plated from a Watt's bath of a pH of 2.2 at 40 amps per square foot and 55°C. They were plated on a bright, buffed nickel surface. Before plating the foil, the nickel base panel was passivated by anodic electrocleaning at 100 amps per square foot and 80°C. This treatment prevented the foil from adhering tightly to the base panel. The commercial plastics used



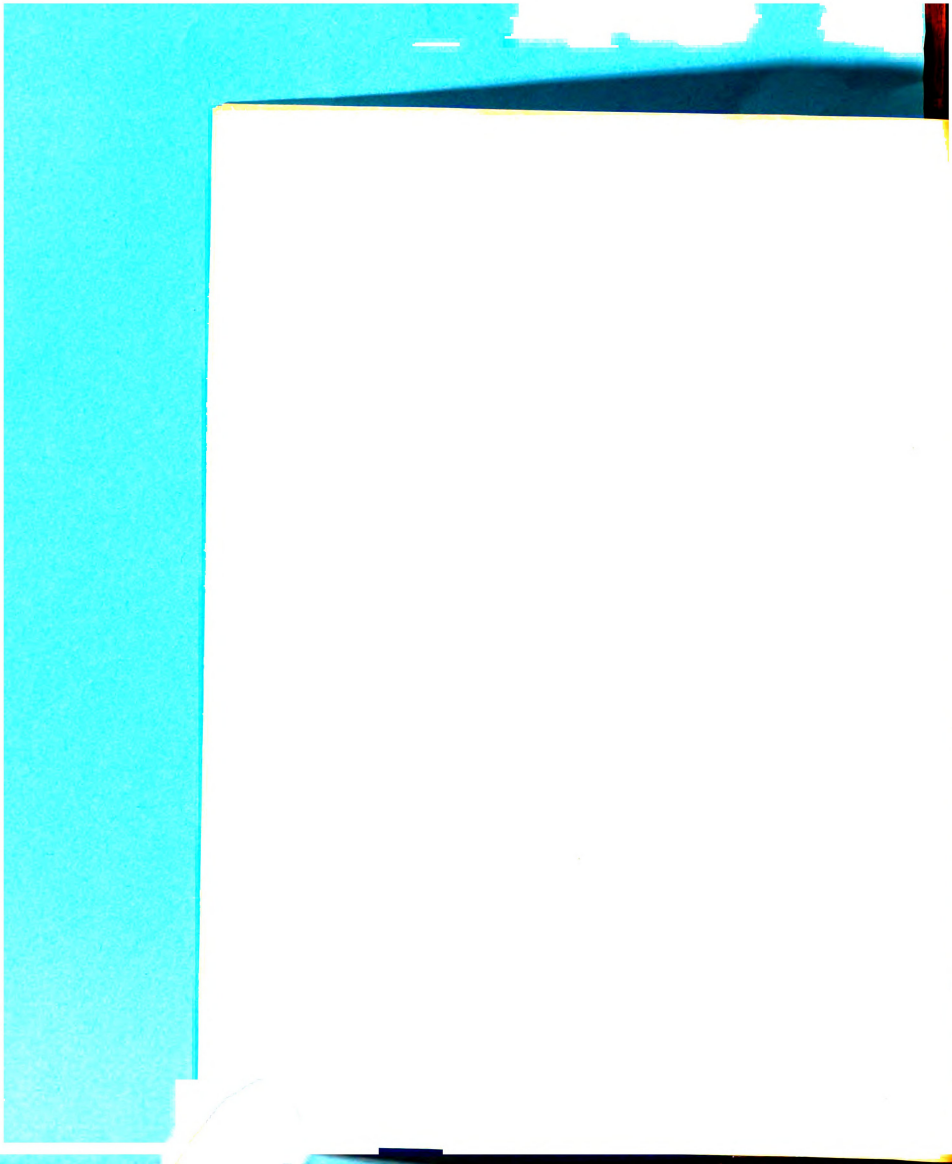
were Trithene, Visqueen, Bakelite, Tenite Butyrate, Tenite Acetate, and Kodapak F122. The zinc was distilled out of the brass in a furnace in a vycor tube. The pressure in the tube was maintained at a very low value during the distillation.

The results show that for the diffusion of a gas through a barrier, the pressure increase on the left side of the system is a linear function of the time and the rate of diffusion for the metal foils is a linear function of the overpressure. Diffusion through the metal foils follows Graham's law, whereas the diffusion through plastic foils does not. Using the nickel foils, an attempt was made to separate diborane from nitrogen and from hydrogen. The mixtures of diborane and hydrogen showed a marked increase in the concentration of hydrogen after diffusing. The mixture of diborane and nitrogen was not measurably separated. This was due to the fact that the two gases have almost identical rates of diffusion, and corresponds to the fact that they have almost identical molecular weights. Individual rates showed that a separation is possible if sufficient passes through the barrier could be made. The rate constants were determined for all of the foils for each gas. The constants were of the order of 10^{-6} cm./min. A graph was constructed which showed the efficiency of separation as a function of porosity of the foil.



ACKNOWLEDGMENTS

The author wishes to express his sincere appreciation for the direction and assistance given by the late Dr. D. T. Ewing during the course of the research and the subsequent guidance and understanding of Dr. L. L. Quill, who directed the completion of the research and the preparation of this manuscript.



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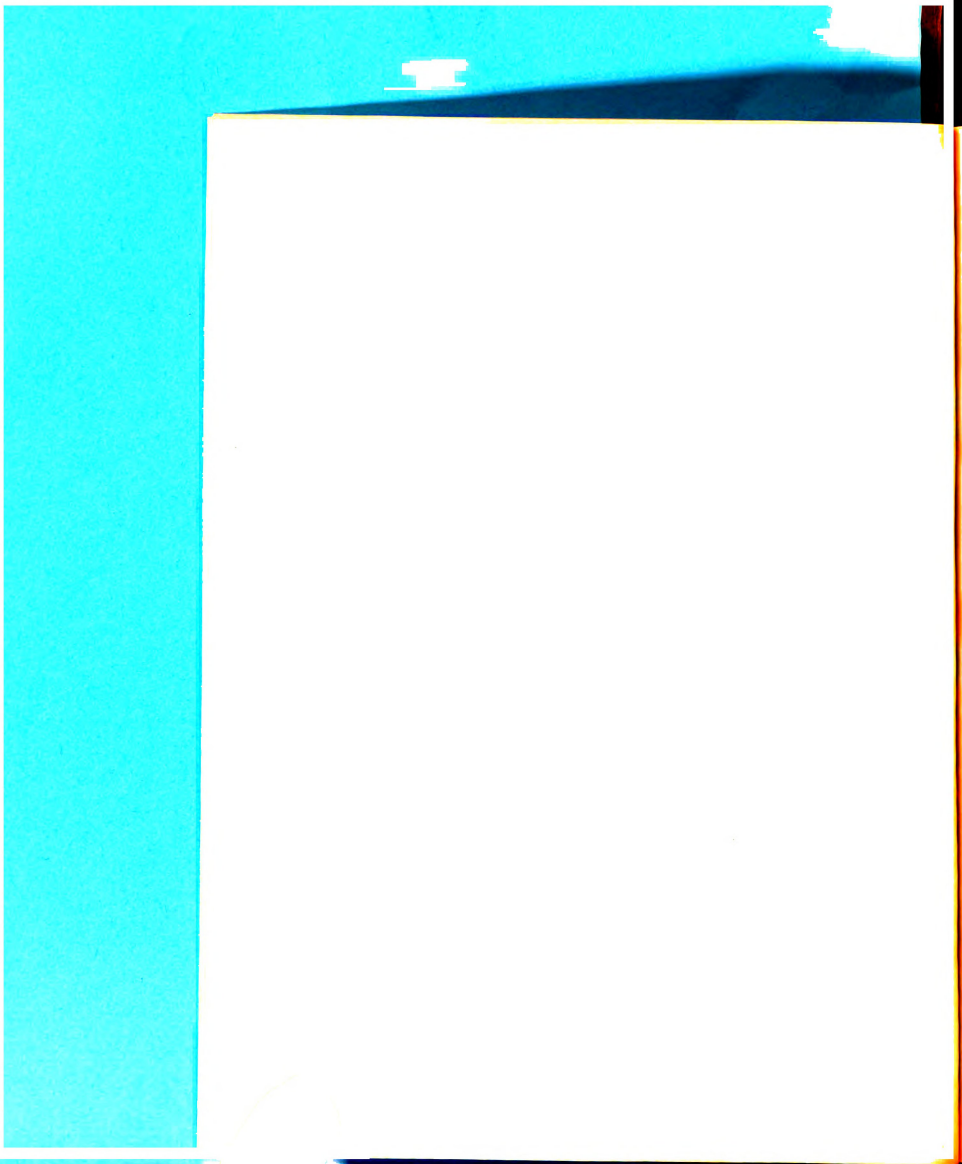

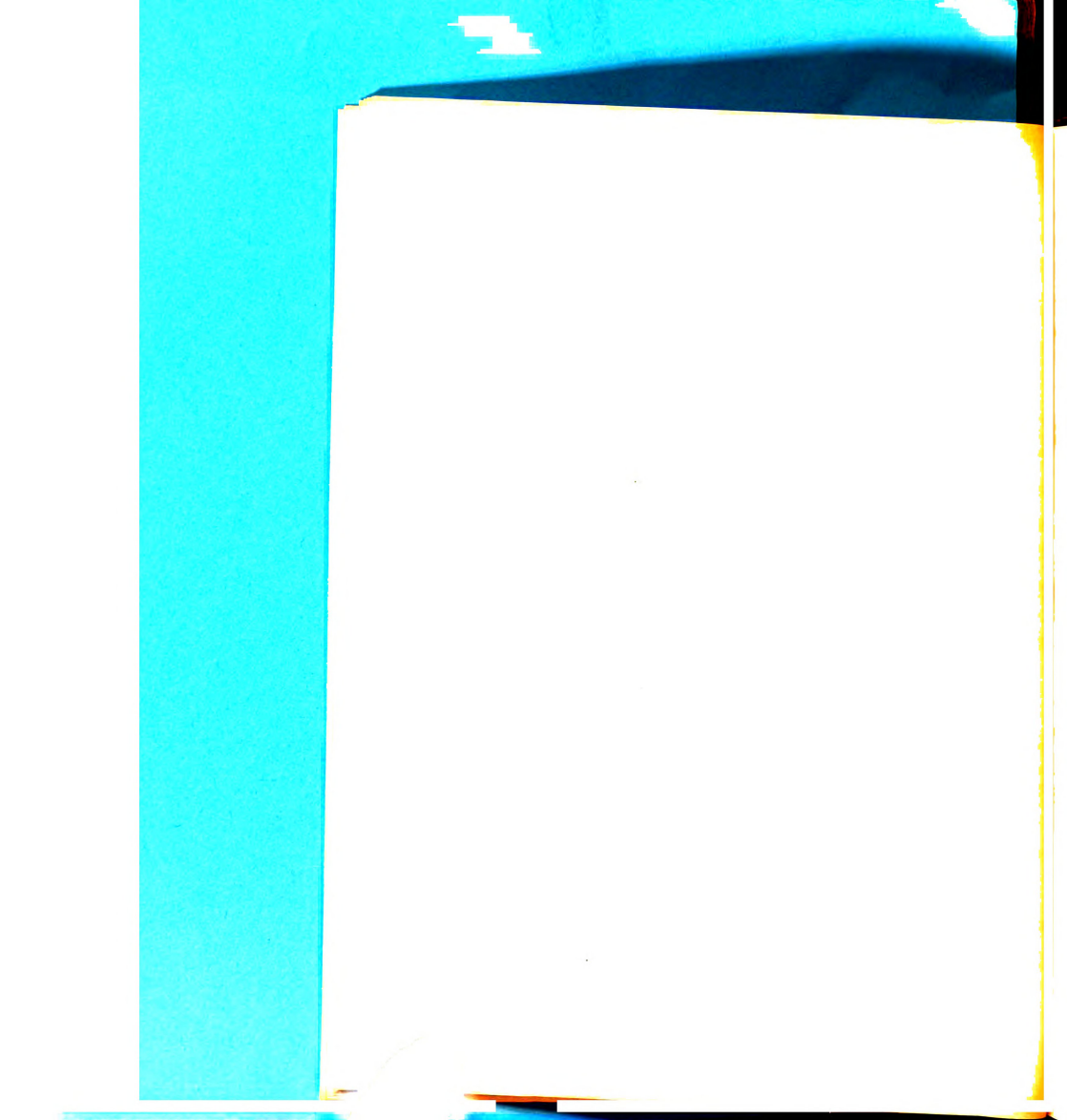

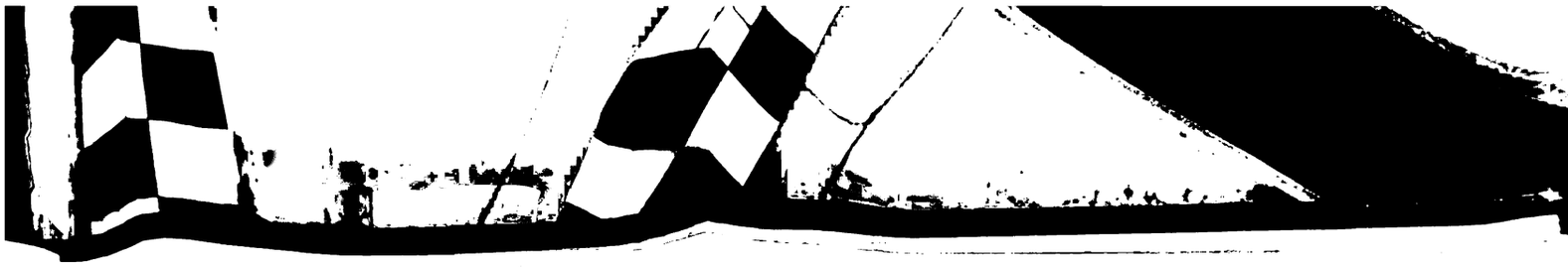

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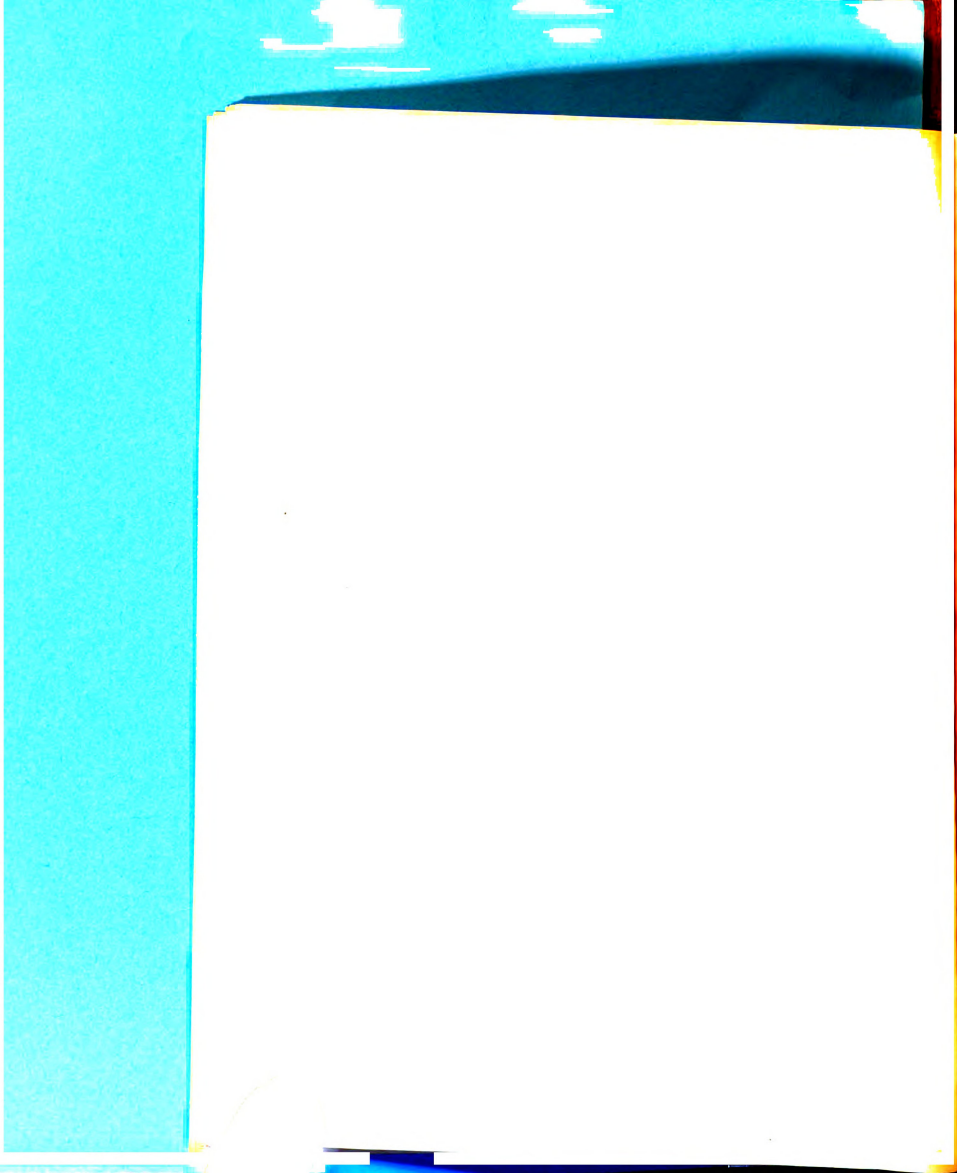


INTRODUCTION

In the laboratory preparation of diborane, the product invariably contains relatively large amounts of hydrogen and nitrogen. The hydrogen occurs as a reaction product, whereas the nitrogen is present by virtue of its use as an inert atmosphere preventing the spontaneous combustion of diborane. Many uses of diborane require that the product be pure. The most obvious method of purification is to freeze out the diborane with liquid nitrogen. This method, however, is quite expensive, and the need for a more economical method becomes evident. The method which suggested itself was that of diffusion (7), and thermal diffusion (17) was excluded immediately because of the effect of heat on diborane. To use low-temperature thermal diffusion would again involve the expense of cooling. The alternative was to diffuse through a barrier (12) of some kind.

The problem which was undertaken, then, involved a separation of diborane from hydrogen and nitrogen. The separation was expected to result by virtue of the differences in the rates of diffusion of the gases. It was decided to study only binary systems since analysis of the three-component system proved to be virtually impossible.

Also, the binary systems would yield results which were as valid and as effective in showing the nature of the separation as the three-component system. It then became necessary to choose a diffusion barrier and a diffusion system. In this instance, the background and experience of Dr. D. T. Ewing became a deciding factor. In accordance with this background, electrolytic nickel foils (18) were used as barriers, and a high-vacuum system designed to study the porosity of electrodeposits was adapted. Alterations in the system were necessary to render the system effective for the handling of binary mixtures. Later, preliminary studies were made on other barriers such as brass from which the zinc had been distilled, and plastics. The references given at the end of this work deal with the development of this technique, the nature of plating strip deposits, and the method used to distill zinc from brass.

An attempt is made in this report to present the material in a reasonably logical sequence. A historical introductory chapter is concluded by a theoretical section in which the mathematical considerations are treated. Attention is given only to the mathematics involved in rates of diffusion and not that involved in the mechanism (2) of diffusion. This study is not concerned with the mechanism of diffusion, but only with separations which are dependent on the

rate of diffusion. The experimental procedure is then given along with a description of the apparatus. A suitable drawing of the apparatus is included with the description to render the discussion more intelligible. The next chapter lists the results which were obtained. Included with the results are all of the calculations and graphs. The paper is then completed by a discussion and conclusions. As is the case with most research problems, much work remains undone. Many new avenues of approach have been opened by this study. In spite of that which remains to be done, the original purpose set forth in this study has been rather satisfactorily fulfilled. It remains for those who follow to carry on the work.

HISTORICAL

In the initial search for barriers through which to diffuse gases for the purpose of separating one gas from another, it was natural to look to the plating industry for metal barriers and for methods of determining their porosity. The apparatus and procedure (19) used in this work was developed by the American Electroplater's Society in order to test the corrosion-resistant properties of electrodeposits. It was assumed that the resistance to corrosion is a function of the porosity of the deposit. The original work on porosity of electrodeposits was done by Thon and his associates at Princeton University in 1949.

The apparatus (20) which was finally used by Thon was a high-vacuum system which had two identical sides. The two sides were separated by a cell which held the foil through which the gases were to be passed. Also connecting the two sides was a glass tube with stopcocks provided for isolating either side. Joined to this tube was the exhaust system consisting of a mercury-diffusion pump and a mechanical pump. Another tube connected at this point allowed the introduction of gases into either side of the apparatus. Each side also contained a McLeod gage for measuring the pressure on each

side. Later, manometers were added to each side in order to allow high-pressure measurements (9). The foil holder was built so that the foil was held between the two halves of the metal holder. Thus the foil was supported by metal and held in place by pulling the two halves of the holder together with screws.

Originally, the low-pressure gas permeability method (19) was used. This consisted in placing gas at 0.1 mm. of mercury pressure on one side of the foil, a vacuum on the other side, and observing the pressure changes on both sides as a function of time. This method was shown to be reproducible within the experimental error on the same sample as well as on different samples from the same foil. Also by this method, the variation of intrinsic porosity with thickness of deposit was clearly shown, as well as the fact that electrodeposited metals actually have an intrinsic structural porosity.

The low-pressure gas permeability method was discarded in favor of the more versatile and accurate low-pressure constant overpressure method. In this case, one side of the apparatus was open to the atmosphere, whereas the other side had been evacuated. The pressure in the side which was open to the atmosphere effectively remained constant during a diffusion study. Later a manometer

was added to each side so that any gas could be subjected to diffusion studies at any overpressure. By this method, a relationship between porosity and crystal structure was shown to exist (20). Foils were then subjected to various corrosive atmospheres and the increase in permeability was noted (21). The effect of surface structure of the base metal on the permeability and corrodability was then studied (22). Other corrosion rate studies were carried on by Thon (23) and his associates. The effect of brighteners (24) in nickel-plating baths was then studied. Diffusion studies on electrolytic nickel as a measure of resistance to corrosion were continued by R. Fay (9) at Michigan State College.

