A STUDY OF THE EFFECTIVENESS OF A COPPER
FOIL TRAP FOR MERCURY VAPOR IN VACUUM

by

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PREFACE

Vacuum systems employing mercury diffusion pumps ordinarily use a liquid nitrogen trap to keep the mercury vapor from diffusing throughout the vacuum system. These traps require frequent attention as well as considerable expense if they are maintained over a period of months. Thus it is desirable to replace the liquid nitrogen trap with a trap which requires less attention and expense.

Alpert (1) found that a copper foil trap could be used to replace a liquid nitrogen trap when oil diffusion pumps were used. Since it was known that mercury amalgamates readily with copper it was suggested that the Alpert copper foil trap might keep mercury from diffusing from the pumps to the working part of the vacuum system.

INTRODUCTION

Lambert (Harrison, et al, 3) stated that a collimated, monochromatic beam of radiation traversing a homogenous, isotropic medium will suffer a fractional change in intensity equal to e^{-kl} where k is the coefficient of absorption of the medium and is a function of the frequency, and l is the thickness of the absorbing layer. The equation

$$I = I_o \, e^{-kl}$$

is known as Lambert's Law.

If the medium is a gas, it is clear that the coefficient of absorption will depend on the pressure of the gas. For an

ideal gas, the coefficient of absorption is directly proportional
to the pressure since the number of potential absorbers (atoms)
is directly proportional to the pressure.

In this experimental study the 2537 angstrom line from
the $6^1S_0 - 6^3P_1$ transition of mercury was the source of the
monochromatic beam. The absorbing medium was a mixture of gases
with a maximum total pressure of approximately two microns, but
it was assumed that the only absorber of any consequence for
the wavelength being used was mercury vapor. Since mercury
approaches an ideal gas in the temperature and pressure range
used, one might expect a plot of kl versus pressure to be linear.
As will be seen later, however, this curve was not a straight
line so the phenomenon of absorption of resonance radiation by
a gas will be discussed further.

Mitchell and Zemansky (5) have written on the subject of
the absorption of an emission line by gas of the same element
which emitted the line. They stated that if the pressure is
of the order of 10^{-7} to 10^{-4} millimeters of mercury or less
with an absorbing layer thickness not greater than 0.1 to 3.0
centimeters, the only measurable absorption occurs in the central
part of the line. Thus, in this region of pressure and absorbing
layer thickness and in this region only, should one expect
absorption approximately according to Lambert's Law with a single
value for the absorption coefficient.

They stated further that if the pressure of the absorbing
gas is from 10^{-4} to 10^{-2} millimeters and the thickness of the
absorbing layer is from 10 to 50 centimeters, the absorption in

the central region of the line is so large that in any experiment this region would be completely absorbed. Only in the edges of the line would a measurable amount of radiation be transmitted.

The above statements may be summarized as follows: When there is weak absorption, the central region of the line plays the main role; and the absorption coefficient is that for the center part of the line being absorbed. Whereas, when there is very strong absorption, the edges of the line are important; and the absorption coefficient corresponds to the edges of the line.

APPARATUS AND METHOD

Optical System

Lambert's Law for absorption requires that the radiation be collimated and monochromatic. A monochromatic beam was obtained by using a low pressure mercury discharge tube, specifically a Sylvania G4T4/1, 4 watt germicidal lamp, in conjunction with a Bausch and Lomb grating monochromator[1]. The output lens of the monochromator was removed, and the output slits were adjusted so they formed approximately a 0.25 millimeter diameter hole.

A fused quartz lens whose focal length for 2537 A. radiation was determined to be 10.8 centimeters was stopped down to a one

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[1]Bausch & Lomb Grating Monochromator, focal length 500 millimeters, grating area 100 X 100 millimeters, Bausch & Lomb Optical Company.

centimeter diameter and mounted 10.8 centimeters from the output
slits of the monochromator. Thus a one centimeter diameter
collimated beam of radiation was produced.

The absorption tube was 80 centimeters in length and
constructed of 22 millimeter pyrex tubing with fused quartz
windows in the ends. This tube was oriented so that the one
centimeter diameter collimated beam passed down its longitudinal
axis. Small platforms were constructed at both ends of the
absorption tube for the positioning of a photomultiplier detector.
A baffle with a one centimeter diameter hole was attached to the
platform on the input end of the absorption tube to diminish
errors in collimation of the beam.