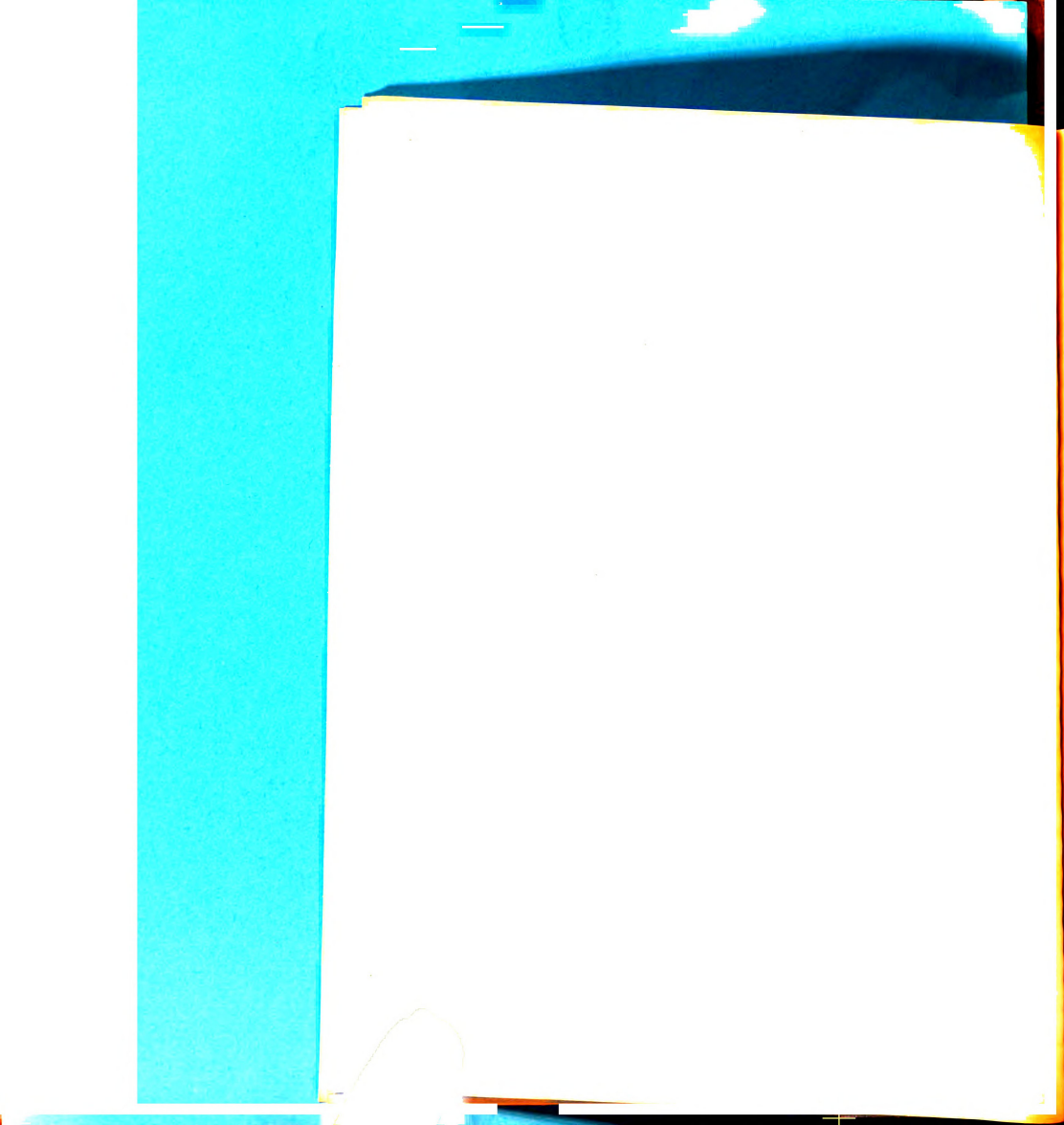
Theory (20)

The low-pressure constant overpressure method was suggested by an equation in the theoretical treatment of the low-pressure gas permeability method. The latter method is based on the equalization of pressure on the two sides of the system. The basic equation for the equalization of pressure is

$$\frac{-d\Delta p}{dt} = \frac{A}{V/2} \times k \times \Delta p \qquad (1)$$

where

Δp is the pressure difference at time t



A is the area of the foil exposed to gas

V is the volume equal on both sides

Now

$$\Delta p = P - p$$

where

P is the pressure on the high side

p is the pressure on the low side

Thus

$$\frac{-d(P-p)}{dt} = \frac{A}{V/2} \times k \times (P-p) \qquad (2)$$

$$\frac{-dP + dp}{dt} = \frac{A}{V/2} \times k \times (P-p) \qquad (3)$$

Since

$$P = P_0 - p$$

where

P_0 is the initial pressure on the high-pressure side

p is the pressure on the low-pressure side

$$\frac{-d(P_0 - p) + dp}{dt} = \frac{A}{V/2} \times k \times (P-p) \qquad (4)$$

Since P_0 is maintained constant

$$\frac{+2dp}{dt} = \frac{A}{V/2} \times k \times (P-p) \qquad (5)$$

where p is very small

$$(P-p) = P_0$$

Thus

$$dp/dt = (A/V) \times k \times P_0 \qquad (6)$$

Integrating

$$p = (A/V) \times k \times P_0 \times t \qquad (7)$$

Equation (6) expresses rate as a function of the overpressure. Equation (7) relates pressure on the low-pressure side at any given time, t, to the overpressure and the foil and apparatus constants. These are the basic equations on which diffusion studies by the low-pressure constant overpressure method are based. Equation (7) can also be obtained directly from equation (1):

$$\frac{-d\Delta p}{dt} = \frac{A}{V/2} \times k \times \Delta p \qquad \text{or,} \qquad (1)$$

$$\frac{-d(P-p)}{dt} = \frac{A}{V/2} \times k \times (P-p) \qquad (2)$$

$$\frac{-d(P-p)}{(P-p)} = \frac{A}{V/2} \times k \times dt \qquad (8)$$

Integrating

$$-\ell n\frac{(P-p)}{(P-p)_0} = \frac{2A}{V} \times k \times t \qquad (9)$$

$$-\ell n\frac{P_0-2p}{P_0} = \frac{2A}{V} \times k \times t \qquad (10)$$

$$-\ell n\left[1 - \frac{2p}{P_0}\right] = \frac{2A}{V} \times k \times t \qquad (11)$$

Using the approximation formula

$$\ell n \frac{P_0}{P_0 - p} = \frac{2p}{P_0} \tag{12}$$

The equation becomes

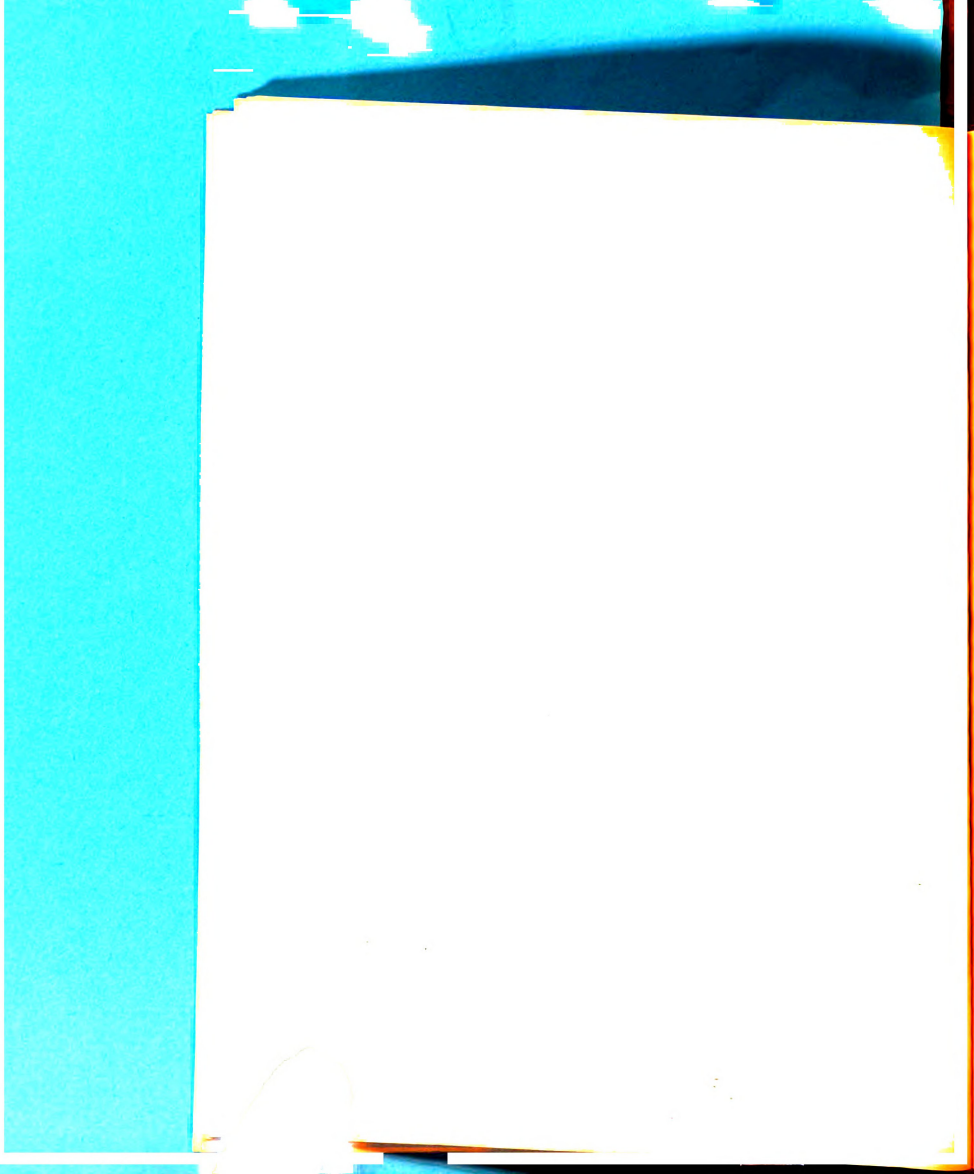
$$\frac{2p}{P_0} = \frac{2A}{V} \times k \times t \tag{13}$$

or, solving for p,

$$p = (A/V) \times k \times P_0 \times t \tag{14}$$

which is the same as equation (7). From these equations, it can be seen that the necessary data to determine the diffusion rate constant are the McLeod pressure, p, the overpressure, P_0, and the time, t. The area of the foil, A, and the volume of the system, V, are empirical constants which were measured. In calculating the results, equation (6) was used. Since the same apparatus was used in all determinations, the area-volume relationship, A/V, was always constant, but was not included in k since k is the permeability constant which is characteristic of the foil but not the apparatus.

The mathematical treatment of the mechanism (2) by which the gas proceeds through a barrier has not been given, however. In this study, the nature of the gas is of considerable importance since the molecular weight is a primary factor in effecting a separation. Accordingly, the relationship known as Graham's Law, that



the rate of diffusion is inversely proportional to the square root of the molecular weight, is applicable for this study.

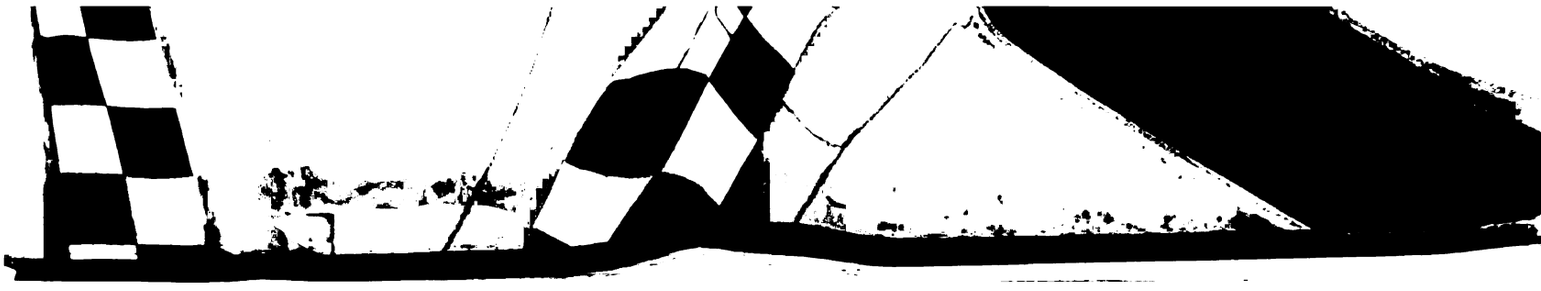
EXPERIMENTAL

Since three kinds of films--electrodeposited nickel, porous copper, and plastic--were used as diffusion barriers, one step in the experimental procedure involved the preparation of the electrodeposited foils. For this part of the study, nickel was deposited from a special nickel-plating solution on a carefully conditioned nickel-plated brass panel, and then stripped from the panel. For this operation, two different Watt's nickel-plating baths and one alkaline electrocleaning solution were necessary. One of the Watt's baths was used in the preparation of the base panel and the other for the deposition of the nickel films.

The Watt's dull nickel-plating solutions (14) used have the following composition:

$NiSO_4 \cdot 6H_2O$	240 g/l.
$NiCl_2 \cdot 6H_2O$	45 g/l.
H_3BO_3	30 g/l.

The chloride (1) in the bath is used to increase the corrosion of the anode and the boric acid (25) is present to act as a buffer and to cause a degree of smoothness in the deposit.



The pH was raised to about 5.2 by adding solid nickel carbonate and allowing the mixture to stand overnight. Activated charcoal was then added, and the solution was heated for about fifteen minutes at about 80°C. The solution was filtered several times to insure that all of the solid nickel carbonate and charcoal had been removed. For electroplating the base panel, the bath was then ready for plating at a pH of 5.2. For depositing a nickel foil, the pH of a Watt's bath of pH of 5.2 was lowered to 2.2 by adding sulfuric acid. The solution was then brought back to the original volume by adding water to replace that which had evaporated. The solution was then ready for use as a plating bath. Standard operating conditions (14) with this bath are with a current density of 40 amps per square foot of cathode area, and at 55°C.

To prepare a base panel on which the nickel foil was to be plated, brass shim stock was used. The thickness, which was not critical, of the brass base panel was about 0.0015 inch. The brass panel was highly buffed and then electrocleaned in the following electrocleaning (5) solution at 100 amps per square foot and about 80°C.

NaOH	21 g/l.
$Na_2Si_2O_3$	15 g/l.

Na_3PO_4 . . . . . 18 g/l.

Na_2CO_3 . . . . . 6 g/l.

$NaC_2H_3O_2$ . . . . . 7.5 g.l.

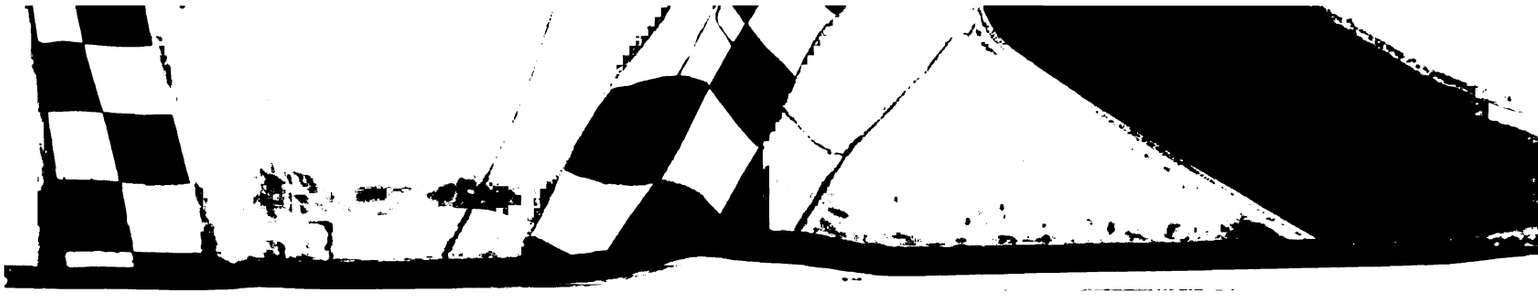
In the cleaning process, the brass panel was made the cathode and a nickel panel was made the anode. The current was allowed to pass for twenty seconds. The panel was immersed in 20 percent sulfuric acid for a period of twenty seconds. It was then rinsed off with distilled water. If the water film broke anywhere on the surface which was to be plated, the cleaning had to be repeated because a nickel plate will not adhere under these conditions.

The clean brass panel was then plated for about ten or fifteen minutes at 40 amps per square foot and 55°C. in the Watt's nickel bath with a pH of 5.2 to obtain a very smooth, dull deposit. This deposit on the entire panel was then buffed to a very high finish. In this process, it was imperative that there be no flaws on the surface after the buffing since flaws would always occur at the same points on the nickel foil plated on this buffed nickel.

The next step after preparing the nickel-plated panels was the plating of the foil. The buffed nickel-plated base panel was placed in the electrocleaner as the anode, with another nickel panel as the cathode. At the anode, oxygen is liberated, and causes a thin

layer of oxide to form over the buffed nickel surface. When nickel is subsequently plated on such a surface it will adhere only very loosely or not at all. Thus the foil can be lifted off the base metal with little or no difficulty. In case there is no adherence on the passive surface, the adhesion occurs around the back of the panel, holding the plate on until it is detached. To render the base metal passive, the panel was cleaned anodically for twenty seconds, treated with a 20 percent acid dip for twenty seconds, recleaned anodically for twenty seconds, and then subjected to a final twenty-second acid dip. The panel was then placed in the plating bath and plated. At the conclusion of the plating time, the panel was removed and dried. The electrodeposited foil was cut with a razor blade along all four edges, lifted off the base panel, and stored in a vacuum desiccator.

In the actual plating process, the prepared base panel is made the cathode, and a bar of rolled nickel the anode. Since the degree of agitation is uncontrollable, at least with respect to work done by other investigators, there was no agitation of the solution in this work. Whenever a bubble was observed to form on the surface, it was removed by mechanically tapping the panel support. Agitation was not deemed advisable since the effect of dirt or anode sludge adhering to the deposit increases porosity beyond reason or



even causes visible pores. The anode sludge is a basic nickel salt and can be controlled by placing a bag around the anode to retain the sludge in the bag and prevent it from contaminating the solution. The anode bags were made out of a heavy duck material and were treated with acid and sodium carbonate to remove all of the materials which would otherwise be extracted by the plating solution.

The designation given to the foils had the following significance: in the case of the foil, 0M4, the zero signifies that it was plated in a Watt's bath of a pH of 2.2 at forty amps per square foot of area and at 55°C., the M indicates that it was a dull foil plated on a bright base panel, and the 4 indicates the thickness in tenths of a thousandth of an inch. The thickness is known from the time of plating; one-tenth of a thousandth of an inch in thickness requires three minutes of plating time at the conditions given above. Five foils, ranging in thickness from 0.0001 to 0.0005 inch, were plated for this experiment. The thickest of these was ruined in that the foil-holder edge cut it. The results on the foil 0M5 were therefore discarded and are not submitted in this report.

In an effort to discover a foil which would pass the gases at a faster rate, a porous copper foil was prepared by distilling the zinc out of brass shim stock. The brass foil was placed in a vycor

tube used in an ordinary combustion furnace. The foil was therefore curved around in order to fit in the tube. The tube was sealed on one end and connected to the oil pump on the other end. After pumping overnight, the central portion of the tube was heated in the furnace to a temperature over 600°C. At this temperature (10), the zinc was distilled from the brass and condensed in cooler parts of the tube. The foil which resulted had the characteristic pink color usually associated with pure, unoxidized copper. The heating element was then turned off but the oil pump was kept running until the tube had cooled to room temperature. The foil was removed and placed either in a vacuum desiccator or directly in the foil holder.

Of the large number of commercial plastic membranes on the market, a limited number were used in this study to serve as examples of diffusion through high-polymer (3) material and to give a measure of their potentialities in a separation. The plastics which were used are Trithene and Visqueen, manufactured by the Visking Corporation; Bakelite VBA 9925 Natural, made by the Bakelite Company; and Tenite Butyrate, Tenite Acetate, and Kodapak F122, from the Tennessee Eastman Company (compositions are given on page 20).

Three gases, hydrogen, nitrogen, and diborane, were used in the diffusion studies on the three types of foils. Each gas was

diffused through each nickel foil at six or seven overpressures. Also, each foil was subjected to the diffusion of three mixtures of hydrogen and diborane and of nitrogen and diborane. The mixtures corresponded to compositions of 1:2, 1:1, and 2:1. The analysis of the final mixture after passage through the foil was accomplished by freezing out the diborane with liquid nitrogen on both sides of the system, pumping off the nitrogen or hydrogen, and allowing the left side to come back to room temperature.

The apparatus used for the diffusion studies is given in Figure 2. A foil is placed in the foil holder C and the holder is replaced in position by sealing the ball-and-socket joints, J, with apiezon W sealing wax. The surface of the foil exposed to the gases was about a five-centimeter circle. The system was then evacuated through K, which led to a mercury-diffusion pump and an oil pump (Kenny). During this process, V, D, L, and R were open, whereas B was closed. After the system had reached a pressure below 0.05 micron, L was closed and gas was introduced into the system. The hydrogen was introduced into E through H, the nitrogen was introduced into E through N, and the diborane was introduced directly into W through B. In the case of binary mixtures, all of the gas in E is forced into W by raising the mercury in E. After the gas

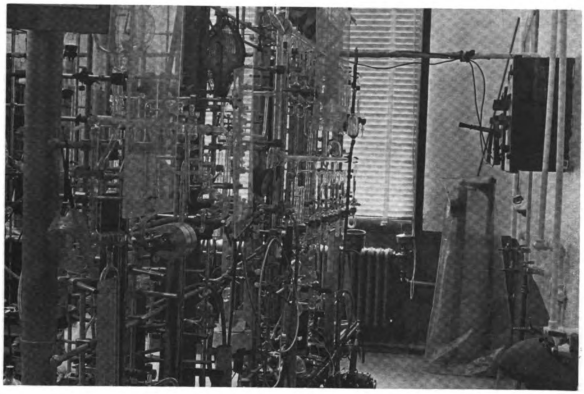


Figure 1. High-vacuum manifold.

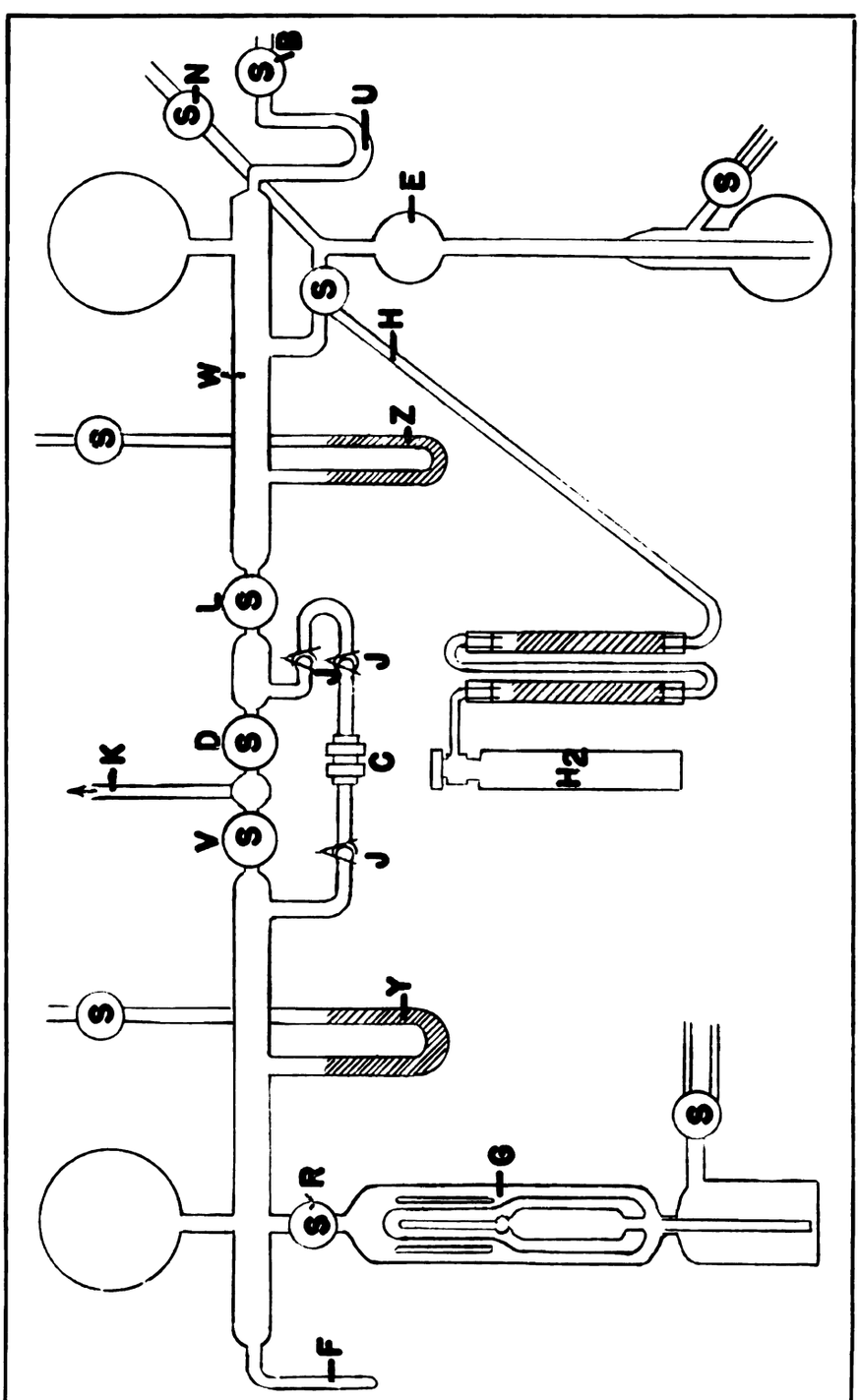


Figure 2. Gaseous diffusion apparatus.

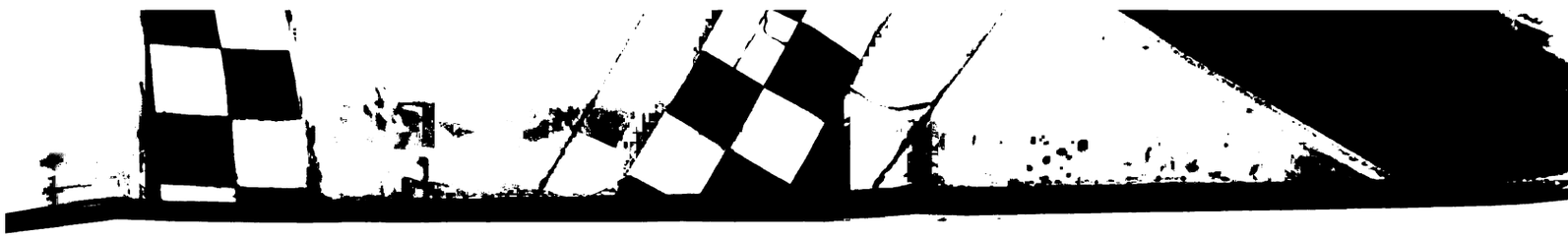
had been placed in E, it was allowed to pass into W rather slowly until the approximate overpressure desired was indicated on the manometer Z. At this point, the stopcocks A and D were closed, and L was opened. The gas was then diffusing and the pressure was followed on the McLeod gage G. In the case where the rate of diffusion was faster than the gage could follow, the pressure was followed on the manometer Y with the aid of a cathetometer. In the analysis of mixtures, the diborane was frozen out in the traps F and U.

The plastics listed on page 16 have the following chemical nature: Bakelite is a vinyl-type polymer, the tenite materials are cellulose acetate and cellulose acetate butyrate, Visqueen is a polyethylene plastic, Trithene is trifluorochloroethylene, and Kodapak is a cement for cellulosic sheets.

RESULTS

The following tables of data are given such that the experimental data are listed first, followed by the calculated data. The curves follow the data from which they were taken as closely as possible. Since all of the curves of pressure-versus-time data are similar, only two curves, Figures 3 and 5, of pressure versus time are given as samples. Each rate-versus-overpressure curve, Figures 4, 6, 7, and 8, follows the last set of data for that particular foil. Each rate constant-versus-thickness curve, Figures 9 and 10, follows the data of that particular series of foils.

In the tables of experimental data, the following notations are to be observed: P_0 is the overpressure which is constant for a run, p_0 is the initial pressure on the left side of the system, p_{30} is that pressure after closing off the system for thirty minutes with a vacuum on the other side of the foil. After the time, the pressure readings of the left side of the system are given as taken every two minutes. The pressures are in microns in all cases except that in which the pressure is followed by an asterisk (*), in which case the pressure is in millimeters of mercury. For the data on mixtures, Table IV, VIII, XII, and XVI, the gas added represents one component



of the mixture, the other component always being diborane. The bottom line of the mixture data sheet is the final pressure of diborane on the left side.

Calibration of the System

For the diffusion studies, it was necessary to calibrate the volume of the system. The volume of the McLeod gage was determined first by actually measuring the volume of mercury which would fill it. With this known volume, the volume of the remainder of the left side of the system was determined by letting gas at a known pressure into the McLeod gage, closing the stopcock between the McLeod gage and the rest of the system, evacuating the rest of the system, allowing the gas in the McLeod gage to expand into the rest of the left side of the system, and reading the new pressure. The volume was calculated with the aid of the ideal gas law.

Volume of McLeod . . . . . 425 ml.

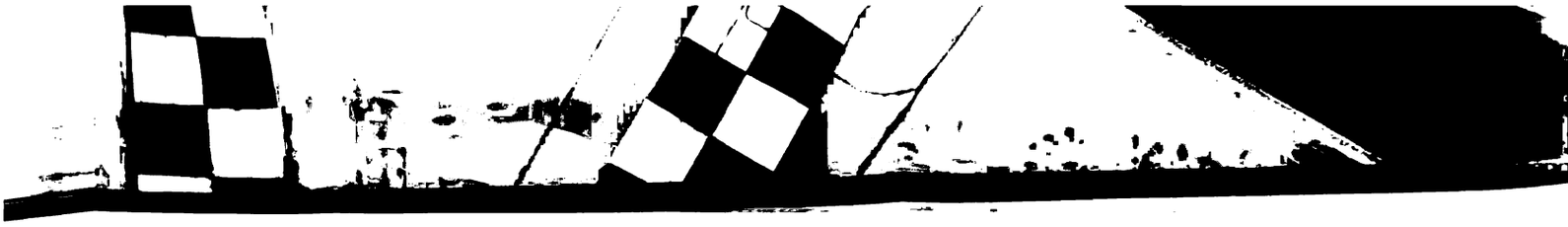
Pressure in McLeod . . . . . 737.5 mm.

Pressure left section . . . . . 209.0 mm.

$PV = P'V'$

$(737.5 \times 425)/209.0 = V'$

$V' = 1500$ ml., which is the volume of the remainder of the



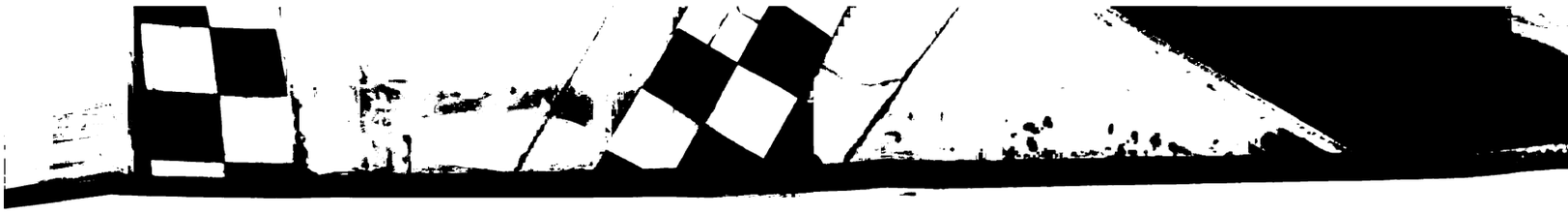
left side. The volume of the entire left side, including the McLeod gage, is 1500 + 425 = 1925. This is the volume into which the gas diffused.

In the calibration of the system for volume, only the left side of the system had to be determined, since the right side remained at constant overpressure and its volume did not affect the rate of diffusion. The calibration involved the use of the ideal gas law. Under certain conditions this law does not hold accurately, and, accordingly, the volume as determined could be inaccurate. No attempt was made to know the volume to the nearest milliliter since the experiment itself was not that accurate and the ideal gas law inaccuracy defeated the purpose of an accurate volume determination. The volume was determined to the nearest ten milliliters or so. This represents an error of only 0.5 percent. This is greater accuracy than the experimental measurements.

Permeability Constants

As indicated previously, the data necessary to calculate the rate constant are the McLeod pressure, p, the overpressure, P_0, and the time, t. To determine the rate, dp/dt, for use in equation (6),

$$\frac{dp}{dt} = \frac{A}{V} \times k \times P_0 \qquad (6)$$



the McLeod pressure, p, is plotted against the time, t, for a given overpressure, P_0; the slope is equal to the rate. Equation (6) can be used directly to determine the rate constant or it can be used graphically. For the nickel foils, the graphical method was used by plotting rate versus overpressure, P_0. The slope is equal to (A/V) x k, and since A and V are known, k can be calculated. For all other foils, since everything was known except k, this value could be calculated directly from equation (6).

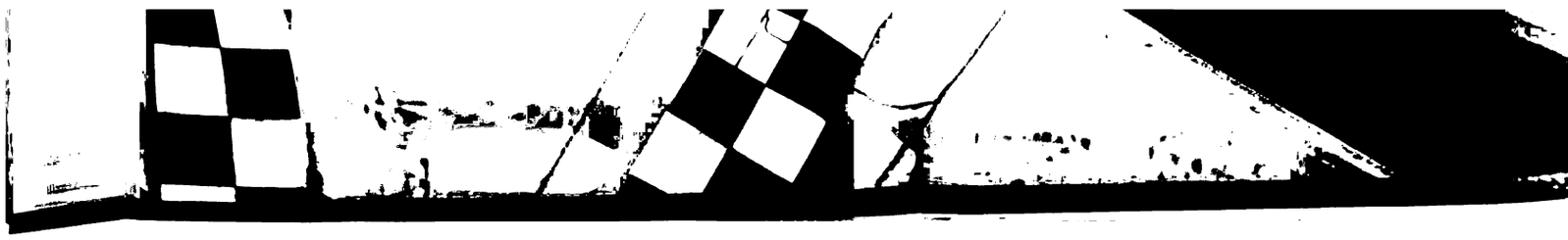


TABLE I

McLEOD PRESSURE VERSUS TIME FOR NICKEL FOIL 0M4
FOR NITROGEN AT VARIOUS OVERPRESSURES
(pressure in microns; time in minutes)

Item	Overpressure						
	20	49	92	134	178	231	275
p_0	0.02	0.03	0.03	0.02	0.04	0.05	0.03
p_{30}	0.21	0.21	0.22	0.23	0.23	0.22	0.22
t°C	25.2	25.2	25.4	25.7	25.6	25.3	25.3
Time 2	0.13	0.24	0.42	0.62	0.90	1.3	1.4
4	0.26	0.48	0.84	1.2	1.8	2.6	2.8
6	0.39	0.72	1.3	1.8	2.7	3.9	4.2
8	0.52	0.96	1.7	2.5	3.6	5.2	5.5
10	0.65	1.2	2.1	3.1	4.5	6.5	6.9
12	0.78	1.4	2.6	3.7	5.4	7.8	8.3
14	0.91	1.7	3.0	4.3	6.3	9.1	9.7
16	1.0	1.9	3.4	5.0	7.2	10	11
18	1.2	2.1	3.8	5.6	8.1	11	12
20	1.3	2.4	4.3	6.2	9.0	12	14
22	1.4	2.6	4.7	6.8	10	13	15
24	1.6	2.9	5.1	7.5	11	14	17
26	1.7	3.1	5.5	8.1	12	15	18
28	1.8	3.4	5.9	8.7	13	16	20
30	2.0	3.6	6.3	9.3	14	17	21

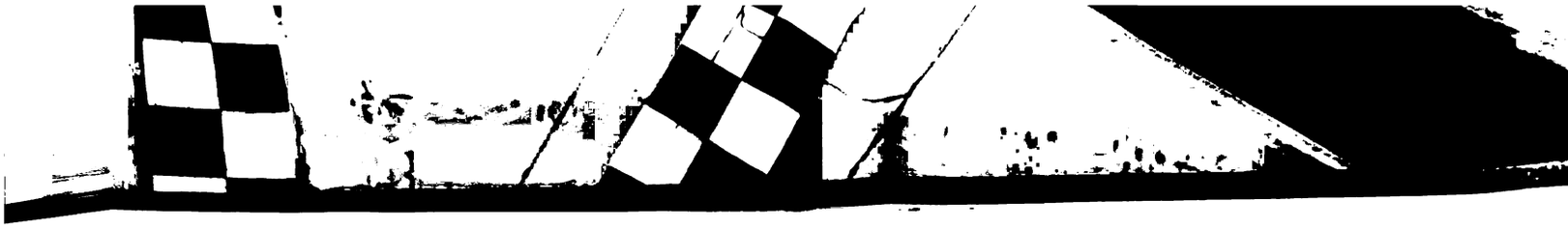


TABLE II

McLEOD PRESSURE VERSUS TIME FOR NICKEL FOIL 0M4
FOR HYDROGEN AT VARIOUS OVERPRESSURES
(pressure in microns; time in minutes)

Item	Overpressure							
	24	51	91	138	175	219	256	293
p_0	0.02	0.04	0.05	0.03	0.03	0.05	0.04	0.03
p_{30}	0.23	0.24	0.25	0.25	0.25	0.23	0.22	0.22
t°C	25.3	25.4	25.7	25.8	25.5	25.2	25.1	25.0
Time 2 . .	0.84	0.90	1.6	2.3	3.0	3.8	4.5	5.1
4 . .	1.7	1.8	3.2	4.6	6.0	7.6	9.0	10.2
6 . .	2.5	2.7	4.7	6.9	9.0	11.4	13.5	15.2
8 . .	3.4	3.6	6.2	8.3	12.0	15.2	18.0	20.4
10 . .	4.2	4.5	7.8	10.5	15.1	19.0	22	26
12 . .	5.1	5.4	9.3	12.8	18.1	22.8	27	31
14 . .	5.9	6.3	10.9	14.1	21.1	26.6	31	36
16 . .	6.8	7.2	12.4	16.4	24	30	36	42
18 . .	7.8	8.1	13.9	18.8	27	34	40	47
20 . .	8.9	9.1	15.5	20.9	30	38	44	52
22 . .	9.8	10.0	17.1	23	33	42	49	56
24 . .	10.4	10.9	18.7	27	36	45	53	61
26 . .	10.9	11.8	20	29	39	49	58	66
28 . .	11.8	12.7	22	32	42	53	63	70
30 . .	12.6	13.6	23	35	45	57	68	75

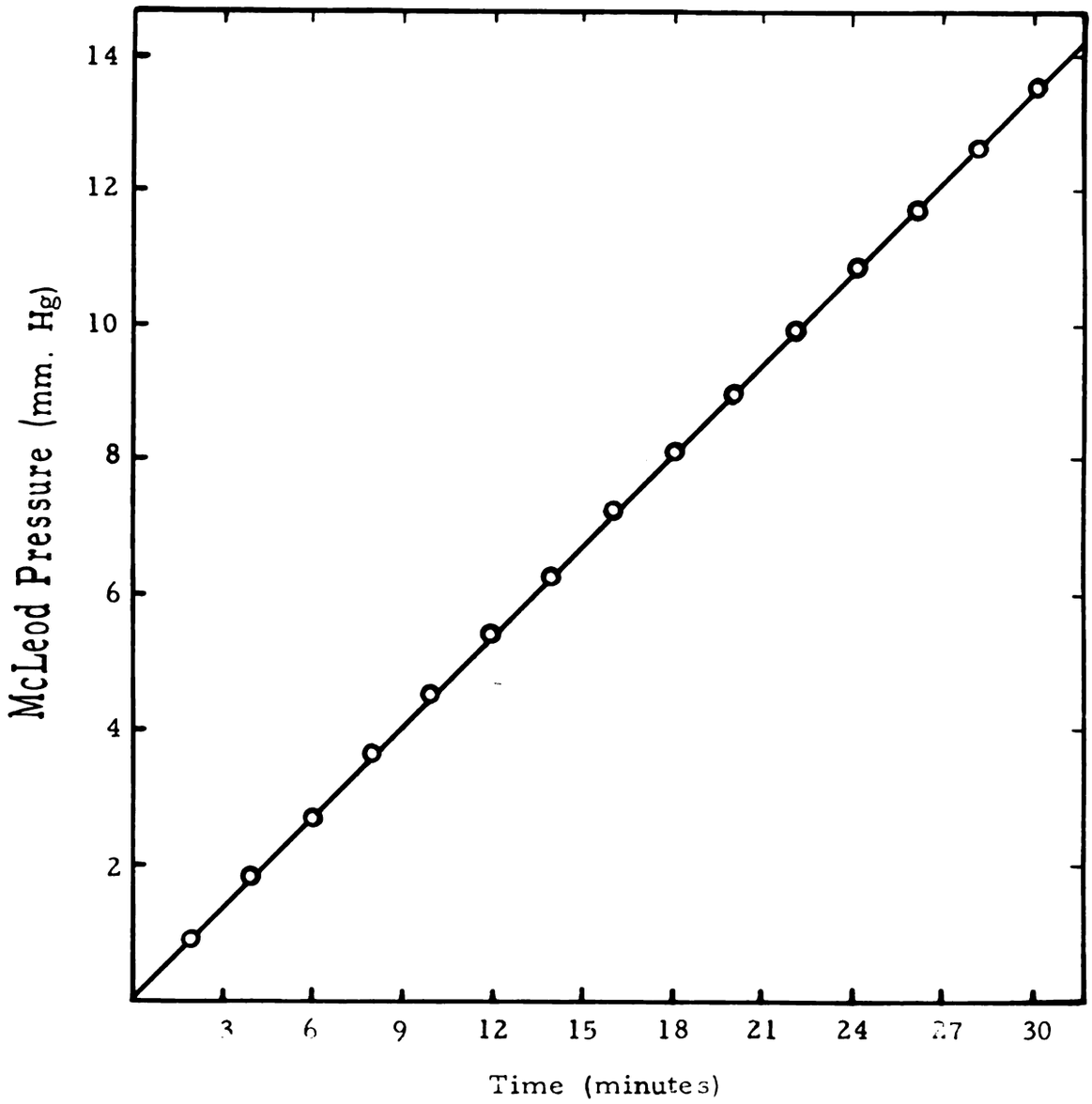


Figure 3. Nickel foil 0M4 for hydrogen at an overpressure of 51 mm. Hg, McLeod pressure versus time.

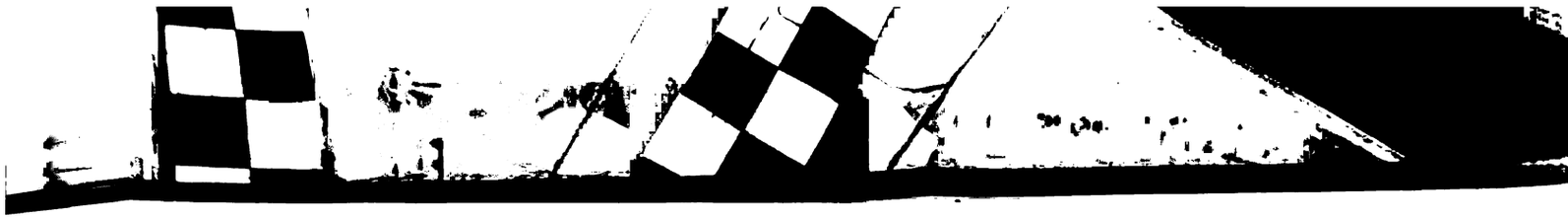


TABLE III

McLEOD PRESSURE VERSUS TIME FOR NICKEL FOIL 0M4
FOR DIBORANE AT VARIOUS OVERPRESSURES
(pressure in microns; time in minutes)

Item	Overpressure						
	38	92	133	173	221	260	292
P_0	0.02	0.03	0.03	0.04	0.02	0.03	0.03
P_{30}	0.23	0.25	0.26	0.26	0.25	0.25	0.25
t°C	25.3	25.6	25.8	25.9	25.7	25.7	25.6
Time 2	0.24	0.48	0.68	0.90	1.1	1.3	1.5
4	0.48	0.96	1.4	1.8	2.2	2.6	3.0
6	0.72	1.4	2.0	2.7	3.3	3.9	4.5
8	0.96	1.9	2.7	3.6	4.4	5.2	6.0
10	1.2	2.4	3.4	4.5	5.5	6.5	7.5
12	1.5	2.9	4.1	5.4	6.6	7.8	9.0
14	1.7	3.3	4.7	6.4	7.7	9.1	10
16	2.0	3.8	5.4	7.3	8.8	10	12
18	2.2	4.3	6.1	8.0	9.9	11	13
20	2.4	4.8	6.8	8.8	11	13	15
22	2.7	5.2	7.4	9.5	12	14	17
24	3.0	5.7	8.1	10	13	15	18
26	3.2	6.2	8.8	11	14	17	20
28	3.4	6.7	9.4	12	15	18	21
30	3.6	7.2	10	13	16	20	23

TABLE IV

McLEOD PRESSURE VERSUS TIME FOR NICKEL FOIL 0M4
FOR DIBORANE MIXTURES WITH
HYDROGEN OR NITROGEN
(pressure in microns; time in minutes)

Item	Gas Added to Diborane					
	H_2	H_2	H_2	N_2	N_2	N_2
P_0 B_2H_6	95	141	203	75	140	200
P_0 H_2 or N_2	203	140	90	206	150	87
P_0 total	298	281	293	281	290	287
p_0	0.02	0.03	0.05	0.04	0.02	0.04
p_{30}	0.26	0.27	0.27	0.25	0.24	0.23
t°C	25.8	25.9	26.0	25.6	25.4	25.2
Time 2	2.9	2.2	2.1	1.4	1.4	1.5
4	5.8	4.4	4.2	2.8	2.8	3.0
6	8.7	6.6	6.3	4.2	4.2	4.5
8	11	8.8	8.4	6.6	6.6	6.0
10	14	11	10	7.9	8.0	7.5
12	17	14	12	8.3	9.3	9.0
14	20	16	14	9.7	11	10
16	23	18	16	11	12	12
18	26	21	19	12	14	13
20	29	23	21	14	15	15
22	32	25	23	15	16	16
24	35	27	25	17	18	18
26	37	29	27	18	19	20
28	40	31	29	20	21	21
30	43	33	32	21	22	23
p B_2H_6 after removal of other	6	10	15	5.5	10	15

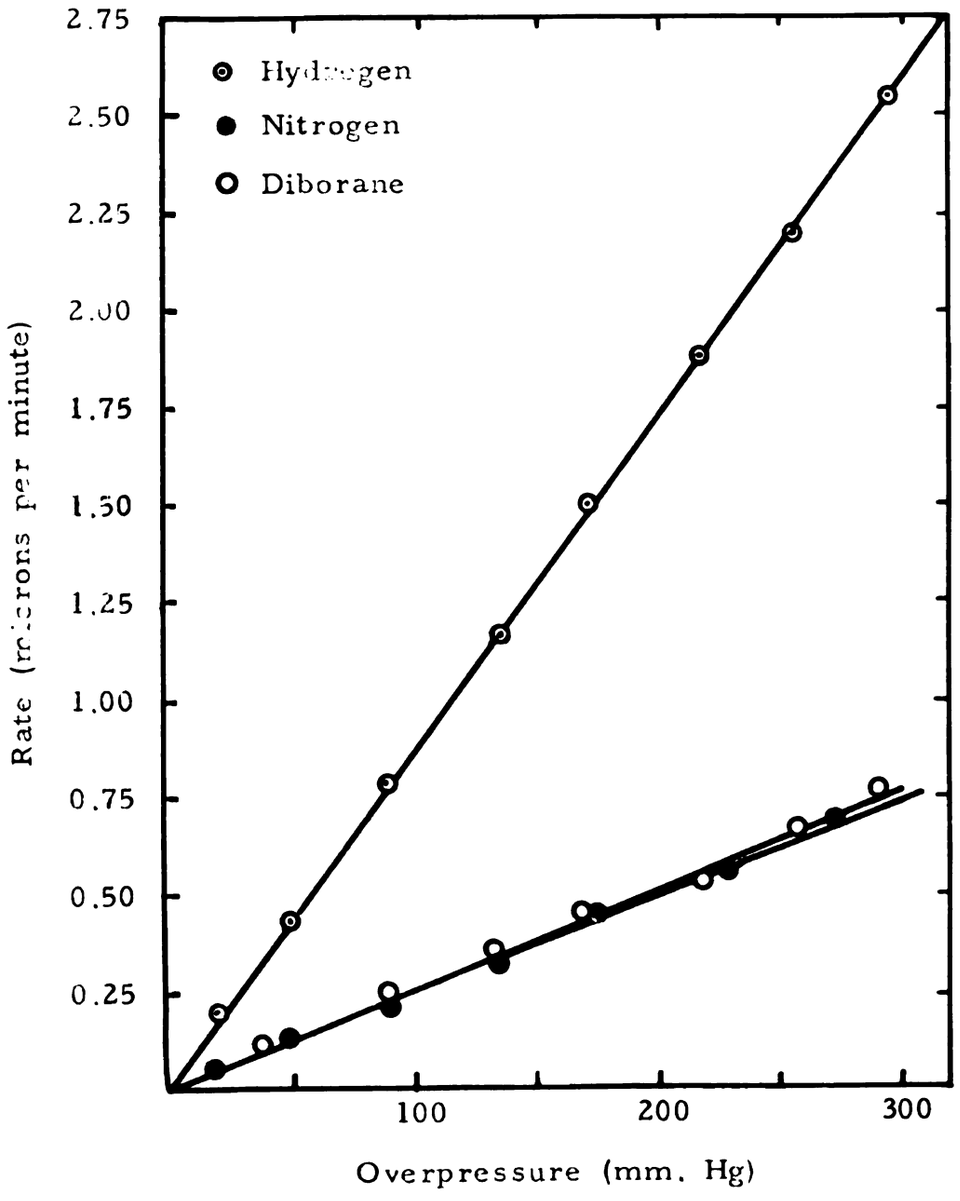


Figure 4. Nickel foil 0M4, rate versus overpressure.