Two views of the system as described are shown in Plate I.

Optical alignment of the system was accomplished by
utilizing one of the visible lines from the discharge tube.
This insured alignment in the horizontal plane; but it was
necessary to cover the exit end of the absorption tube with
a cap having a one millimeter hole in its center, place the
photomultiplier on the output platform, and peak in the 2537 A.
line in order to be certain the beam was aligned with the tube
in the vertical plane.

A Photovolt photometer[1] with a 1P28 photomultiplier
detector was used in the search unit to measure intensities.
The photometer is near the center of the table and the search

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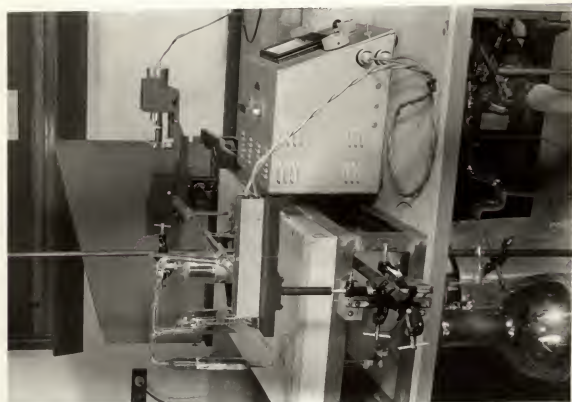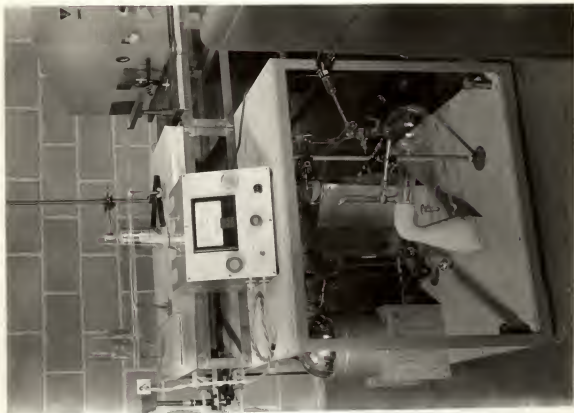[1]Model 520-M Photovolt Multiplier Photometer, Photovolt
Corp.

EXPLANATION OF PLATE I

Two Views of the Experimental Equipment.

Shown are:

1. Monochromator.
2. Absorption tube.
3. Photometer with search unit attached.
4. Small dewar flask containing mercury
   reservoir for calibration.

PLATE I

unit is shown in position on the output platform in Plate I.
Although the room was darkened by blackout curtains when
intensity measurements were being made, an ultra violet filter[1]
was used with the search unit to keep the remaining visible
light in the room from reaching the photomultiplier. The
visible radiation came primarily from the electron tubes and
panel lights of the photometer even though these sources were
shielded somewhat by an enclosure. This enclosure as well as
shielding around the source on the monochromator was removed
when the photographs for Plate I were made. Background intensity
in the region of the absorption tube was always less than 1 unit
while the lowest intensity encountered in the absorption
measurements was 43.1 units.

Vacuum System

A photograph of the copper foil trap used in this study is
shown on Plate II. It consists of corrugated copper foil which
has been rolled up so as to fit snugly into 25 millimeter pyrex
tubing. A trap of this design has been shown by Alpert (1)
and in this laboratory to work satisfactorily with an oil
diffusion pump to produce vacuums of the order of 10^{-9} milli-
meters of mercury or better.

Plate III is a block diagram showing the essential
arrangement of the components of the vacuum system. A Welch

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[1]Ultra violet filter for 200 to 400 milli-micron wave-
length band, catalog no. 5265, Photovolt Corp.