TABLE V

McLEOD PRESSURE VERSUS TIME FOR NICKEL FOIL 0M3
FOR NITROGEN AT VARIOUS OVERPRESSURES
(pressure in microns; time in minutes)

Item	Overpressure						
	50	120	166	204	232	265	295
p_0	0.02	0.02	0.04	0.04	0.04	0.05	0.04
p_{30}	0.23	0.25	0.26	0.25	0.22	0.22	0.23
t°C	25.3	25.7	25.9	25.6	25.0	25.2	25.0
Time 2	0.54	1.3	1.8	2.2	2.6	2.9	3.2
4	1.1	2.6	3.6	4.4	5.0	5.8	6.4
6	1.6	3.9	5.4	6.6	7.5	8.7	9.6
8	2.1	5.2	7.2	8.8	10	11	13
10	2.7	7.5	9.0	11	12	14	16
12	3.2	8.8	11	13	14	17	19
14	3.7	10	13	15	17	20	22
16	4.3	11	15	17	19	22	26
18	4.8	12	16	19	22	25	29
20	5.3	14	18	22	24	28	32
22	5.8	15	20	24	27	31	35
24	6.3	16	21	26	29	34	38
26	6.8	17	23	28	32	37	42
28	7.6	18	25	30	35	40	45
30	8.3	19	26	33	38	43	48

TABLE VI

McLEOD PRESSURE VERSUS TIME FOR NICKEL FOIL 0M3 FOR HYDROGEN AT VARIOUS OVERPRESSURES
(pressure in microns, except as noted; time in minutes)

Item		Overpressure						
		44	85	120	171	204	256	290
p_0		0.02	0.04	0.03	0.06	0.04	0.02	0.05
p_{30}		0.25	0.25	0.25	0.23	0.23	0.21	0.20
t°C		25.6	25.7	25.6	25.3	25.2	24.9	24.8
Time	2	2.0	7.0	8.5	13.5	16.5	20.5	23
	4	4.0	14.0	17.0	27.0	33	41	46
	6	6.0	21.0	25.5	40	49	61	69
	8	8.1	28.1	34.1	54	67	82	88
	10	10.1	35	42	67	83	0.10*	0.11*
	12	12.1	42	51	81	0.10*	0.13*	0.14*
	14	14.0	49	59	94	0.12*	0.15*	0.16*
	16	16.0	56	68	0.11*	0.13*	0.17*	0.19*
	18	17.9	63	76	0.12*	0.15*	0.19*	0.21*
	20	19.9	70	85	0.14*	0.16*	0.21*	0.23*
	22	21.9	77	93	0.15*	0.18*	0.24*	0.26*
	24	24.0	84	0.10*	0.16*	0.20*	0.26*	0.28*
	26	26.0	91	0.11*	0.18*	0.21*	0.28*	0.31*
	28	28.0	98	0.12*	0.19*	0.23*	0.30*	0.33*
	30	30.0	0.10*	0.13*	0.20*	0.25*	0.32*	0.35*

* Pressure in millimeters.

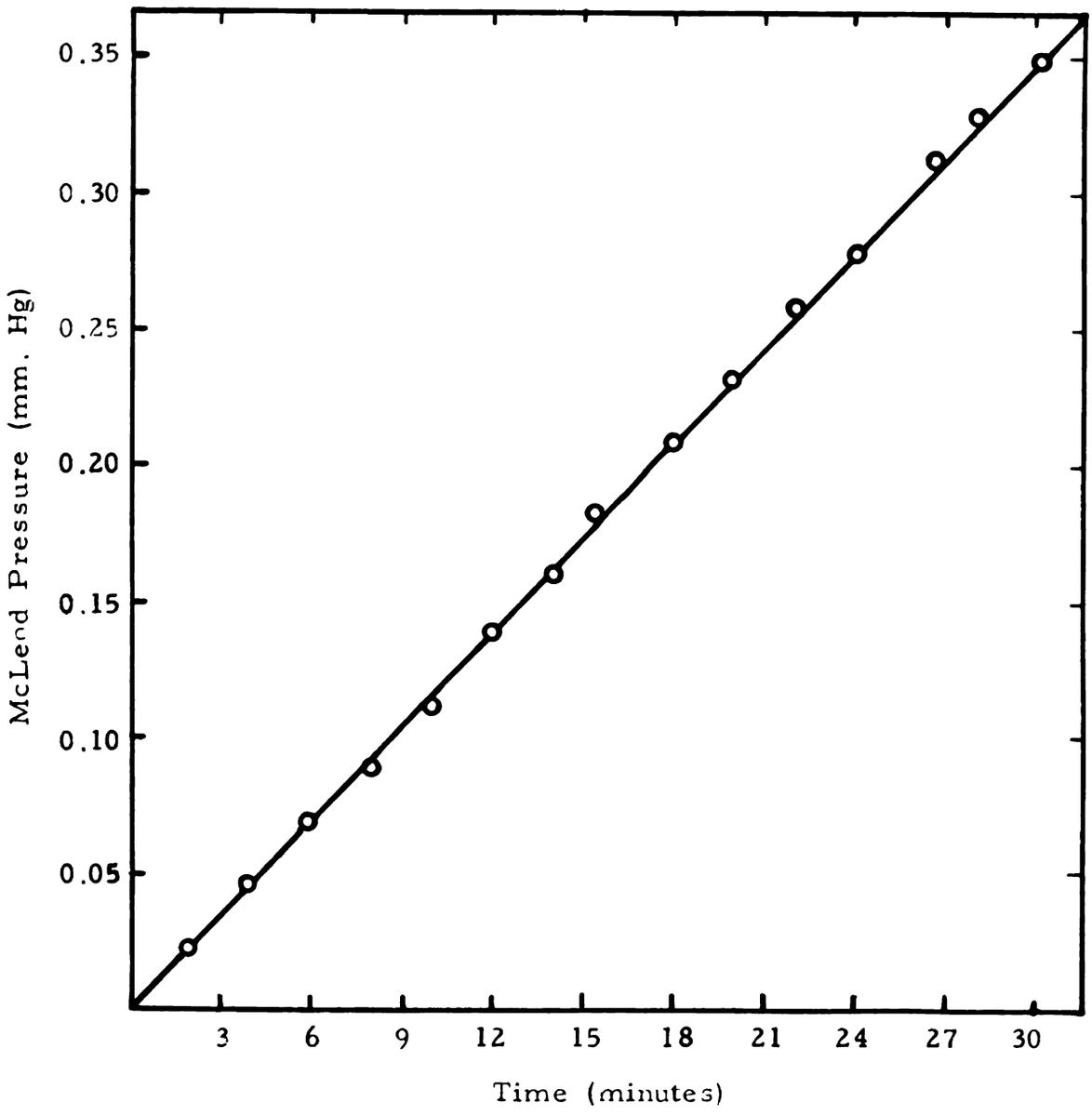


Figure 5. Nickel foil 0M3 for hydrogen at an overpressure of 290 mm. Hg, McLeod pressure versus time.

TABLE VII

McLEOD PRESSURE VERSUS TIME FOR NICKEL FOIL 0M3 FOR DIBORANE AT VARIOUS OVERPRESSURES
(pressure in microns; time in minutes)

Item		Overpressure						
		41	80	127	175	220	260	289
P_0		0.02	0.02	0.02	0.04	0.03	0.03	0.02
P_{30}		0.23	0.23	0.25	0.26	0.23	0.21	0.20
t°C		25.2	25.4	25.7	25.9	25.2	24.9	24.7
Time	2	0.42	0.94	1.5	2.0	2.6	3.0	3.3
	4	0.84	1.9	3.0	4.0	5.2	6.0	6.6
	6	1.2	2.8	4.5	6.0	7.8	9.0	9.9
	8	1.7	3.7	6.0	8.0	10	12	13
	10	2.1	4.6	7.5	10	12	15	16
	12	2.5	5.5	10	12	15	18	20
	14	2.9	6.4	11	14	17	21	23
	16	3.4	7.4	13	16	20	24	26
	18	3.8	8.3	14	18	23	27	30
	20	4.2	9.2	16	20	26	30	33
	22	4.7	10	17	22	28	33	36
	24	5.1	11	19	24	31	36	40
	26	5.5	12	20	26	34	39	43
	28	5.8	13	22	28	37.	42	46
	30	6.3	14	23	31	39	45	50

TABLE VIII

McLEOD PRESSURE VERSUS TIME FOR NICKEL FOIL 0M3
FOR DIBORANE MIXTURES WITH
HYDROGEN OR NITROGEN
(pressure in microns; time in minutes)

Item	Gas Added to Diborane					
	H_2	H_2	H_2	N_2	N_2	N_2
P_0 B_2H_6	93	147	193	87	178	201
P_0 H_2 or N_2	194	127	85	189	150	82
P_0 total	287	274	278	276	328	283
p_0	0.02	0.02	0.03	0.02	0.03	0.03
p_{30}	25.2	25.4	25.7	25.1	24.9	24.8
Time 2	6.7	5.3	4.7	3.1	3.7	3.3
4	13	10	9.4	6.2	7.4	6.6
6	20	16	14	9.3	11	9.9
8	27	21	19	12	14	12
10	33	26	23	15	18	15
12	39	31	28	18	21	19
14	46	37	32	21	25	22
16	52	42	37	25	29	25
18	59	47	42	28	33	28
20	66	53	46	31	37	32
22	72	58	51	34	40	35
24	78	64	56	37	44	38
26	85	69	61	40	48	42
28	92	75	65	43	52	45
30	100	80	70	46	56	49
p B_2H_6 after removal of other	15	25	33	15	31	32

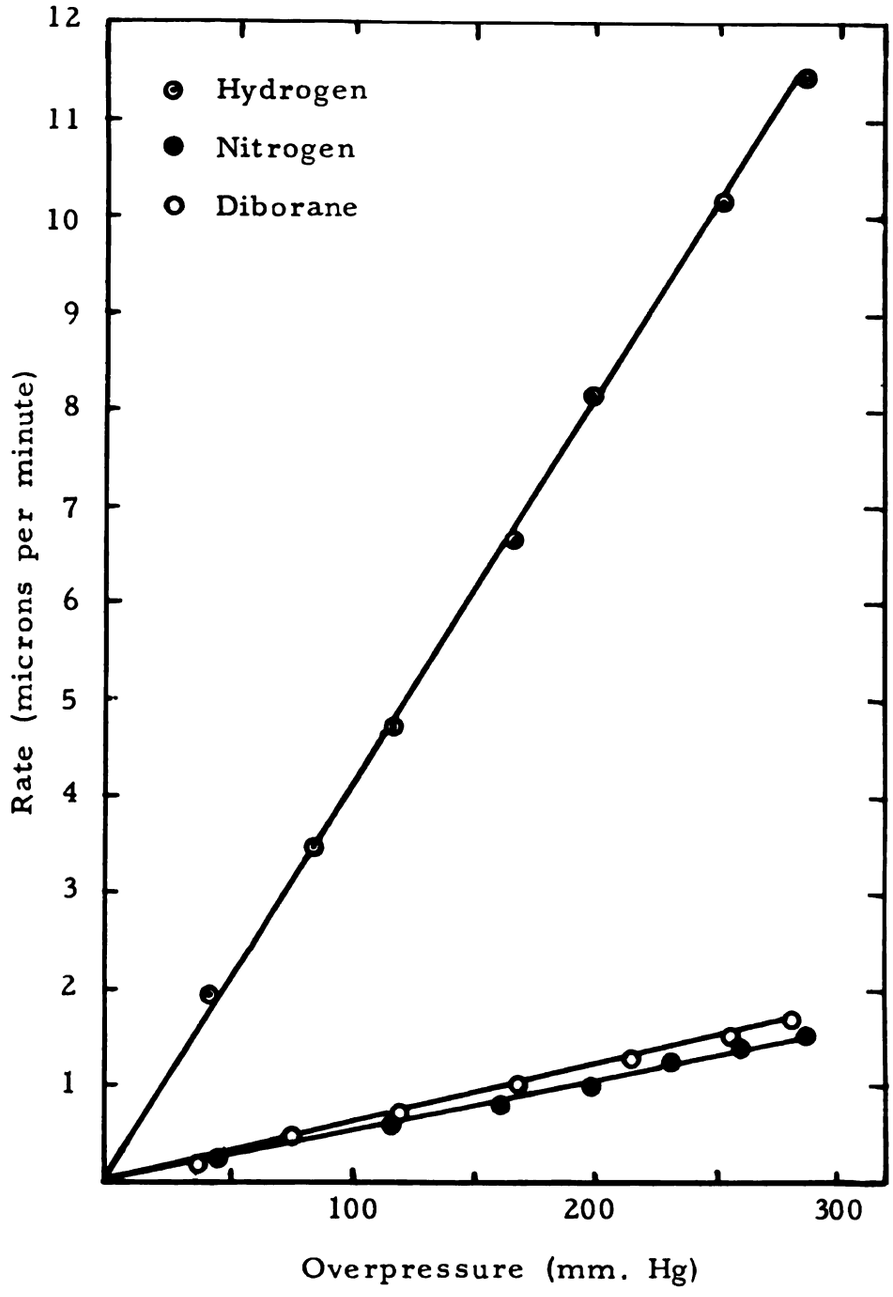


Figure 6. Nickel foil 0M3, rate versus overpressure.

TABLE IX

McLEOD PRESSURE VERSUS TIME FOR NICKEL FOIL 0M2 FOR NITROGEN AT VARIOUS OVERPRESSURES
(pressure in microns; time in minutes)

Item	Overpressure							
	49	106	138	158	195	224	260	300
p_0	0.02	0.02	0.03	0.05	0.02	0.03	0.04	0.05
p_{30}	0.31	0.31	0.30	0.30	0.30	0.31	0.32	0.32
t°C	25.6	25.4	25.3	25.3	25.4	25.5	25.8	25.7
Time 2 . .	1.0	1.7	2.2	2.9	3.2	4.0	4.5	5.2
4 . .	1.9	2.5	4.4	5.7	6.3	8.0	9.1	10
6 . .	2.9	4.2	6.7	8.5	9.5	12	13	15
8 . .	3.8	5.9	9.0	11	12	16	18	21
10 . .	4.7	7.6	11	14	15	20	22	26
12 . .	5.6	9.2	13	17	18	24	27	32
14 . .	6.5	11	16	20	22	28	31	37
16 . .	7.4	13	18	22	25	32	36	42
18 . .	8.3	15	20	25	28	36	40	47
20 . .	9.2	16	22	28	32	40	45	52
22 . .	10	18	25	31	35	44	49	58
24 . .	11	20	27	34	38	48	53	63
26 . .	12	21	30	37	42	52	58	68
28 . .	13	23	32	40	45	56	64	73
30 . .	14	24	34	42	48	60	68	78

TABLE X

McLEOD PRESSURE VERSUS TIME FOR NICKEL FOIL 0M2 FOR HYDROGEN AT VARIOUS OVERPRESSURES
(pressure in microns, except as noted; time in minutes)

Item	Overpressure						
	50	87	140	172	198	248	294
P_0	0.05	0.02	0.02	0.02	0.04	0.05	0.10
P_{30}	0.27	0.28	0.29	0.29	0.30	0.29	0.28
t°C	24.8	25.1	25.3	25.4	25.5	25.3	25.2
Time 2	3.5	5.5	9.2	11.4	13.6	17.1	20.0
4	7.0	11.0	18.3	22 8	27.4	34	40
6	10.5	16.5	27.6	34	39	51	60
8	14.0	22.0	36	45	53	68	80
10	17.5	27.6	46	56	67	85	0.10*
12	21	33	55	68	81	0.10*	0.12*
14	24	39	64	79	94	0.12*	0.14*
16	28	44	72	90	0.11*	0.14*	0.16*
18	31	49	79	0.10*	0.12*	0.15*	0.18*
20	35	55	85	0.11*	0.13*	0.17*	0.20*
22	38	61	91	0.12*	0.15*	0.18*	0.22*
24	42	67	0.10*	0.13*	0.16*	0.20*	0.24*
26	46	73	0.12*	0.14*	0.17*	0.22*	0.26*
28	49	80	0.13*	0.16*	0.18*	0.24*	0.28*
30	53	86	0.14*	0.17*	0.20*	0.25*	0.30*

* Pressure in millimeters.

TABLE XI

McLEOD PRESSURE VERSUS TIME FOR NICKEL FOIL 0M2 FOR DIBORANE AT VARIOUS OVERPRESSURES
(pressure in microns; time in minutes)

Item	Overpressure						
	24	51	94	138	190	237	289
p_0	0.02	0.03	0.05	0.02	0.02	0.03	0.05
p_{30}	0.24	0.25	0.25	0.25	0.24	0.23	0.23
t°C	25.1	25.3	25.4	25.4	25.2	24.9	24.8
Time 2	0.36	0.94	1.5	2.4	3.2	4.0	4.8
4	0.72	1.8	3.0	4.8	6	8	10
6	1.1	2.8	4.5	7	9	12	15
8	1.5	3.7	6.0	10	13	16	19
10	1.9	4.7	7	12	16	20	24
12	2.2	5.6	9	15	19	24	29
14	2.6	6.5	10	18	23	28	33
16	3.0	7.4	12	21	26	32	38
18	3.3	8.3	13	23	29	36	43
20	3.7	9.2	15	26	33	40	48
22	4.0	10	16	28	36	44	52
24	4.4	11	18	31	39	48	57
26	4.7	12	19	33	43	52	62
28	5.0	13	21	35	46	56	67
30	5.4	14	23	36	49	60	72

TABLE XII

McLEOD PRESSURE VERSUS TIME FOR NICKEL FOIL 0M2
FOR DIBORANE MIXTURES WITH
HYDROGEN OR NITROGEN
(pressure in microns, except as noted; time in minutes)

Item	Gas Added to Diborane					
	H_2	H_2	H_2	N_2	N_2	N_2
P_0 B_2H_6	89	140	205	95	146	199
P_0 H_2 or N_2	190	149	78	195	146	75
P_0 total	279	289	283	290	292	274
p_0	0.02	0.03	0.03	0.02	0.04	0.05
p_{30}	0.24	0.23	0.21	0.21	0.22	0.21
t°C	25.7	25.6	25.3	25.3	25.4	25.2
Time 2	10	9.6	7.2	5.0	5.0	4.6
4	20	19	14	10	10	9.2
6	30	28	22	15	15	14
8	41	38	29	20	20	18
10	51	47	36	25	25	23
12	62	57	44	30	30	27
14	72	66	51	35	35	32
16	83	76	66	40	40	36
18	93	85	73	45	45	41
20	0.11*	94	80	50	50	45
22	0.12*	0.10*	86	55	55	49
24	0.13*	0.11*	93	60	60	54
26	0.14*	0.12*	99	65	65	58
28	0.15*	0.13*	0.10*	70	70	63
30	0.16*	0.14*	0.11*	75	75	69
p B_2H_6 after removal of other	20	34	49	23	36	49

* Pressure in millimeters.

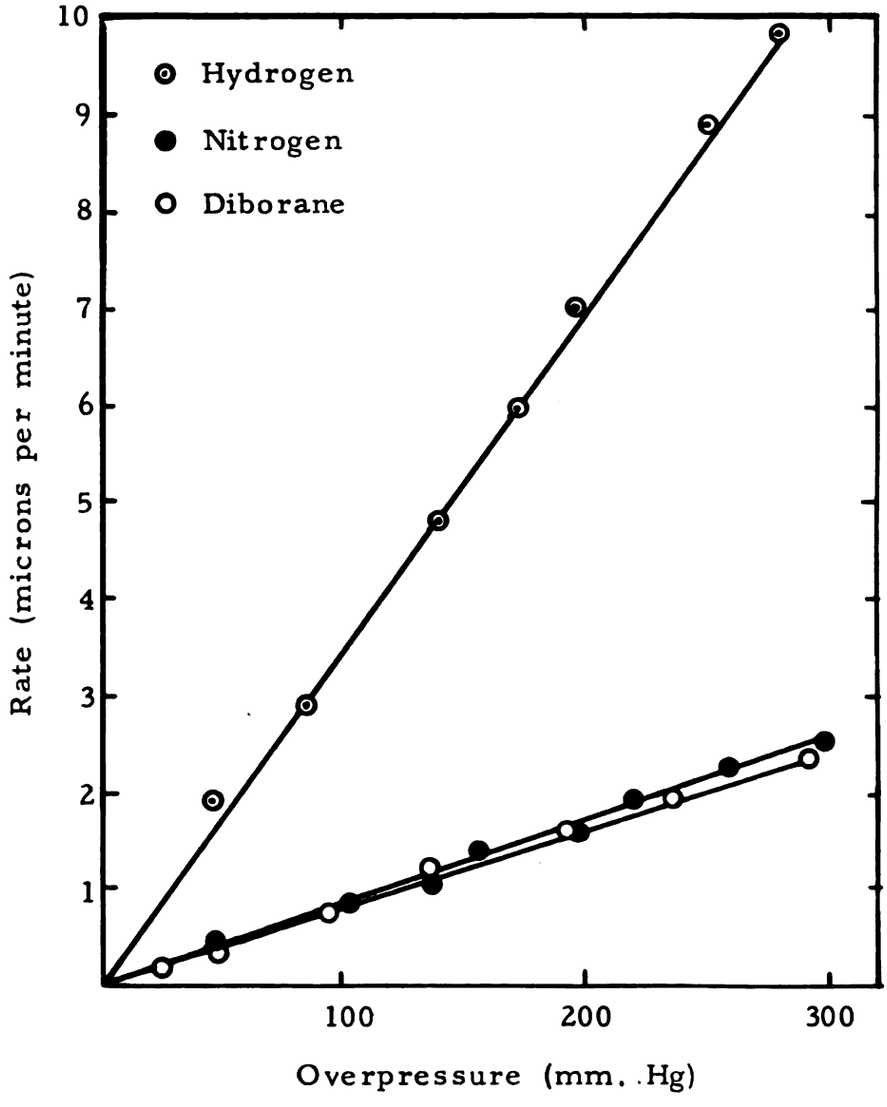


Figure 7. Nickel foil 0M2, rate versus overpressure.

TABLE XIII

McLEOD PRESSURE VERSUS TIME FOR NICKEL FOIL 0M1 FOR NITROGEN AT VARIOUS OVERPRESSURES
(pressure in microns, except as noted; time in minutes)

Item		Overpressure						
		25	99	133	175	218	253	289
p_0		0.02	0.03	0.03	0.04	0.02	0.04	0.05
p_{30}		0.20	0.21	0.21	0.22	0.18	0.18	0.18
t°C		25.6	25.7	25.8	25.8	24.3	24.3	24.2
Time	2	1.6	3.2	4.6	5.6	7.1	8.4	9.5
	4	3.2	6.3	9.2	11	14	17	18
	6	4.8	9.8	13	16	21	25	27
	8	6.3	13	17	22	28	34	37
	10	8.0	16	22	27	35	42	46
	12	9.7	19	26	33	42	51	55
	14	11	23	31	38	49	59	64
	16	12	25	35	44	56	67	74
	18	14	28	40	49	64	76	83
	20	16	33	45	55	71	84	92
	22	18	36	49	61	77	93	96
	24	20	39	54	67	84	97	0.11*
	26	22	42	59	73	91	0.11*	0.12*
	28	24	45	64	79	0.10*	0.12*	0.13*
	30	26	48	69	85	0.11*	0.13*	0.14*

* Pressure in millimeters.

TABLE XIV

McLEOD PRESSURE VERSUS TIME FOR NICKEL FOIL 0M1 FOR HYDROGEN AT VARIOUS OVERPRESSURES
(pressure in microns, except as noted; time in minutes)

Item	Overpressure						
	49	102	151	190	222	261	285
p_0	0.02	0.02	0.04	0.05	0.02	0.04	0.05
p_{30}	0.18	0.19	0.20	0.20	0.16	0.15	0.15
t°C	25.2	25.4	25.7	25.8	24.6	24.5	24.4
Time 2	6.4	12.4	19	23	28	32	36
4	12.8	24.8	36	46	56	64	72
6	19.3	37	55	69	84	96	0.11*
8	25.7	49	74	94	0.10*	0.11*	0.14*
10	32	62	93	0.12*	0.14*	0.14*	0.18*
12	38	74	0.11*	0.14*	0.17*	0.17*	0.21*
14	45	86	0.13*	0.16*	0.20*	0.21*	0.25*
16	51	99	0.15*	0.18*	0.23*	0.24*	0.28*
18	57	0.11*	0.17*	0.21*	0.27*	0.27*	0.32*
20	63	0.12*	0.19*	0.23*	0.30*	0.30*	0.36*
22	69	0.14*	0.21*	0.25*	0.33*	0.34*	0.40*
24	76	0.15*	0.22*	0.25*	0.36*	0.37*	0.43*
26	82	0.16*	0.24*	0.30*	0.39*	0.41*	0.47*
28	90	0.18*	0.26*	0.33*	0.42*	0.45*	0.51*
30	97	0.19*	0.28*	0.35*	0.45*	0.49*	0.54*

* Pressure in millimeters.

TABLE XV

McLEOD PRESSURE VERSUS TIME FOR NICKEL FOIL 0M1 FOR DIBORANE AT VARIOUS OVERPRESSURES
(pressure in microns, except as noted; time in minutes)

Item	Overpressure						
	51	96	133	179	212	257	289
p_0	0.02	0.02	0.03	0.03	0.02	0.05	0.05
p_{30}	0.15	0.16	0.17	0.17	0.17	0.13	0.12
t°C	25.3	25.5	25.7	25.8	25.8	24.6	24.4
Time 2	1.7	3.4	4.6	6.0	7.2	8.0	9.6
4	3.4	6.8	9.2	12	14	16	18
6	5.0	10	14	18	21	24	28
8	6.8	13	18	24	28	32	37
10	8.4	17	23	30	36	40	47
12	10	20	27	36	43	47	56
14	11	24	32	42	50	56	66
16	13	27	35	47	57	63	75
18	15	30	39	54	64	72	85
20	17	33	44	60	72	80	94
22	18	36	49	67	79	88	0.10*
24	20	40	54	72	86	94	0.11*
26	22	44	59	78	93	0.10*	0.12*
28	24	47	64	84	0.10*	0.11*	0.13*
30	26	50	69	90	0.11*	0.13*	0.14*

* Pressure in millimeters.

TABLE XVI

McLEOD PRESSURE VERSUS TIME FOR NICKEL FOIL 0M1 FOR DIBORANE MIXTURES WITH HYDROGEN OR NITROGEN

(pressure in microns, except as noted; time in minutes)

Item	Gas Added to Diborane					
	H_2	H_2	H_2	N_2	N_2	N_2
P_0 B_2H_6	88	143	197	91	142	210
P_0 H_2 or N_2	191	140	92	182	146	70
P_0 total	279	283	289	273	288	280
p_0	0.02	0.04	0.05	0.02	0.05	0.05
p_{30}	0.17	0.17	0.17	0.15	0.15	0.14
t°C	25.7	25.7	25.8	25.2	25.1	25.0
Time 2	31	26	21	11	9.4	9.2
4	62	51	40	22	19	18
6	93	78	63	33	27	27
8	0.12*	0.10*	84	43	37	36
10	0.15*	0.12*	0.11*	54	46	45
12	0.18*	0.15*	0.13*	65	56	54
14	0.22*	0.17*	0.15*	77	65	63
16	0.25*	0.20*	0.19*	88	74	72
18	0.28*	0.22*	0.20*	99	84	81
20	0.31*	0.25*	0.22*	0.11*	93	90
22	0.35*	0.28*	0.24*	0.12*	0.10*	0.10*
24	0.38*	0.30*	0.26*	0.14*	0.11*	0.11*
26	0.41*	0.33*	0.28*	0.15*	0.12*	0.12*
28	0.44*	0.36*	0.30*	0.16*	0.13*	0.13*
30	0.47*	0.39*	0.31*	0.17*	0.14*	0.14*
P B_2H_6 after removal of other	44	72	97	46	71	0.10*

* Pressure in millimeters.

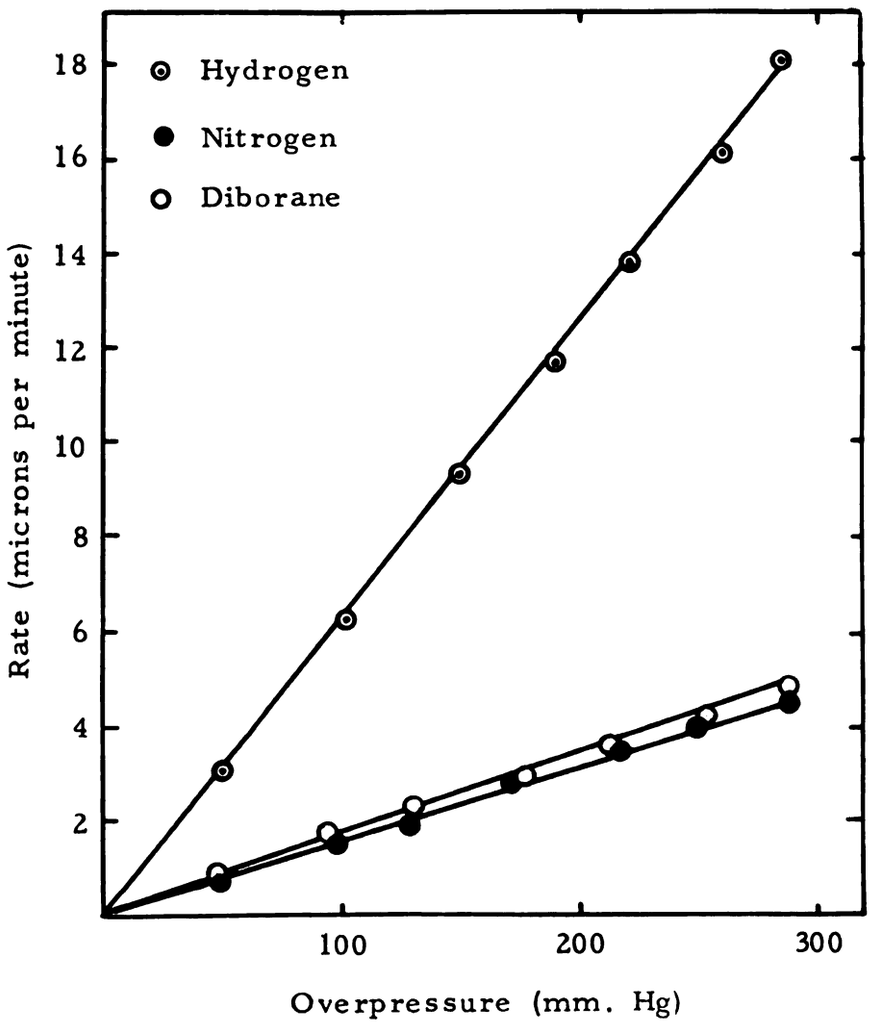


Figure 8. Nickel foil 0M1, rate versus overpressure.

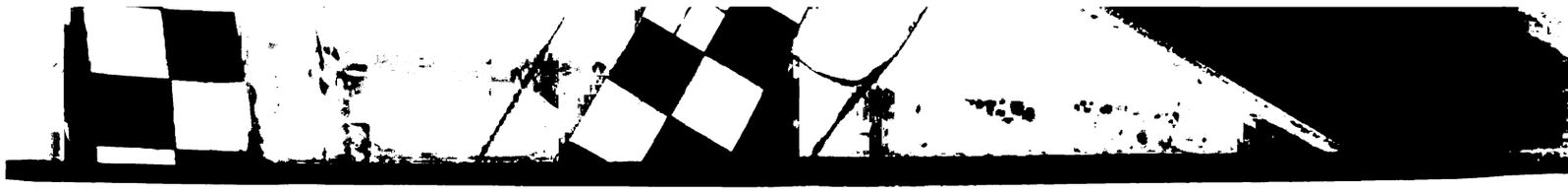


TABLE XVII

PLASTIC AND BRASS DESIGNATION

Plastic			
No.	Plastic	Manufacturer	Thickness (inch)
1	Trithene	Visking Corp.	0.0005
2	Trithene	Visking Corp.	0.001
3	Visqueen	Visking Corp.	0.002-0.0025
4	Visqueen	Visking Corp.	0.003-0.0035
5	Bakelite VBA9925	Bakelite Co.	0.001
6	Tenite Acetate $043H_4$	Tennessee Eastman Co.	0.001
7	Tenite Butyrate 460MH	Tenessee Eastman Co.	0.001
8	Tenite Butyrate 265MH	Tennessee Eastman Co.	0.008
9	Kodapak F122	Tennessee Eastman Co.	0.009

Brass[1]	
No.	Thickness (inch)
B10	0.0010
B15	0.0015
B20	0.0020
B30	0.0030
B40	0.0040

[1] All brass is the commercial 70-30 shim stock manufactured by Precision Steel Warehouse, Inc.

TABLE XVIII

McLEOD PRESSURE VERSUS TIME FOR TRITHENE PLASTIC FOR THREE GASES AT APPROXIMATELY A SINGLE OVERPRESSURE
(pressure in microns; time in minutes)

Item	Foil 1			Foil 2		
	N_2	H_2	B_2H_6	N_2	H_2	B_2H_6
P_0	48	49	49	49	49	49
p_0	0.04	0.04	0.02	0.02	0.04	0.03
p_{30}	0.40	0.43	0.42	0.39	0.40	0.39
t°C	24.2	24.8	24.5	23.6	24.0	23.8
Time 2	0.8	6.0	2.4	0.5	3.6	1.7
4	1.6	12	5.5	1.0	7.1	3.4
6	2.3	18	8.0	1.5	11	5.2
8	2.9	24	11	2.0	14	7.1
10	3.7	30	14	2.5	18	9.0
12	4.5	36	17	3.0	22	11
14	5.2	42	19	3.5	25	13
16	5.9	48	23	4.0	28	15
18	6.7	54	26	4.5	32	17
20	7.5	60	28	5.0	35	19
22	8.1	66	31	5.5	39	21
24	8.9	72	34	6.0	42	23
26	9.6	78	37	6.5	46	25
28	10	84	40	7.0	49	27
30	11	90	43	7.5	52	29

TABLE XIX

McLEOD PRESSURE VERSUS TIME FOR VISQUEEN AND BAKELITE PLASTICS FOR THREE GASES AT APPROXIMATELY A SINGLE OVERPRESSURE

(pressure in microns; time in minutes)

Item	Foil 3			Foil 5[a]		
	N_2	H_2	B_2H_6	N_2	H_2	B_2H_6
P_0	49	50	49	47	49	50
p_0	0.02	0.04	0.04	0.10	0.10	0.05
p_{30}	0.47	0.45	0.49	0.44	0.44	0.44
t°C	24.7	24.4	25.0	24.2	24.2	24.2
Time 2	0.07	0.45	0.10	0.70	2.0	0.36
4	0.13	0.8	0.15	1.0	4.2	0.7
6	0.16	1.2	0.19	1.3	6.4	1.0
8	0.20	1.7	0.22	1.6	8.6	1.3
10	0.23	2.1	0.26	2.0	11	1.8
12	0.28	2.5	0.29	2.3	13	2.2
14	0.31	2.9	0.33	2.6	15	2.5
16	0.35	3.4	0.36	2.9	17	2.9
18	0.40	3.8	0.40	3.3	19	3.2
20	0.43	4.2	0.43	3.7	22	3.6
22	0.48	4.7	0.47	4.0	24	3.9
24	0.52	5.1	0.50	4.3	26	4.3
26	0.56	5.5	0.54	4.6	28	4.6
28	0.60	5.9	0.57	5.0	31	4.9
30	0.63	6.4	0.61	5.3	33	5.4

[a] Foil 4 did not allow any gas to pass through.

TABLE XX

McLEOD PRESSURE VERSUS TIME FOR TENITE ACETATE AND BUTYRATE PLASTICS FOR THREE GASES AT APPROXIMATELY A SINGLE OVERPRESSURE

(pressure in microns; time in minutes)

Item	Foil 6			Foil 7		
	N_2	H_2	B_2H_6	N_2	H_2	B_2H_6
P_0	47	49	50	50	49	49
p_0	0.1	0.1	0.05	0.04	0.04	0.02
p_{30}	0.44	0.44	0.44	0.40	0.40	0.40
t°C	24.2	24.2	24.2	23.6	23.6	23.5
Time 2	0.08	1.2	0.09	0.5	5.2	0.9
4	0.15	2.3	0.15	1.0	11	1.9
6	0.21	3.4	0.20	1.5	16	2.9
8	0.29	4.5	0.28	2.0	21	3.9
10	0.33	5.6	0.33	2.5	27	5.0
12	0.40	6.7	0.37	3.0	32	6.2
14	0.46	7.8	0.44	3.5	37	7.2
16	0.53	8.9	0.49	4.0	41	8.3
18	0.60	10	0.55	4.5	45	10
20	0.70	11	0.60	5.0	49	12
22	0.75	12	0.66	5.5	54	14
24	0.80	13	0.73	6.0	59	17
26	0.85	14	0.79	6.5	64	19
28	0.90	15	0.85	7.0	69	22
30	0.97	17	0.90	7.5	74	24

TABLE XXI

McLEOD PRESSURE VERSUS TIME FOR TENITE BUTYRATE AND KODAPAK F122 PLASTICS FOR THREE GASES AT APPROXIMATELY A SINGLE OVERPRESSURE
(pressure in microns; time in minutes)

Item	Foil 8			Foil 9		
	N_2	H_2	B_2H_6	N_2	H_2	B_2H_6
P_0	50	49	49	49	49	49
p_0	0.04	0.04	0.02	0.04	0.05	0.02
p_{30}	0.40	0.40	0.40	0.49	0.43	0.44
t°C	23.6	23.6	23.5	25.8	24.5	25.1
Time 2	0.5	5.2	0.9	0.16	0.2	0.09
4	1.0	11	1.9	0.26	0.42	0.16
6	1.5	16	2.9	0.33	0.7	0.20
8	2.0	21	3.9	0.42	0.9	0.28
10	2.5	27	5.0	0.48	1.1	0.34
12	3.0	32	6.2	0.53	1.3	0.38
14	3.5	37	7.2	0.58	1.5	0.40
16	4.0	41	8.3	0.7	1.7	0.42
18	4.5	45	10	0.75	1.9	0.47
20	5.0	49	12	0.8	2.1	0.51
22	5.5	54	14	0.85	2.3	0.60
24	6.0	59	17	0.9	2.5	0.65
26	6.5	64	19	0.95	2.7	0.7
28	7.0	69	22	1.0	2.9	0.75
30	7.5	74	24	1.05	3.1	0.8

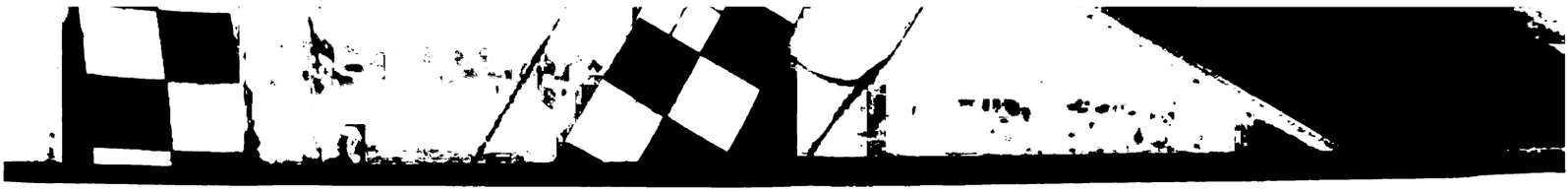


TABLE XXII

McLEOD PRESSURE VERSUS TIME FOR BRASS FOILS B10 AND B20 FOR THREE GASES AT APPROXIMATELY A SINGLE OVERPRESSURE

(pressure in microns, except as noted; time in minutes)

Item	Foil B10			Foil B20		
	N_2	H_2	B_2H_6	N_2	H_2	B_2H_6
P_0	49	50	49	50	49	49
p_0	0.04	0.04	0.03	0.03	0.03	0.05
p_{30}	0.40	0.47	0.48	0.40	0.41	0.41
t°C	23.9	25.1	25.3	24.0	24.2	24.2
Time 2	3.5	12	3.6	1.0	3.7	1.1
4	7.0	24	7.2	2.1	7.5	2.1
6	10.5	36	10.8	3.0	10.2	3.2
8	14.0	48	14.5	3.9	14.0	4.2
10	17.5	60	18.1	5.0	15.7	5.3
12	21.0	72	21.6	5.9	22.5	6.4
14	24.5	84	25.2	7.0	26.3	7.4
16	28.0	96	28.8	8.1	30.0	8.4
18	31	0.11*	32.4	9.1	34	9.5
20	35	0.12*	36	10.1	38	10.5
22	38	0.13*	40	11.0	41	11.5
24	42	0.14*	43	12.0	45	12.5
26	45	0.16*	47	12.9	48	13.5
28	49	0.17*	51	13.9	51	14.5
30	53	0.18*	54	15.0	53	15.5

* Pressure in millimeters.

TABLE XXIII

McLEOD PRESSURE VERSUS TIME FOR BRASS FOILS B15 AND B30 FOR THREE GASES AT APPROXIMATELY A SINGLE OVERPRESSURE
(pressure in microns; time in minutes)

Item	Foil B15			Foil B30		
	N_2	H_2	B_2H_6	N_2	H_2	B_2H_6
P_0	49	49	49	49	50	49
p_0	0.02	0.04	0.05	0.05	0.05	0.04
p_{30}	0.41	0.41	0.41	0.75	0.70	0.71
t°C	24.1	24.1	24.1	24.1	23.4	23.6
Time 2	1.7	6.4	1.8	0.6	1.7	0.6
4	3.4	12.8	3.5	1.0	3.5	1.0
6	5.1	19.3	5.3	1.5	5.0	1.5
8	6.9	26.2	7.1	2.0	6.7	2.1
10	8.5	32	8.8	2.4	8.5	2.6
12	10.2	38	10.5	2.9	10.0	3.2
14	11.8	45	12.2	3.4	11.7	3.7
16	13.6	51	14.0	3.9	13.5	4.3
18	15.3	57	15.8	4.4	15.0	4.8
20	17.0	64	17.5	4.9	17.0	5.3
22	17.7	71	18.4	5.5	18.5	6.0
24	19.4	77	20.1	6.0	20.5	6.5
26	20	84	21	6.5	22	7.0
28	22	90	23	7.0	24	7.5
30	23	96	24	7.5	25	8.0

TABLE XXIV

McLEOD PRESSURE VERSUS TIME FOR BRASS FOIL B40 FOR THREE GASES AT APPROXIMATELY A SINGLE OVERPRESSURE
(pressure in microns; time in minutes)

Item	Foil B40		
	N_2	H_2	B_2H_6
P_0	50	49	50
p_0	0.04	0.03	0.03
p_{30}	0.70	0.70	0.70
t°C	24.0	24.0	24.0
Time 2	0.45	1.3	0.48
4	0.9	2.4	0.95
6	1.3	3.6	1.4
8	1.7	4.8	1.7
10	2.0	6.0	2.1
12	2.3	7.2	2.4
14	2.6	8.4	2.7
16	3.0	9.6	3.1
18	3.3	10.8	3.5
20	3.6	12.0	3.8
22	4.0	13.2	4.3
24	4.3	14.4	4.6
26	4.6	15.6	4.9
28	4.9	16.8	5.3
30	5.3	18.0	5.6

TABLE XXV

PERMEABILITY CONSTANTS x 10^{-6}

Foil	Gas		
	H_2	N_2	B_2H_6
0M4	0.91	0.244	0.252
0M3	2.00	0.535	0.567
0M2	3.20	0.853	0.857
0M1	5.95	1.582	1.682
1	6.17	0.77	2.95
2	3.58	0.514	1.99
3	0.44	0.0433	0.0419
5	0.227	0.38	0.364
6	0.561	0.0321	0.296
7	5.09	0.505	1.65
8	5.08	0.505	1.65
9	0.213	0.0723	0.055
B40	1.235	0.367	0.377
B30	1.72	0.51	0.55
B20	3.64	1.01	1.065
B15	6.59	1.58	1.648
B10	12.11	3.64	3.71

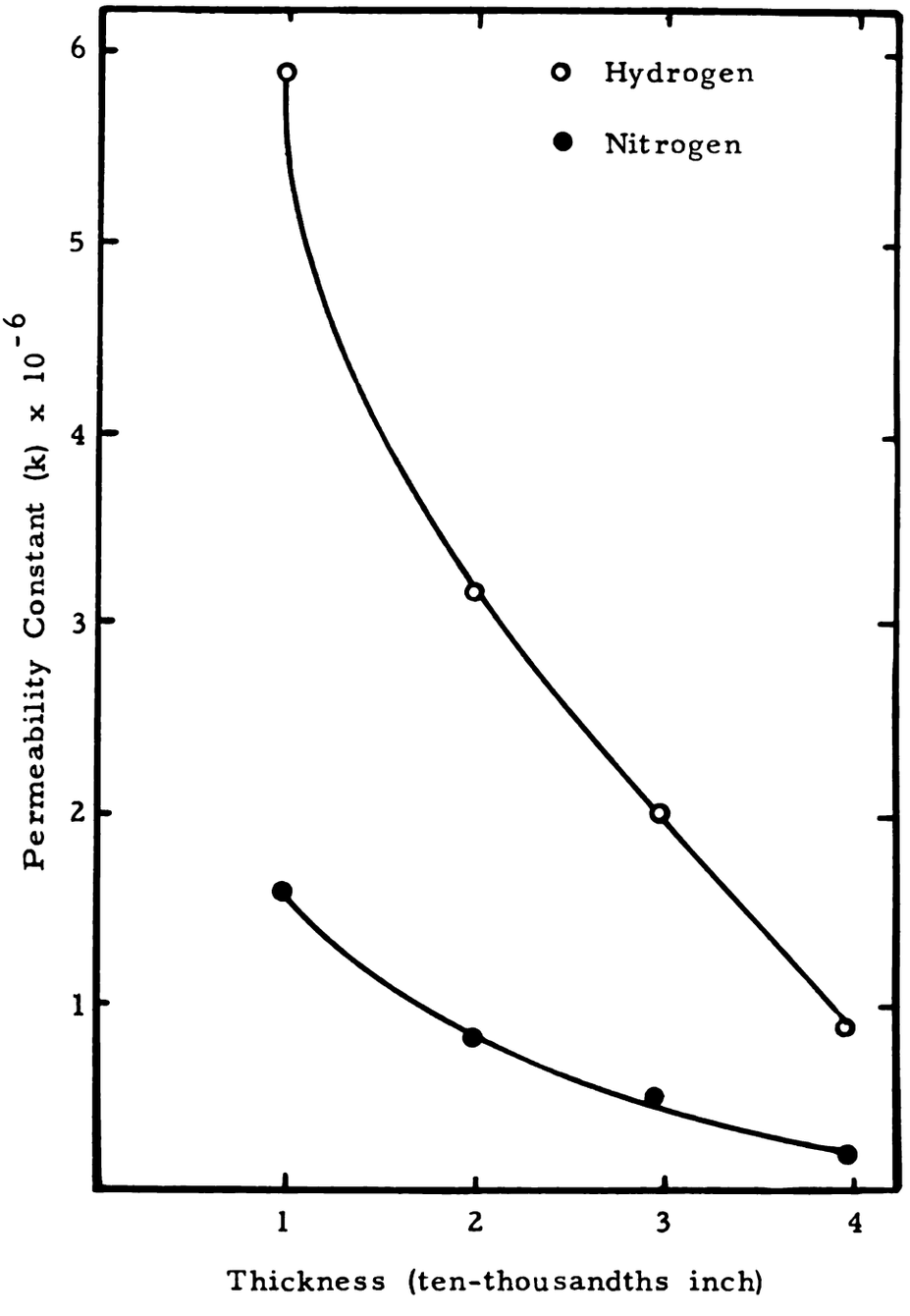


Figure 9. Nickel foils, k versus thickness.