PLATE II

2A620
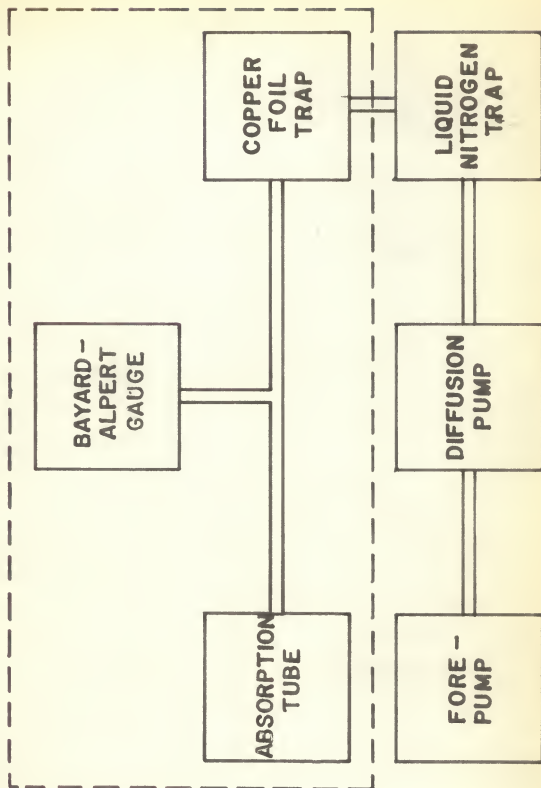
EXPLANATION OF PLATE III

Block diagram showing essential arrangement
of components of vacuum system. Broken line
encloses components which were baked in oven.

Plate III

COPPER FOIL TRAP

LIQUID NITROGEN TRAP

BAYARD – ALPERT GAUGE

DIFFUSION PUMP

ABSORPTION TUBE

FORE – PUMP

Duo-Seal forepump was used with a single stage, water cooled mercury diffusion pump. A Bayard-Alpert ionization gauge (2), manufactured by Westinghouse, was used for pressure measurements.

After the absorption tube, Bayard-Alpert gauge, and copper foil trap had been baked at 400° Centigrade for 12 hours with the liquid nitrogen trap in operation, an ultimate pressure of 8.3×10^{-9} millimeters of mercury was attained with the aid of the ion pump. Without the ion pump the pressure could be maintained at 4.0×10^{-7} millimeters.

Experimental Method

When the system had reached the equilibrium pressure of 4.0×10^{-7} millimeters, the liquid nitrogen was removed from the trap, exposing the copper foil trap to mercury vapor. The passage of mercury through the copper foil was monitored by measuring the absorption of resonance radiation as a function of time. This involved simply alternating the position of the photometer search unit from the input platform to output platform. It was essential that the input intensity be periodically measured since the light output of the source varied with time even though its voltage supply was stabilized to less than 1 per cent variation.

In order to show independently that mercury vapor was present in the absorption tube, a spectrogram of the discharge from the tube when excited by a tesla coil was made.

To obtain a calibration curve, i.e. a correlation between

the measurements of I and I_o and mercury vapor pressure, a
flask containing a pool of mercury was attached to the absorption
tube. This flask was placed in a small dewar flask so that the
temperature of the pool of mercury could be controlled. With
the absorption tube and flask evacuated and sealed off from the
pumps, the mercury vapor pressure in the tube was controlled
entirely by the temperature of the pool of mercury in the flask
since the pool was never above room temperature. It was possible
to firmly establish the absorption corresponding to 0° and 24.5^o
Centigrade since these measurements corresponded to the relatively
stable conditions of ice and water in equilibrium in the dewar
flask and water at room temperature. The absorption corresponding
to zero mercury vapor pressure was established by measurements
before mercury was introduced into the vacuum system. Absorption
corresponding to other temperatures was obtained by pouring a
1:1 ratio by weight of ethyl alcohol and ice into the dewar
flask when it contained a small amount of liquid nitrogen. The
liquid nitrogen froze part of the alcohol and maintained
effectively zero absorption. As the alcohol-ice mixture warmed,
absorption measurements were made corresponding to the instantan-
eous temperature of the mercury pool.

A thermocouple was firmly attached to the outside of the
liquid nitrogen trap where most of the trapped mercury was
apparently frozen to the wall. After the trap was cooled to
operating temperature, the liquid nitrogen was removed; and the
temperature of the trap as a function of time was measured.

RESULTS

Data collected from absorption measurements starting with
time zero as the time when the liquid nitrogen was removed from
the trap are summarized in Table 1. I_o and I are the quantities
that were actually measured and kl was calculated from Lambert's
Law. To simplify the results kl itself was not tabulated in
Table 1 but a quantity kl^*, which is the value of kl adjusted
so that the absorption for zero mercury vapor pressure was zero,
is given. The quantity kl^* was obtained from the following
relation:

$$kl^* = kl - \log_e (I_o/I)_o$$

where $(I_o/I)_o$ is the ratio of the input to output intensity
with zero mercury vapor pressure in the absorption tube. The
results from two separate trials are presented in the table.