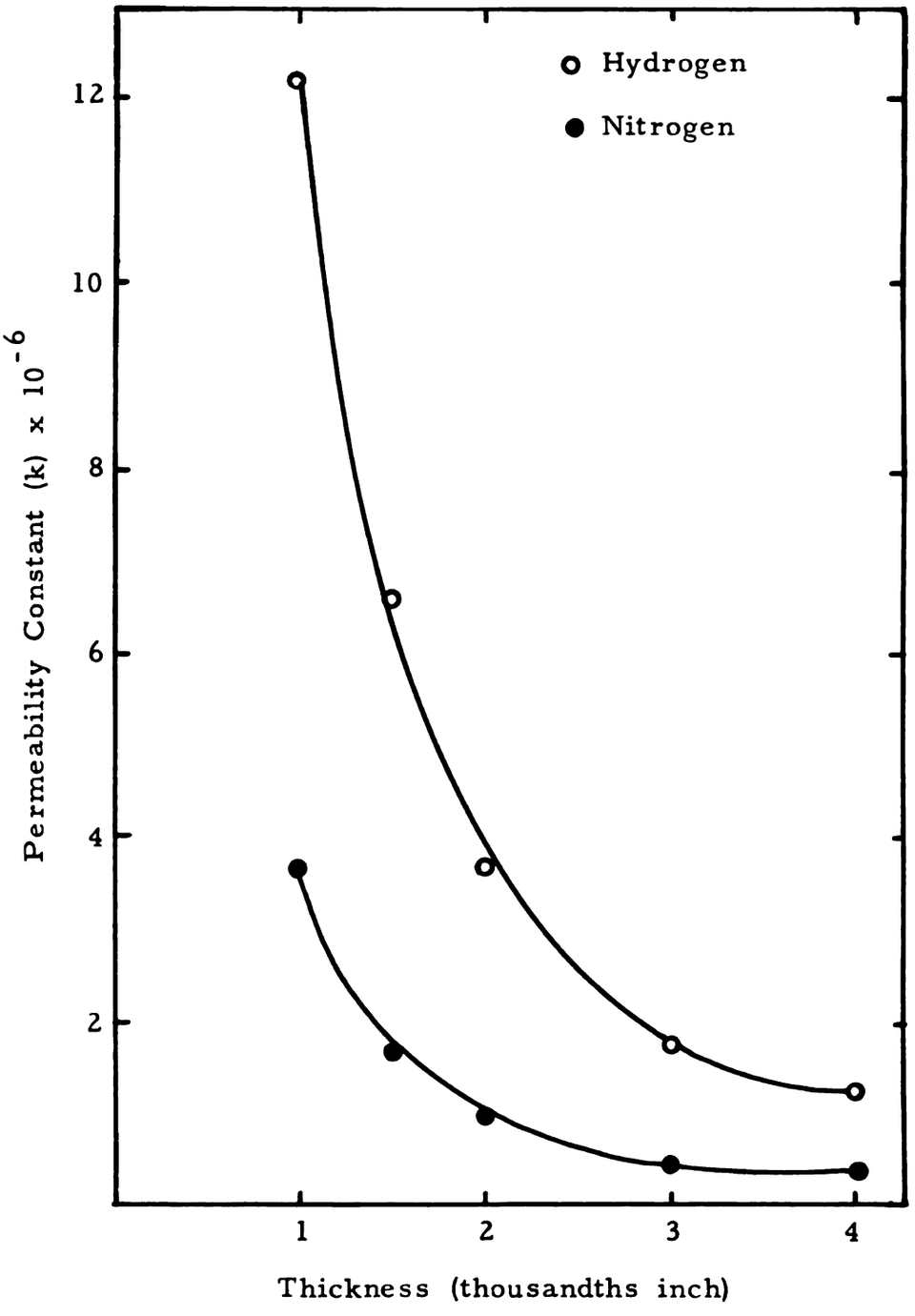


Figure 10. Brass foils, k versus thickness.

Graham's Law

The relationship that the rate of diffusion is inversely proportional to the square root of the molecular weight is known as Graham's Law. Since the rate is proportional to the rate constant,

$$k_1/k_2 = \sqrt{M_2/M_1}$$

Using the rate constant of hydrogen as the known rate constant, the above formula allows the calculation of the rate constants for nitrogen and diborane which can then be compared with the experimental values.

$k_1 = k_2 \sqrt{M_2/M_1}$ $\qquad$ $k_1 = (0.91) \sqrt{2/28}$

$k_1 = 0.243$ calculated $\qquad$ $k_1 = 0.244$ experimental

TABLE XXVI

RATE CONSTANT

Foil	Nitrogen		Diborane	
	Calculated	Experimental	Calculated	Experimental
0M4	0.244	0.243	0.245	0.252
0M3	0.534	0.535	0.537	0.567
0M2	0.855	0.853	0.861	0.857
0M1	1.590	1.582	1.600	1.682
B40	0.330	0.367	0.332	0.377
B30	0.46	0.51	0.463	0.55
B20	0.972	1.01	0.98	1.065
B15	1.76	1.58	1.771	1.648
B10	3.23	3.64	3.26	3.71

TABLE XXVII

SEPARATION OF DIBORANE FROM HYDROGEN OR NITROGEN THROUGH NICKEL FOIL 0M1

Item		Run 1	Run 2	Run 3
P_0	H_2	191 mm	140 mm	92 mm
P_0	B_2H_6	88 mm	143 mm	197 mm
P_0	total	279 mm	283 mm	289 mm
p	B_2H_6	44 μ	72 μ	97 μ
p	total	0.47 mm	0.39 mm	0.31 mm
%	B_2H_6 in P	31.5%	50.6%	68.3%
%	B_2H_6 in p	9.36%	18.45%	31.3%
P_0	N_2	182 mm	146 mm	70 mm
P_0	B_2H_6	91 mm	146 mm	210 mm
P_0	total	273 mm	292 mm	280. mm
p	B_2H_6	46 μ	71 μ	0.10 mm
p	total	0.17 mm	0.14 mm	0.14 mm
%	B_2H_6 in P	33.3%	50.0%	75.1%
%	B_2H_6 in p	27.1%	50.7%	71.4%

TABLE XXVIII

SEPARATION OF DIBORANE FROM HYDROGEN OR NITROGEN THROUGH NICKEL FOIL 0M2

Item		Run 1	Run 2	Run 3
P_0	H_2	190 mm	149 mm	78 mm
P_0	B_2H_6	89 mm	140 mm	205 mm
P_0	total	279 mm	289 mm	283 mm
p	B_2H_6	20 μ	34 μ	49 μ
p	total	0.16 mm	0.14 mm	0.11 mm
%	B_2H_6 in P	31.9%	48.4%	72.4%
%	B_2H_6 in p	12.5%	24.3%	44.6%
P_0	N_2	195 mm	146 mm	75 mm
P_0	B_2H_6	95 mm	146 mm	199 mm
P_0	total	290 mm	292 mm	274 mm
p	B_2H_6	23 μ	36 μ	49 μ
p	total	75 μ	75 μ	69 μ
%	B_2H_6 in P	32.7%	50.0%	72.7%
%	B_2H_6 in p	30.7%	48.0%	71.0%

TABLE XXIX

SEPARATION OF DIBORANE FROM HYDROGEN OR NITROGEN THROUGH NICKEL FOIL 0M3

Item		Run 1	Run 2	Run 3
P_0	H_2	194 mm	127 mm	85 mm
P_0	B_2H_6	93 mm	147 mm	193 mm
P_0	total	287 mm	274 mm	278 mm
p	B_2H_6	15 μ	25 μ	33 μ
p	total	100 μ	80 μ	70 μ
%	B_2H_6 in P	32.4%	53.7%	69.4%
%	B_2H_6 in p	15.0%	31.2%	47.1%
P_0	N_2	189 mm	150 mm	82 mm
P_0	B_2H_6	87 mm	178 mm	201 mm
P_0	total	276 mm	328 mm	283 mm
p	B_2H_6	15 μ	31 μ	32 μ
p	total	46 μ	56 μ	49 μ
%	B_2H_6 in P	31.5%	54.3%	71.0%
%	B_2H_6 in p	32.6%	55.4%	65.3%

TABLE XXX

SEPARATION OF DIBORANE FROM HYDROGEN OR NITROGEN THROUGH NICKEL FOIL 0M4

Item		Run 1	Run 2	Run 3
P_0	H_2	203 mm	140 mm	90 mm
P_0	B_2H_6	85 mm	141 mm	203 mm
P_0	total	288 mm	281 mm	293 mm
p	B_2H_6	6 μ	10 μ	15 μ
p	total	43 μ	33 μ	32 μ
%	B_2H_6 in P	29.5%	50.2%	69.3%
%	B_2H_6 in p	13.9%	30.3%	46.9%
P_0	N_2	206 mm	150 mm	97 mm
P_0	B_2H_6	75 mm	140 mm	200 mm
P_0	total	281 mm	290 mm	297 mm
p	B_2H_6	5.5 μ	10 μ	15 μ
p	total	21 μ	22 μ	23 μ
%	B_2H_6 in P	26.7%	48.3%	71.7%
%	B_2H_6 in p	26.2%	45.5%	65.2%

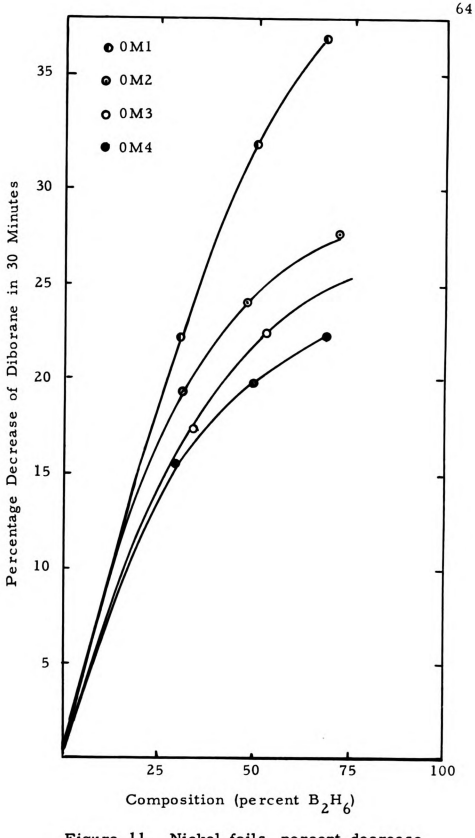
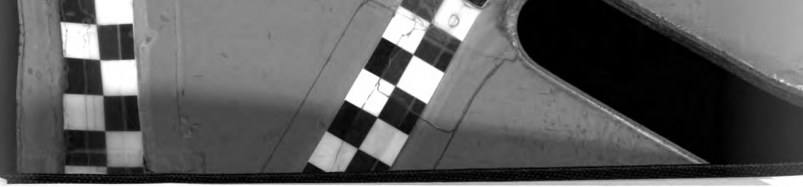


Figure 11. Nickel foils, percent decrease versus composition.

DISCUSSION

The data given are those which list the McLeod pressures versus time readings. These readings are as accurate as the McLeod gage scale. This scale has a different accuracy over different parts of the scale. Below one micron, the scale can be read to the hundredth of a micron; between one micron and ten microns, the scale can be estimated to the nearest tenth of a micron; between ten and one hundred microns, the scale can be estimated to the nearest micron; above one hundred microns, the scale could be estimated to the nearest hundredth of a millimeter of mercury. The gage had three separate capillaries with three overlapping scales to increase the range of pressures which could be read. The consistency between these three scales was within the experimental reading error. The data also include information on the temperature and a correction for the desorption of gas from the walls of the system, p_{30}. This information was not used in this discussion since there was no reason to use the temperature and the correction factor was below the experimental error of each reading. It has been included for the benefit of those who may find a use for this study in the future.



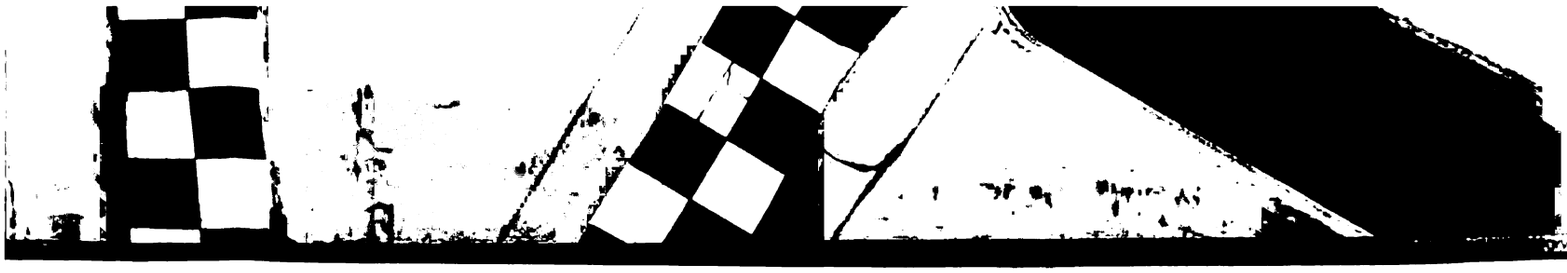
Two sample curves (Figures 3 and 5) of McLeod pressure versus time are included to serve as illustrations. From these curves, the rate of diffusion, expressed as microns per minute, of gas passing through the barrier is determined by the slope. The rate can also be determined by dividing the pressure at time t by that time t. This latter method was favored in some cases where the curve was doubtful. It is noted that for the curve in Figure 3, all of the points fall on the straight line, whereas for the curve in Figure 5, all of the points do not fall on the straight line. The reason for the difference between the curves is in the reading of the McLeod gage itself. In the first case, the actual increase corresponded to the gage scale, whereas in the second case it did not. For example, in the case where the pressure is increasing at 1.5 microns every two minutes and it is possible to read the scale to the nearest micron only, the curve has a slight S shape corresponding to the reading at which the extra half micron was added.

Another error which was encountered at any given reading was the possibility of trapping gas in the lower portions of the McLeod gage. This gas then did not go into the tip and the reading was therefore too low. This error was usually obvious and was corrected by lowering the mercury and running it up again. The

inconsistency of readings was enhanced to some extent by the variation in rate at which the mercury was raised in the McLeod gage; this caused a different pressure in the system itself during readings. It was difficult to take the readings at exactly two-minute intervals, although this was never more than five seconds fast or slow.

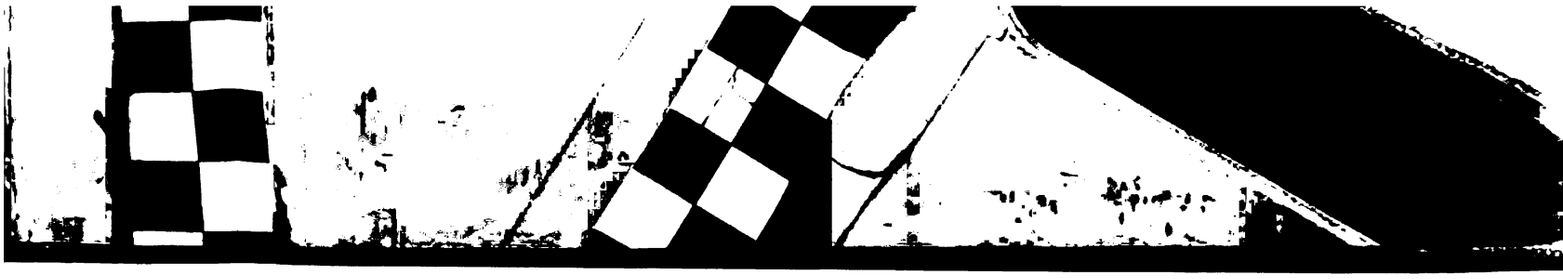
The rate versus overpressure curve (Figures 4, 6, 7, and 8) shows that, just as in the case of p versus t, the curve is linear. This corresponds to equation (6) on page 8. Thus the experimental work verifies the theoretical equation. The rate which was plotted in these curves was determined by taking the slope of the p versus t curve or by dividing the McLeod pressure after thirty minutes by the time, thirty. The method used depended upon the particular run and the nature of the p versus t plot. It can be seen from these curves that hydrogen diffuses faster than nitrogen or diborane, and that the nitrogen and diborane diffuse at approximately the same rate with diborane being the faster. With these curves, as well as others, it is seen that the points do not always fall on the line. The variations involved in these plots are ones involving first the experimental error of reading and then the error of drawing the curves from which the rates were taken.

The theory that diffusion through plastics takes place by a process of solution is partially supported by the work involving plastics in this study. The permeability constants given in Table XXV show rather well that none of these plastics follow Graham's Law of Diffusion. Further investigation of the table which lists the rate constants shows that the rates of diffusion through Trithene and Visqueen are functions of the thickness of the film. It also shows that the Tenite Butyrate is rather consistent from batch to batch. The effect of thickness may be caused by a concentration gradient of gas through the thickness. Otherwise, thickness would not be expected to affect the rate of diffusion, at least in the light of the solubility theory. The values of the constant for the various plastics for the three gases obey no law of diffusion which has its foundation in the kinetic theory of gases. Further study will have to be done to determine the effect of overpressure on the rate and possibly even solubility studies to determine if they do behave in this way. The rate constant was calculated with the aid of equation (6). It cannot be said whether their relationship is valid or not since it is not known whether the rate of diffusion through a plastic is a linear function of the overpressure.



The metal foils, on the other hand, follow Graham's Law to a respectable degree. In those cases where the percentage error is fairly high, the absolute values are rather small. The error involved in the determination of the rate constant is one of plotting graphs as well as one of experimental reading error. The constant is determined from the slope of the rate-versus-overpressure curve for each gas and foil. The constant is equal to the slope times V/A where V is the volume, 1945 ml., of the system on the left, and A is the area of the foil which is exposed to the gas. The agreement between the calculated rate constant and the experimental rate constant is fairly good.

It is observed that, although the porous copper foils were ten times as thick as the nickel foils, they were appreciably more permeable. These foils seem to be the most promising diffusion barriers studied. It was also noted during the preparation of the porous copper foils that the size of the foils actually decreased. It was surmised from this fact and from the rate at which the zinc was distilled out of the brass that structural changes had taken place during the intense heating. These changes very probably decreased the permeability. The consistency observed in this study is due to a standardization of the treatment of the shim stock so that each



copper foil was produced in exactly the same manner. The temperature was maintained at the same point for the same length of time. It is believed that the permeability could be considerably increased by distilling the zinc out very slowly at a temperature just above the temperature of melting zinc.

The curves which show the relation between the rate constant and the thickness of both nickel and brass are seen to be somewhat exponential in form. As expected, the thinner foils have higher rate constants. The points fall on the line in every case because there are only four or five points to determine the curve and the curve is not a straight line. It was therefore quite simple to hit all of the points with the curve. Because all of the points fall on the curve does not mean that this curve is more accurate than the linear curve. In fact, these curves are subject to the same sources of error as the linear curves. The logarithmic or exponential form of the curve might also be expected since, as the thickness decreases, the rate should approach that of the unhindered molecule, unhindered even by surrounding gas molecules, since it is diffusing into a vacuum.

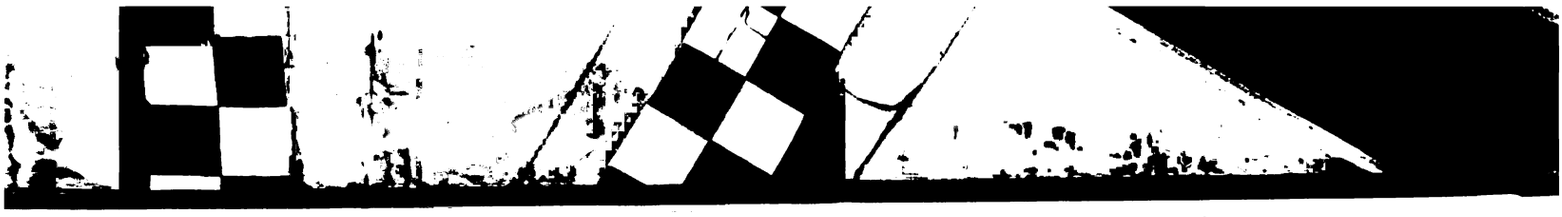
Data were also determined for some separations. It is well known that the rates of diffusion of the components of a mixture are

determined by their individual partial pressure, a fact borne out by the experimental results. The total rate of diffusion of a mixture is equal to the sum of the rates of the individual components of the mixture. As can be observed from Figure 11, the separation of hydrogen from diborane was quite simple and effective. The separation of nitrogen from diborane is an entirely different problem, however. As seen from Figures 4, 6, 7, and 8, and as would be judged from their relative molecular weights, the rates of diffusion of these two gases are very close, and a separation due to this difference is quite difficult in a single-stage operation. The individual rates as determined separately show diborane to diffuse slightly faster than nitrogen, which is expected from Graham's Law. Also, it is noted that the rate for diborane, in addition to being faster than that of nitrogen, is a little faster than predicted by the law. This behavior may be attributed to a slight amount of hydrogen in equilibrium with the diborane or else a slight decomposition of the diborane by the metal foil. The separations of the mixtures, however, show a little less diborane than is expected on the left side of the system after thirty minutes, which may be explained by reviewing the method of analysis. The diborane is frozen out with liquid nitrogen and the other gas is pumped off. In this process, some diborane is undoubtedly

pumped off even though the vapor pressure of diborane at this temperature is less than a micron, as determined by actual measurement. These results clearly indicate that the diffusion of gases through porous metal foils follow Graham's Law.

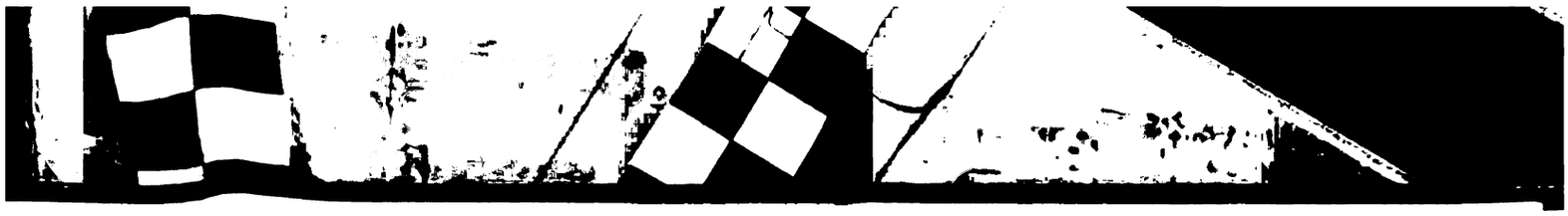
Figure 11 shows the relationship between the composition of the diborane-hydrogen mixtures before and after diffusion in terms of the percentage of diborane present in the initial mixtures. It is noted from this relationship that the percentage of hydrogen with respect to diborane increases with time, which means that, as time proceeds, the effectiveness of the separation increases. In a series of passes, the faster molecule will tend to get away from the slower one, and if enough stages are used, the final stage could be expected to contain pure hydrogen.

In addition to the experimental results listed above, certain observations of a negative character were made. A sample of a porous brass disc produced by the powdered-metallurgy method was obtained from the Precision Metal Products Company. This disc proved to be much too porous for the diffusion studies in this problem. Actually, cigarette smoke could be blown through the disc with little effort.

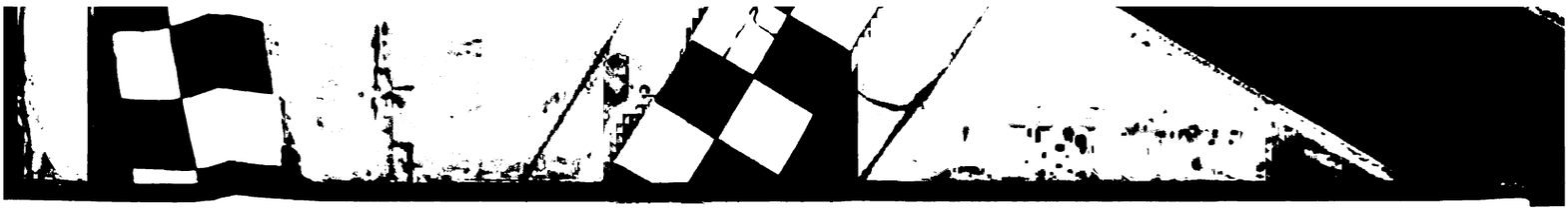


A great deal of time was spent on a plating bath (16) containing nickel sulfate, stannous chloride, and ammonium bifluoride with the intention of obtaining a nickel-tin alloy foil from which the tin could be extracted. Even after it was discovered that the fluoride concentration was critical and that it was necessary to use a polyethylene beaker, the deposits were brittle and stripping was impossible. This method of making a porous deposit was discontinued because of the inability to strip foils of this deposit.

The plating conditions of the Watt's bath were varied over a considerable range. The temperature was varied between 25°C. and 85°C., the current density was varied between 10 and 70 amps per square foot, and the pH was varied between 1.0 and 5.2. It was observed that the standard plating conditions, current density of 40 amps per square foot, temperature of 55°C., and pH of either 2.2 or 5.2 are the only conditions which result in deposits which can be stripped and used as foils. The deposits which were plated at conditions other than standard were generally brittle and under considerable stress. The stress pulled the foil away from the base panel and the brittleness caused the foil to break while in the process of plating.



At one point in the experiment, there was an accidental explosion of diborane. Inexperience can be blamed for storing diborane on one side of a foil and air on the other side. This was done to allow the glass-blower to work on the side which was open to the atmosphere. After a lapse of time, an explosion occurred on the diborane side or left side of the system due to the diffusion of oxygen from the air into the diborane. This immediately suggests a rather satisfactory method of determining the explosion limits of diborane or any other gas which explodes when in contact with air.



CONCLUSIONS

1. The original purpose of the problem was to effect a separation (13) of diborane from hydrogen and nitrogen. The nature of the problem required only that an enrichment of one constituent over the other be obtained to satisfy the purpose of the problem. With regard to hydrogen, this purpose has been quite satisfactorily fulfilled. With nitrogen, the results are indefinite, but since they follow Graham's law, it is indicated that, even in this case, an enrichment could be effected with a sufficient number of passes (7) through a barrier.

2. These separations could even be put on a commercial scale (4, 6, 11) using these foils. In order to render the separation more economical, more porous foils would have to be found.

3. The rate of diffusion of a gas through a metal barrier is a linear function of the overpressure at least up to a pressure of three hundred millimeters of mercury.

4. The McLeod pressure is a linear function of the time regardless of the type of barrier used.

5. Graham's law of diffusion does not apply in the case of plastics (15) since the rates are not related to the molecular weight

of the gases being used. This offers a more efficient separation of diborane from hydrogen and nitrogen, although it may not be more economical.

6. Distillation of zinc from brass gives a copper foil which is more porous than an ordinary metal foil or an electrodeposited foil. It is suspected that the porosity can be controlled within very close limits.

7. In the separation of hydrogen from diborane, the separation becomes more effective as time passes and as the concentration of hydrogen increases in the product.

8. It was not possible to prepare a nickel-tin alloy foil.

9. The only satisfactory nickel foils were obtained from a Watt's bath at a pH of 2.2, a current density of 40 amps per square foot, and a temperature of 55°C.

RECOMMENDATIONS

1. The foil holder of the diffusion apparatus should be redesigned to so hold the foil such that pressures of an atmosphere or more might be used. It might prove worth while to increase the area by a large factor. The foil might have to be supported by some sort of crosshatch network.

2. Some method for the analysis of mixtures should be worked out to give a more accurate determination of the mixture. A sample tube which is removable could be used. This tube could be made of silica or NaCl, and an infrared spectrophotometric determination could be used or a discharge emission spectra could be used. This would not be as good because of the decomposition of the diborane causing a residue to be left in the tube. It might even be possible to hydrolyze the diborane and titrate the boric acid.

3. A Toeppler pump should be placed on the right side to increase the efficiency of mixing and to insure that the mixture is thoroughly mixed.

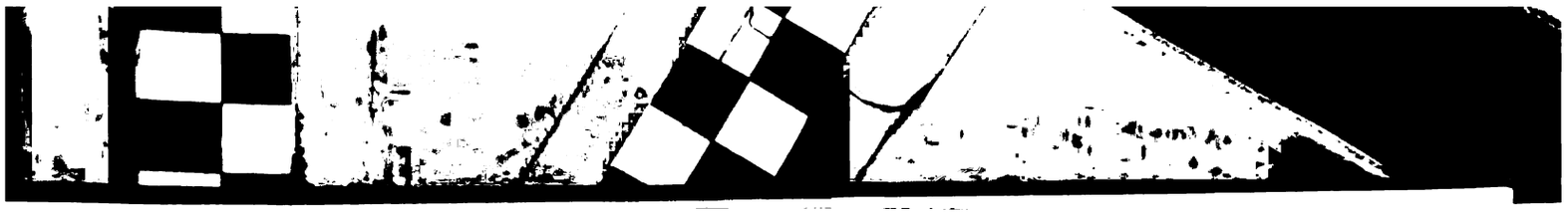
4. It should be determined if there is any decomposition of diborane as it passes through the barrier. The results of this study indicate that there was a slight decomposition. If the method of

analysis were accurate enough, as those listed under number two are, this could be determined simply by analysis.

5. Plastics should be subjected to a complete study of diffusion. Emphasis should be placed on membranes which are more porous than those used in this experiment. It could prove, however, by using thinner membranes, that these give the most efficient and economical separation. Many more types and samples of plastics should be studied also.

6. Porous copper foils obtained by distilling zinc out of brass should be studied further. Foils which are about one-tenth as thick should be used if they are available. The method of distilling the zinc out should be altered, also. In this experiment, the zinc was distilled at a relatively high temperature. This caused the zinc to come out quite rapidly and also caused a decrease in the over-all size of the foil. This indicates that there were some structural changes taking place. These should be avoided by distilling at a temperature just above the melting point of the zinc so that the distillation takes place slowly and the structure of the remaining copper is not changed. This should result in maximum porosity.

7. Other alloys could be investigated for the possibility of removing one of the constituents to leave a porous foil. It may be

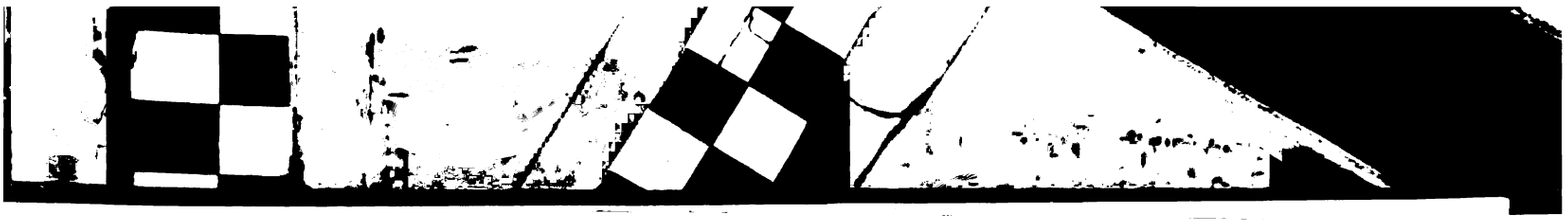


possible to remove the constituent by chemical means or by diffusing it out under the influence of an applied electromotive force in the appropriate solution.

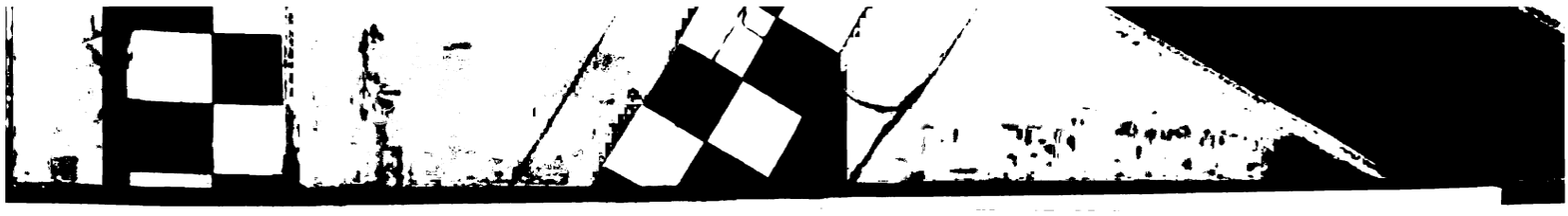
8. Porous nickel foils could be plated by adding colloidal graphite (8) to the plating solution. This requires an experienced electroplater and the foils which result may even be of the visible pore variety.

9. If the porosity has not been increased sufficiently after the suggestions given above have been investigated, there is the possibility of studies using ceramics, powdered-metallurgy discs, and sintered glass. The preparation of these materials requires an expert in these fields and should not be attempted by anyone who is unfamiliar with the techniques involved. The commercial barriers of these materials are too porous for the studies involving separations of gases.

10. When a suitable barrier has been found, one which could be used in the commercial separation and purification of gases, a multiple-stage diffusion (11) system should be set up to study the effectiveness of separation after any number of passes through the barrier. This would correspond to a pilot plant.



11. If the system was commercially feasible, consideration should be given to the possibility of substituting neon for nitrogen as an inert atmosphere in the manufacture of diborane, since the neon would be recovered in the separation and it would afford a much more economical separation due to the difference in molecular weight of neon and diborane in contrast to the difference between nitrogen and diborane.



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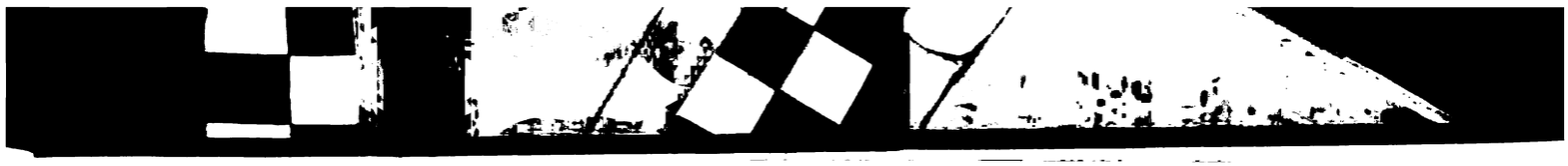
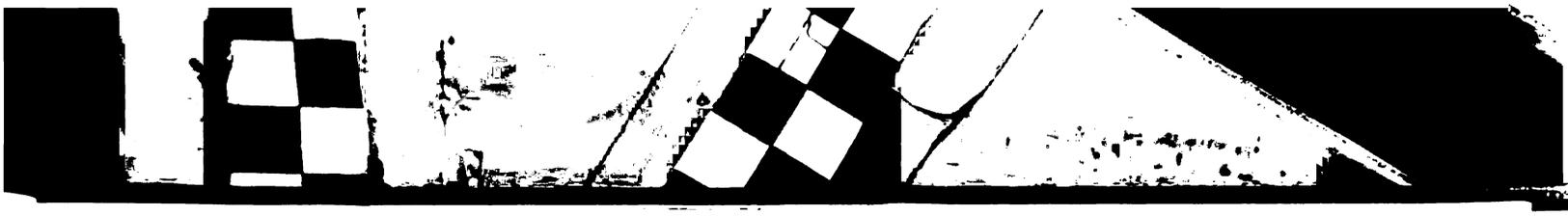


APPENDIX



The material in this appendix is presented as a matter of record. It represents data which were obtained on another project on which the author worked. The data are of a preliminary nature and are not conclusive in any way. The project was discontinued because it was obvious after some time that the expected results were not forthcoming.

The experiment consisted in the determination of the E.M.F. of electrolytic nickel. One half cell contained a nickel wire which had been nickel plated and which was immersed in a solution of nickel sulfate. The other half cell contained mercury, mercurous sulfate, and nickel sulfate. The salt bridge was of nickel sulfate. Air was found to be detrimental to the readings, so an atmosphere of nitrogen was used at all times. The measurements were made on both dull and bright plated nickel wires, as well as on the base nickel metal and on powdered nickel. Powdered nickel is the accepted standard and the electrolytic nickel is seen to approach this after standing in contact with nickel sulfate solution for some time.

TABLE XXXI

E.M.F. OF NICKEL WIRES PLATED WITH WATT'S BATH, pH 2.2, AND IMMERSED IN 1.0 MOLAR $NiSO_4$ MEASURED AGAINST $Hg:Hg_2SO_4$

Item	Wire Number			
	1	2	3	4
Plating time (minutes)	15	30	45	60
Thickness (inches)	0.0005	0.0010	0.0015	0.0020
E.M.F. under nitrogen before plating	0.71774	0.73584	0.75051	0.70449
E.M.F. under nitrogen after plating, tip immersed.	0.63210	0.64633	0.67085	0.67313
E.M.F. under air, after plating, tip immersed	0.65067	0.64058	0.66242	0.66876
E.M.F. under nitrogen, tip covered with paraffin . .	0.68049	0.66132	0.66480	0.68493
E.M.F. after acid dip, tip covered with paraffin . .	0.72268	0.67809	0.67596	0.67858
E.M.F. after three days, tip covered with paraffin . .	-	-	-	-
Bent above paraffin, tip covered with paraffin	0.69184	0.64377	0.66569	0.65948

TABLE XXXI (Continued)

Item	Wire Number			
	5	6	7	8
Plating time (minutes)	75	90	105	120
Thickness (inches)	0.0025	0.0030	0.0035	0.0040
E.M.F. under nitrogen before plating	0.73989	0.73311	0.74375	0.72120
E.M.F. under nitrogen after plating, tip immersed .	0.63846	0.64755	0.63935	0.64465
E.M.F. under air, after plating, tip immersed	0.61583	0.65349	0.64697	0.63512
E.M.F. under nitrogen, tip covered with paraffin . .	0.65301	0.66101	0.65727	0.68971
E.M.F. after acid dip, tip covered with paraffin . .	0.66493	0.69015	0.66938	0.67874
E.M.F. after three days, tip covered with paraffin . .	0.73727	0.72908	0.72123	0.70138
Bent above paraffin, tip covered with paraffin	0.66756	0.65507	0.63728	0.66142

TABLE XXXII

E.M.F. UNDER NITROGEN OF THE CELL Ni:$NiSO_4$(0.1 M)::
$NiSO_4$(0.1 M):Hg_2SO_4:Hg VERSUS TIME

Wire No.	Time (in minutes)				
	0	2	5	10	15
Dull nickel:					
1	0.58298	0.59834	0.59645	0.59118	0.59103
2	0.55751	0.54073	0.54062	0.55050	0.57542
3	0.54545	0.56748	0.59236	0.61884	0.63307
4	0.53875	0.58943	0.59544	0.59625	0.59556
5	0.50362	0.54111	0.55569	0.56462	0.56872
6	0.54119	0.57203	0.57718	0.58361	0.58647
7	0.52670	0.53422	0.53619	0.54282	0.55213
8	0.52748	0.54193	0.55285	0.56087	0.56586
Bright nickel:					
9	0.69850	0.74808	0.72855	0.70675	0.69271
10	0.75360	0.75753	0.73558	0.71647	0.70100
11	0.62987	0.74921	0.73527	0.71876	0.70738
12	0.61761	0.73635	0.72415	0.71100	0.69895
13	0.67821	0.73998	0.73336	0.71807	0.70371
14	0.63872	0.73497	0.73207	0.71627	0.70359
15	0.64781	0.74149	0.73740	0.71273	0.69902
16	0.64793	0.73985	0.73086	0.71875	0.70887

Wir

Dull nic

1 . .

2 . .

3 . .

4 . .

5 . .

6 . .

7 . .

8 . .

Bright n

9 . .

10 . .

11 . .

12 . .

13 . .

14 . .

15 .

16 .

TABLE XXXII (Continued)

Wire No.	Time (in minutes)				
	20	25	30	35	40
Dull nickel:					
1	0.59093	-	0.58817	0.58748	0.58725
2	0.59518	0.60440	0.60914	-	0.61456
3	0.64177	0.64205	0.63987	0.63811	0.63655
4	0.59539	0.59509	0.59529	0.59636	0.59796
5	0.57224	0.57524	0.58049	0.58685	0.59312
6	0.58725	0.58713	0.58679	0.58666	0.58700
7	0.56323	0.57816	0.58467	0.59017	0.59491
8	0.57363	0.58417	0.59015	0.59291	0.59493
Bright nickel:					
9	0.68383	0.67692	0.67092	0.66646	0.66394
10	0.69000	0.67795	0.66864	0.66007	0.65195
11	0.69743	0.68885	0.68094	0.67325	0.66779
12	0.69050	0.68357	0.67764	0.67197	0.66913
13	0.69061	0.68112	0.67468	0.67067	0.66880
14	0.69416	0.68607	0.67920	0.67419	0.66903
15	0.68969	0.68105	0.67374	0.66680	0.66244
16	0.70032	0.69351	0.68719	0.68358	0.68018

TABLE XXXII (Continued)

Wire No.	Time (in minutes)				
	45	50	55	60	1440
Dull nickel:					
1	0.58712	0.58753	0.58834	0.58960	0.65231
2	-	0.61998	-	0.62479	0.63972
3	0.63448	0.63246	0.63017	0.62728	0.60300
4	0.59989	0.60213	0.60443	0.60672	0.65933
5	0.59833	0.60275	0.60660	0.60985	0.64169
6	0.58787	0.58891	0.59102	0.59354	0.63488
7	0.59842	0.60153	0.60372	0.60591	0.65872
8	0.59665	0.59950	0.60217	0.60490	0.67261
Bright nickel:					
9	0.66160	0.65854	0.65606	0.64826	0.68174
10	0.64447	0.63893	0.63471	0.62751	0.64211
11	0.66240	0.65838	0.65557	0.65224	0.65951
12	0.66568	0.66219	0.66010	0.65534	0.63752
13	0.66706	0.66597	0.66414	0.66216	0.78029
14	0.66576	0.66284	0.65958	0.65691	0.77573
15	0.65793	0.65412	0.65195	0.64727	0.78199
16	0.67809	0.67500	0.67261	0.67079	0.77593

TABLE XXXIII

E.M.F. UNDER NITROGEN OF THE CELL Ni:$NiSO_4$(0.1 M)::
$NiSO_4$(0.1 M):Hg_2SO_4:Hg VERSUS TIME
(selected wires from Table XXVII)

Date	Base Metal Nickel	Dull Nickel			
		1	3	4	8
8/18	0.40861	0.76193	0.56711	0.52617	0.57424
9/24	0.38244	0.87338	0.81785	0.83865	0.82515
9/25	0.38384	0.87917	0.85128	0.81800	0.80908
9/26	0.38253	0.85455	0.85904	0.82322	0.80382
9/30	0.38232	0.85657	0.82563	0.83984	0.81531
10/1	0.40455	0.86027	0.84055	0.83027	0.82163
10/3	0.38362	0.85239	0.83116	0.81790	0.80680
10/4	0.38401	0.86112	0.83161	0.82153	0.81491
10/5	0.38430	0.85907	0.82725	0.82195	0.81967
10/6	0.38421	0.86337	0.82495	0.81829	0.82039
10/7	0.38393	0.86139	0.82770	0.82073	0.83956
10/15	0.38965	0.87113	0.84012	0.82787	0.83920
10/20	0.38738	0.87393	0.83237	0.82586	0.84339

TABLE XXXIII (Continued)

Date	Base Metal Nickel	Bright Nickel			
		9	12	15	16
8/18	0.40861	0.72213	0.74269	0.78244	0.78170
9/24	0.38244	0.78718	0.86334	0.85505	0.86193
9/25	0.38384	0.80731	0.86825	0.85064	0.87041
9/26	0.38253	0.78679	0.86361	0.84069	0.86989
9/30	0.38232	0.80254	0.86271	0.85290	0.84533
10/1	0.40455	0.80019	0.87116	0.85319	0.84853
10/3	0.38362	0.80792	0.86410	0.85549	0.85043
10/4	0.38401	0.80639	0.86368	0.85798	0.85290
10/5	0.38430	0.80651	0.86802	0.85881	0.85050
10/6	0.38421	0.81350	0.86882	0.85952	0.85032
10/7	0.38393	0.81339	0.86643	0.86119	0.85707
10/15	0.38965	0.80826	0.85870	0.86537	0.86061
10/20	0.38738	0.80620	0.85529	0.86088	0.86201

TABLE XXXIV

E.M.F. UNDER NITROGEN OF THE CELL Ni:$NiSO_4$(0.1053 M)::
$NiSO_4$(0.1053 M):Hg_2SO_4:Hg VERSUS TIME

Date	Powdered Nickel + Pt	D1	D2	D3	D4
2/27	0.96007	0.84672	0.84240	-	-
3/17	0.95755	0.83163	0.84268	0.81937	0.84306
3/19	0.95754	0.83950	0.84154	0.83021	0.85572
3/20	0.95756	0.83926	0.85253	0.83398	0.85563
3/24	0.95673	0.85316	0.87065	0.84190	0.87785
4/1	0.95761	0.85853	0.87966	0.84894	0.90117
4/3	0.95653	0.86056	0.88107	0.85243	0.90899
4/6	0.95576	0.85801	-	0.86249	0.91728

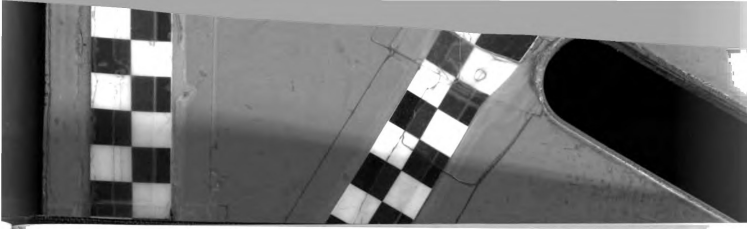
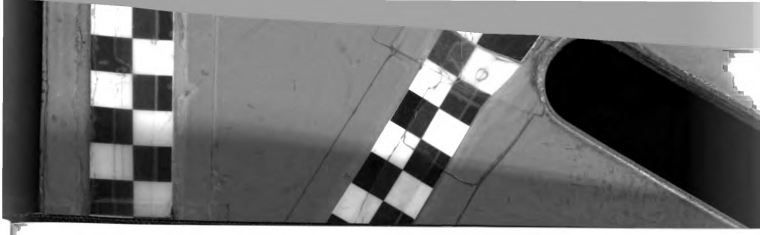


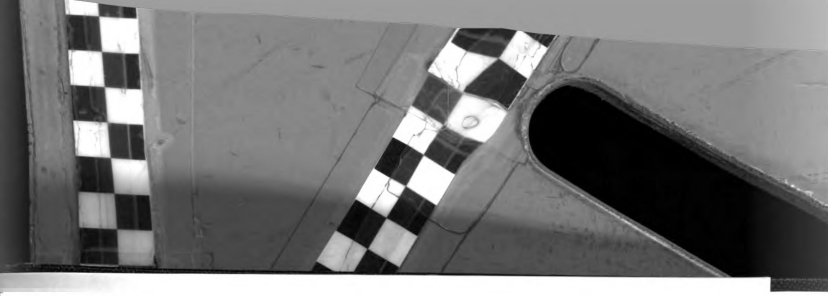
TABLE XXXIV (Continued)

Date	Powdered Nickel + Pt	D5	D6	D7	D8
2/27	0.96007	-	-	-	0.85040
3/17	0.95755	0.85739	0.87597	0.87085	0.72436
3/19	0.95754	0.87275	0.88027	0.88593	-
3/20	0.95756	0.87373	0.88137	0.89411	-
3/24	0.95673	0.88679	0.89815	0.91189	-
4/1	0.95761	0.89117	0.90851	0.91506	0.83792
4/3	0.95653	0.89324	0.91096	0.91506	0.83540
4/6	0.95576	0.89515	0.91134	0.91589	0.84371



SUPPLEMENT I

DATA AND CONCLUSIONS ON SILICONE-TYPE PLASTICS



The following data and conclusions on silicone-type plastic membranes were assembled after completion of the thesis. Two were Silastic 50 of 0.001 and 0.0005 inch thicknesses, and one was Silastic 80 of 0.001 inch thickness. All three films were subjected to the diffusion of diborane, hydrogen, and nitrogen at four different overpressures. Time did not permit studies of any mixtures.

TABLE XXXV

McLEOD PRESSURE VERSUS TIME FOR SILASTIC FOIL S80-001 FOR HYDROGEN AT VARIOUS OVERPRESSURES
(pressures in microns, except as noted; time in minutes)

Item	Overpressure			
	272	226	186	110
P_0	0.03	0.04	0.03	0.05
P_{30}	0.7	0.75	0.7	0.7
t°C	25.0	25.9	25.1	25.1
Time 2	42	42	38	29.5
4	0.11*	0.11*	92	64
6	0.17*	0.16*	0.14*	0.11*
8	0.23*	0.21*	0.18*	0.13*
10	0.28*	0.25*	0.23*	0.16*
12	0.32*	0.30*	0.27*	0.19*
14	0.37*	0.35*	0.30*	0.22*
16	0.43*	0.39*	0.34*	0.24*
18	0.48*	0.44*	0.38*	0.27*
20	0.53*	0.48*	0.42*	0.29*
22	0.58*	0.53*	0.47*	0.31*
24	0.63*	0.58*	0.51*	0.34*
26	0.68*	0.63*	0.54*	0.37*
28	0.73*	0.67*	0.58*	0.46*
30	0.78*	0.72*	0.62*	0.42*

* Pressure in millimeters.

TABLE XXXVI

McLEOD PRESSURE VERSUS TIME FOR SILASTIC FOIL S80-001 FOR NITROGEN AT VARIOUS OVERPRESSURES
(pressure in microns, except as noted; time in minutes)

Item	Overpressure			
	267	192	133	90
p_0	0.08	0.03	0.05	0.05
p_{30}	0.75	0.72	0.75	0.75
t°C	28.0	26.3	27.9	26.6
Time 2	11.0	9.6	10.0	7.6
4	32	27.9	21.1	20.0
6	55	45	42	33
8	82	66	58	44
10	0.12*	89	72	55
12	0.14*	0.11*	86	69
14	0.17*	0.13*	0.10*	81
16	0.19*	0.15*	0.11*	93
18	0.20*	0.17*	0.13*	0.11*
20	0.22*	0.19*	0.14*	0.12*
22	0.25*	0.20*	0.16*	0.13*
24	0.27*	0.22*	0.17*	0.14*
26	0.29*	0.23*	0.19*	0.15*
28	0.31*	0.25*	0.20*	0.17*
30	0.33*	0.27*	0.22*	0.18*

* Pressure in millimeters.

TABLE XXXVII

McLEOD PRESSURE VERSUS TIME FOR SILASTIC FOIL S80-001 FOR DIBORANE AT VARIOUS OVERPRESSURES
(pressure in microns, except as noted; time in minutes)

Item	Overpressure			
	276	201	130	64
p_0	0.10	0.10	0.04	0.10
p_{30}	0.70	0.73	0.70	0.70
t°C	25.7	26.6	25.9	26.1
Time 2	93	75	64	48
4	0.20*	0.18*	0.15*	99
6	0.31*	0.27*	0.22*	0.15*
8	0.41*	0.36*	0.28*	0.19*
10	0.52*	0.45*	0.35*	0.22*
12	0.62*	0.54*	0.41*	0.26*
14	0.72*	0.63*	0.49*	0.30*
16	0.82*	0.72*	0.55*	0.33*
18	0.92*	0.81*	0.61*	0.37*
20	1.02*	0.90*	0.68*	0.40*
22	1.12*	0.99*	0.74*	0.44*
24	1.22*	1.08*	0.80*	0.48*
26	1.32*	1.17*	0.87*	0.52*
28	1.42*	1.26*	0.93*	0.56*
30	1.52*	1.35*	0.99*	0.59*

* Pressure in millimeters.

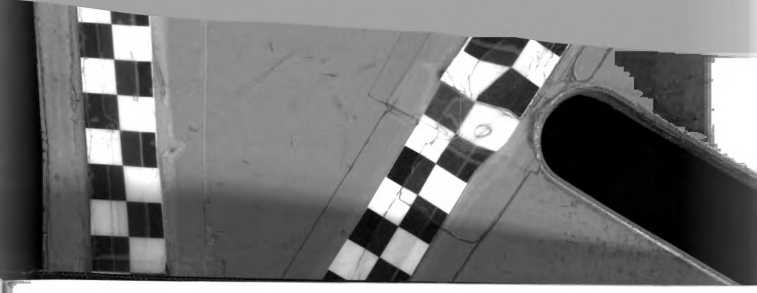


TABLE XXXVIII

McLEOD PRESSURE VERSUS TIME FOR SILASTIC FOIL S50-0005
FOR HYDROGEN AT VARIOUS OVERPRESSURES
(pressure in microns, except as noted; time in minutes)

Item	Overpressure			
	262	170	97	47
P_0	0.02	0.03	0.03	0.05
P_{30}	0.70	0.70	0.70	0.70
t°C	24.9	24.8	25.0	25.0
Time 2	0.27*	0.22*	0.13*	68
4	0.52*	0.40*	0.25*	0.14*
6	0.77*	0.58*	0.36*	0.20*
8	1.02*	0.76*	0.47*	0.25*
10	1.27*	0.95*	0.57*	0.29*
12	1.52*	1.12*	0.68*	0.34*
14	1.78*	1.30*	0.78*	0.39*
16	2.03*	1.48*	0.89*	0.45*
18	-	1.66*	0.99*	0.50*
20	-	1.83*	1.10*	0.55*
22	-	2.00*	1.20*	0.60*
24	-	-	1.31*	0.65*
26	-	-	1.41*	0.70*
28	-	-	1.52*	0.75*
30	-	-	1.62*	0.80*

* Pressure in millimeters.

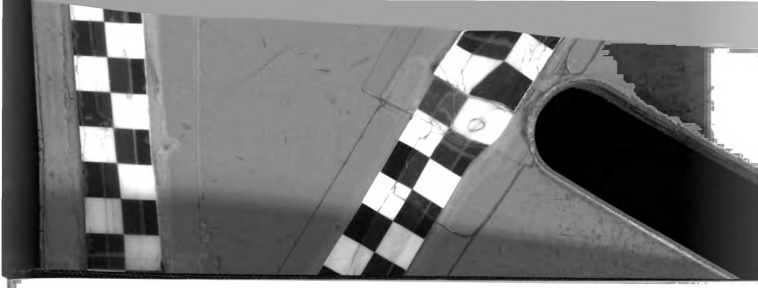


TABLE XXXIX

McLEOD PRESSURE VERSUS TIME FOR SILASTIC FOIL S50-0005 FOR NITROGEN AT VARIOUS OVERPRESSURES
(pressure in microns, except as noted; time in minutes)

Item	Overpressure			
	275	189	111	61
p_0	0.01	0.05	0.03	0.04
p_{30}	0.80	0.70	0.65	0.60
t°C	26.6	23.9	23.0	22.5
Time 2	0.12*	75	50	28.1
4	0.23*	0.17*	0.12*	62
6	0.35*	0.24*	0.17*	99
8	0.47*	0.32*	0.22*	0.13*
10	0.58*	0.40*	0.27*	0.16*
12	0.70*	0.48*	0.32*	0.19*
14	0.81*	0.54*	0.37*	0.22*
16	0.92*	0.61*	0.42*	0.24*
18	1.04*	0.69*	0.47*	0.27*
20	1.16*	0.76*	0.52*	0.30*
22	1.28*	0.83*	0.57*	0.33*
24	1.39*	0.91*	0.62*	0.36*
26	1.50*	0.98*	0.67*	0.38*
28	1.62*	1.05*	0.72*	0.41*
30	1.73*	1.12*	0.77*	0.44*

* Pressure in millimeters.

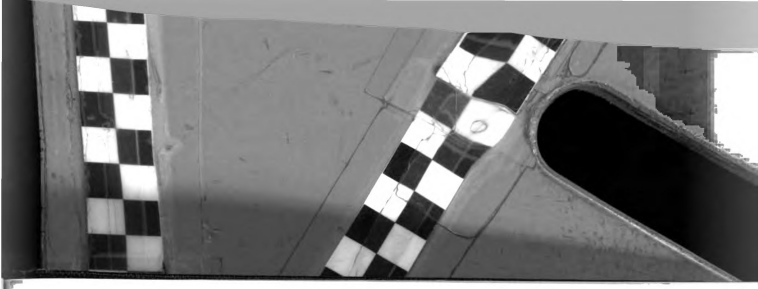


TABLE XL

McLEOD PRESSURE VERSUS TIME FOR SILASTIC FOIL S50-0005 FOR DIBORANE AT VARIOUS OVERPRESSURES
(pressure in microns, except as noted; time in minutes)

Item	Overpressure			
	285	213	132	71
P_0	0.04	0.03	0.10	0.10
P_{30}	0.85	0.70	0.80	0.75
t°C	29.0	24.0	27.6	27.1
Time 2	0.37*	0.23*	0.14*	72
4	0.71*	0.47*	0.27*	0.16*
6	1.04*	0.70*	0.40*	0.23*
8	1.37*	0.92*	0.52*	0.29*
10	1.69*	1.13*	0.65*	0.36*
12	2.02*	1.37*	0.77*	0.42*
14	-	1.58*	0.89*	0.49*
16	-	1.81*	1.01*	0.55*
18	-	2.04*	1.13*	0.62*
20	-	-	1.25*	0.69*
22	-	-	1.37*	0.75*
24	-	-	1.49*	0.82*
26	-	-	1.60*	0.89*
28	-	-	1.72*	0.95*
30	-	-	1.84*	1.02*

* Pressure in millimeters.

TABLE XLI

McLEOD PRESSURE VERSUS TIME FOR SILASTIC FOIL S50-001 FOR HYDROGEN AT VARIOUS OVERPRESSURES
(pressure in microns, except as noted; time in minutes)

Item	Overpressure			
	282	201	112	50
p_0	0.03	0.05	0.05	0.05
p_{30}	0.70	0.70	0.75	0.75
t°C	25.6	25.9	26.3	26.9
Time 2	37	21.7	11.9	7.0
4	75	54	34	21
6	0.14*	89	53	33
8	0.18*	0.13*	76	43
10	0.22*	0.16*	98	55
12	0.26*	0.19*	0.12*	67
14	0.30*	0.21*	0.13*	78
16	0.34*	0.24*	0.15*	89
18	0.38*	0.27*	0.18*	0.11*
20	0.42*	0.29*	0.19*	0.12*
22	0.46*	0.32*	0.21*	0.13*
24	0.50*	0.34*	0.22*	0.14*
26	0.54*	0.37*	0.24*	0.15*
28	0.58*	0.40*	0.26*	0.16*
30	0.62*	0.42*	0.27*	0.17*

* Pressure in millimeters.

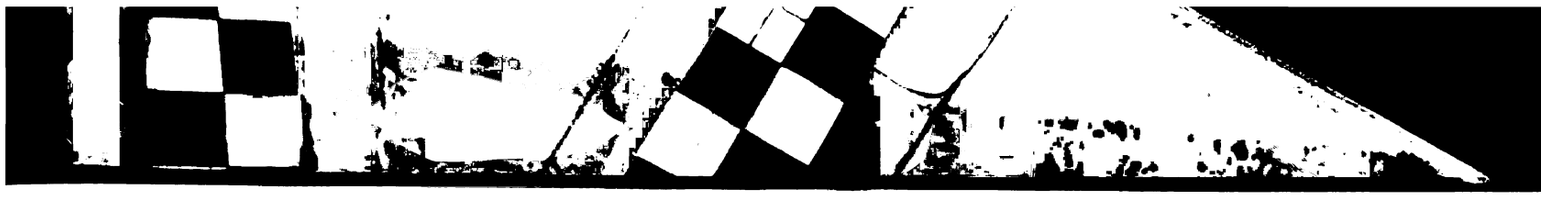


TABLE XLII

McLEOD PRESSURE VERSUS TIME FOR SILASTIC FOIL S50-001 FOR NITROGEN AT VARIOUS OVERPRESSURES
(pressure in microns, except as noted; time in minutes)

Item	Overpressure			
	274	194	120	47
P_0	0.05	0.10	0.10	0.09
P_{30}	0.75	0.75	0.75	0.75
t°C	26.7	27.0	26.9	26.9
Time 2	8.1	5.4	3.7	4.4
4	22.1	15.5	10.8	9.5
6	39	28.5	20.0	14.1
8	58	39	29.3	20.3
10	79	54	38	25.4
12	99	69	48	31.0
14	0.12*	84	58	36
16	0.14*	99	68	39
18	0.16*	0.11*	79	44
20	0.18*	0.13*	89	49
22	0.20*	0.14*	99	54
24	0.22*	0.16*	0.11*	59
26	0.24*	0.17*	0.12*	64
28	0.26*	0.19*	0.13*	69
30	0.28*	0.20*	0.14*	74

* Pressure in millimeters.

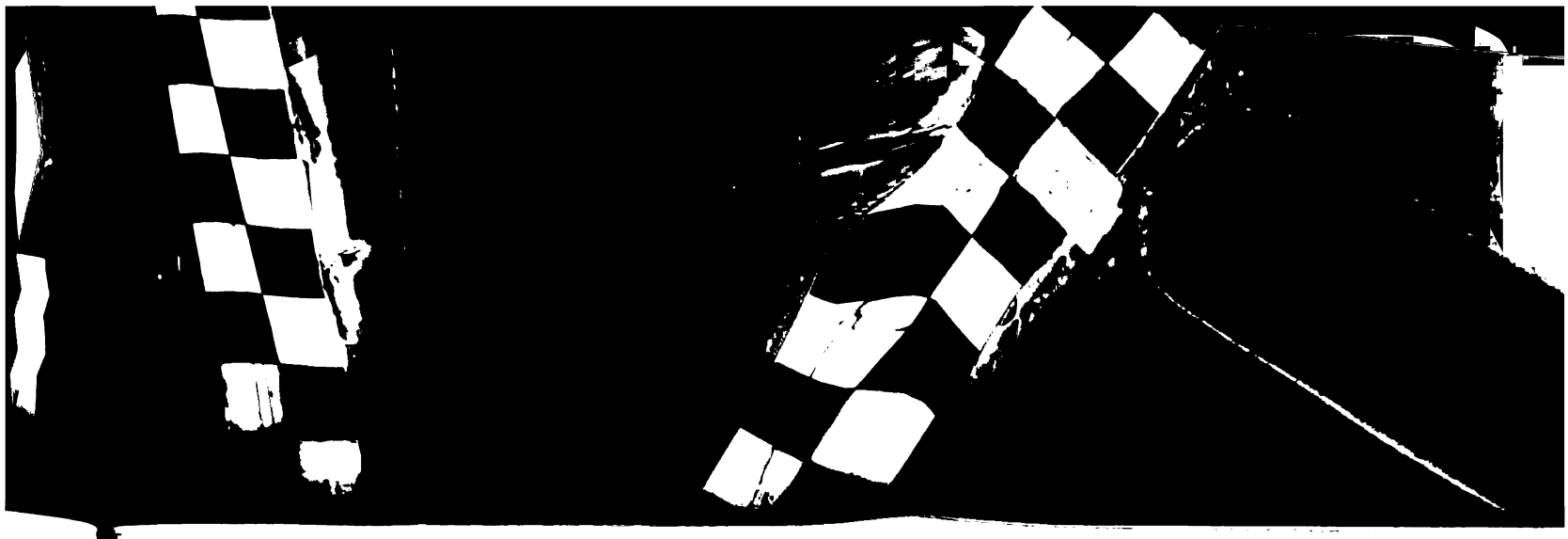


TABLE XLIII

McLEOD PRESSURE VERSUS TIME FOR SILASTIC FOIL S50-001 FOR DIBORANE AT VARIOUS OVERPRESSURES
(pressure in microns, except as noted; time in minutes)

Item		Overpressure			
		297	210	129	55
p_0		0.04	0.10	0.10	0.10
p_{30}		0.75	0.75	0.75	0.75
t°C		26.9	26.9	26.9	26.9
Time	2	93	34	21.5	16.6
	4	0.21*	88	60	44
	6	0.30*	0.15*	0.11*	74
	8	0.39*	0.20*	0.15*	0.10*
	10	0.48*	0.25*	0.18*	0.13*
	12	0.57*	0.30*	0.22*	0.16*
	14	0.66*	0.35*	0.25*	0.18*
	16	0.74*	0.40*	0.29*	0.20*
	18	0.82*	0.45*	0.32*	0.22*
	20	0.90*	0.51*	0.36*	0.24*
	22	0.98*	0.56*	0.39*	0.26*
	24	1.06*	0.61*	0.43*	0.28*
	26	1.13*	0.66*	0.46*	0.30*
	28	1.21*	0.71*	0.50*	0.32*
	30	1.29*	0.76*	0.53*	0.34*

* Pressure in millimeters.

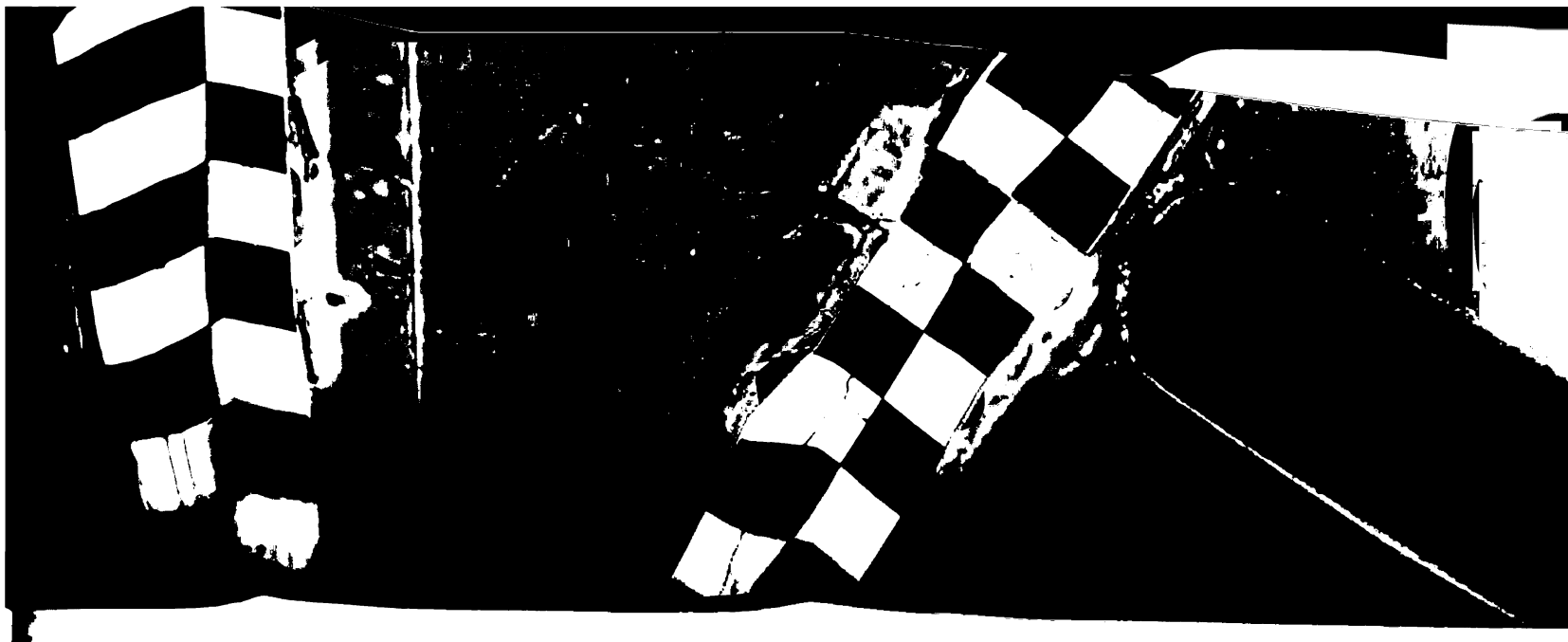


TABLE XLIV

PERMEABILITY CONSTANTS x 10^{-6}

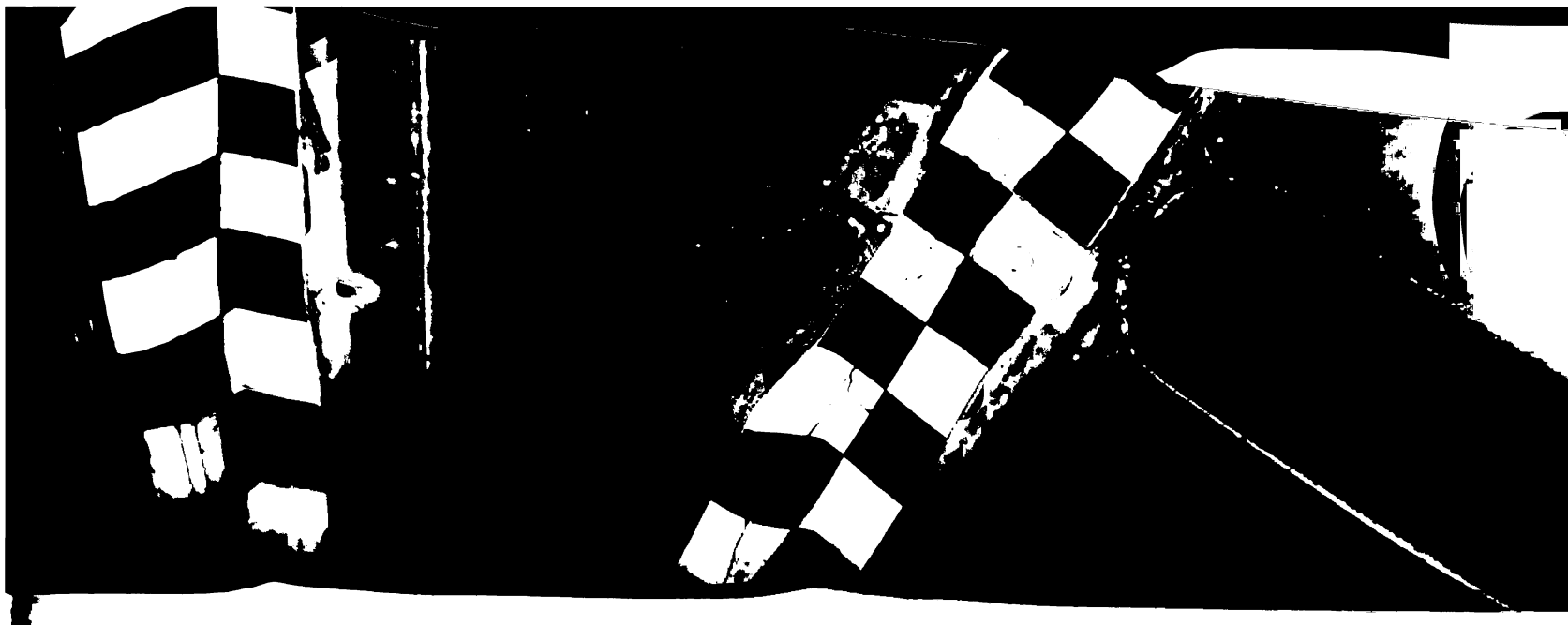
Foil	Gas	k x 10^{-6}
S50-0005	H_2	51.8
S50-0005	N_2	19.8
S50-0005	B_2H_6	63.0
S50-001	H_2	6.46
S50-001	N_2	3.14
S50-001	B_2H_6	9.64
S80-001	H_2	8.70
S80-001	N_2	3.14
S80-001	B_2H_6	16.5

Other than the considerations already given in the thesis it should be noted that diborane diffuses through silastic membranes faster than either hydrogen or nitrogen. This was not caused by a decomposition since the diffusion product was condensed by liquid nitrogen. It is also noted that the silastic membranes permit a faster diffusion of all three gases than any foil used in the past. These foils are the most practical membranes studied with regard to the separation of diborane from hydrogen and nitrogen. The observations may be summarized as follows:

1. The diffusion does not seem to follow Graham's law. Evidence for this statement is (a) that the diborane passes through the membranes more rapidly than does both hydrogen and nitrogen, (b) that the calculated rates of diffusion for nitrogen and hydrogen are not in the same ratio as the calculated values for these two gases.

2. Diffusion of the three gases, hydrogen, nitrogen, and diborane, is faster through the Silastic films than through the metal films or the other plastic films studied.

3. The thinnest Silastic film was five times as thick as the thinnest metal film, yet the diffusion for hydrogen was approximately five to six times as fast through the Silastic film. Diborane, as observed, diffused even faster than the hydrogen.



4. It is observed that Silastic 80 is more porous toward hydrogen and diborane than the 50 for equivalent thicknesses. It is also noted that for the same thicknesses of 50 and 80, the rates for nitrogen are apparently the same.

5. The faster rate for the diborane cannot be due to a decomposition of the material since, following a diffusion, the diffusion product was condensed with liquid nitrogen. After cooling it was observed that the pressure due to the condensed material was not readable on the manometer. Any appreciable amount of hydrogen would have produced a noticeable pressure. This does not preclude the dissociation of diborane into BH_3 within the plastic membrane followed by subsequent reformation of B_2H_6 on emission of the gas from the membrane. Even the formation of BH_3 in the membrane is not in itself an adequate explanation for the rapid transfer of diborane since the molecular weight of BH_3 is about six times that of hydrogen.