Calibration data are given in Table 2. Each of the values
of kl^* is associated with a known temperature of the pool of
mercury in the dewar flask. The mercury vapor pressures which
are listed correspond (4) to the liquid mercury temperatures
at which the absorption measurements were made. The information
in Table 2 is given in graphical form in Plate IV.

With the aid of Plate IV, it was possible to determine
the mercury vapor pressure corresponding to each value of kl^*
in Table 1 and thus to plot mercury vapor pressure in the
absorption tube versus time in Plate V.

The plot of mercury vapor pressure in the liquid nitrogen
trap after removal of the liquid nitrogen from the trap, Plate V,

Table 1. Mercury vapor pressure in absorption tube as function of time.

Time: Min.:	k_1		Mercury vapor pressure (μ)	
	First trial	Second trial	First trial	Second trial
0	0	0	0	0
3		0.374		0.004
4		0.729		0.014
5		0.283		0.002
6	0.309	0.280	0.002	0.002
7	0.381	0.331	0.003	0.003
8	0.420	0.351	0.004	0.003
9	0.409		0.004	
10	0.291	0.614	0.002	0.008
11	0.351	0.624	0.002	0.009
12	0.448	0.624	0.005	0.009
13	0.453	0.676	0.005	0.011
14	0.683	0.733	0.012	0.014
15	0.754		0.015	
16	1.026	0.722	0.033	0.013
17	1.126	0.880	0.043	0.022
18	1.330	1.034	0.073	0.035
19	1.450	1.195	0.097	0.053
21		1.519		0.113
22	1.752	1.638	0.194	0.149
23		1.742		0.188
24		1.871		0.263
25		1.948		0.323
27	2.106	2.071	0.530	0.470
28	2.158	2.134	0.727	0.604
29	2.186	2.180	0.824	0.820
30		2.214		0.924
31	2.230	2.251	1.037	1.083
32		2.286		1.224
33		2.311		1.324
35		2.346		1.484
36		2.368		1.540
37		2.382		1.597
43	2.329		1.390	
46	2.332		1.394	
51	2.335		1.398	
53		2.464		1.918
67		2.468		1.920
90	2.337		1.401	
203		2.435		1.820
341	2.322		1.375	
367		2.479		1.970
679		2.546		2.275
703	2.379		1.590	
1354	2.497		2.055	
1410		2.511		2.110

Table 1. (concl.)

Time: Min.:	kl* First trial	Second trial	Mercury vapor pressure (μ) First trial	Second trial
2031	2.310		1.337	
2035		2.385		1.620
2831	2.415		1.720	
2898		2.405		1.680
3106	2.406		1.700	
6298	2.487		2.020	

Table 2. Calibration data.

Liquid mercury temperature ($^{\circ}C.$)	Mercury vapor pressure (μ)	kl*
	0	0
-23.0	0.0124	0.634
-17.0	0.0265	0.886
-10.0	0.0606	1.259
0.5	0.196	1.702
1.7	0.221	1.758
4.0	0.276	1.893
8.8	0.440	2.069
15.3	0.797	2.148
15.4	0.804	2.122
19.4	1.143	2.339
20.0	1.201	2.243
24.4	1.753	2.417
24.6	1.784	2.401

(kl* is kl adjusted so that the absorption is zero for zero mercury vapor pressure.)

was made by assuming that the vapor pressure in the trap corresponded precisely to the vapor pressure of mercury at the temperature indicated by the thermocouple on the outer wall of the trap. Data for the plot are given in Table 3.

Plate VI is the spectrogram made by exciting the gas in the absorption tube when equilibrium pressure had been reached after the liquid nitrogen had been removed from the trap. The spectrum from the absorption tube is compared with that of the

EXPLANATION OF PLATE IV

Calibration curve — shows relation between kl^* and mercury vapor pressure where kl^* is the exponent in Lambert's Law adjusted so that absorption is zero for zero mercury vapor pressure.

PLATE IV

MERCURY VAPOR PRESSURE (microns)
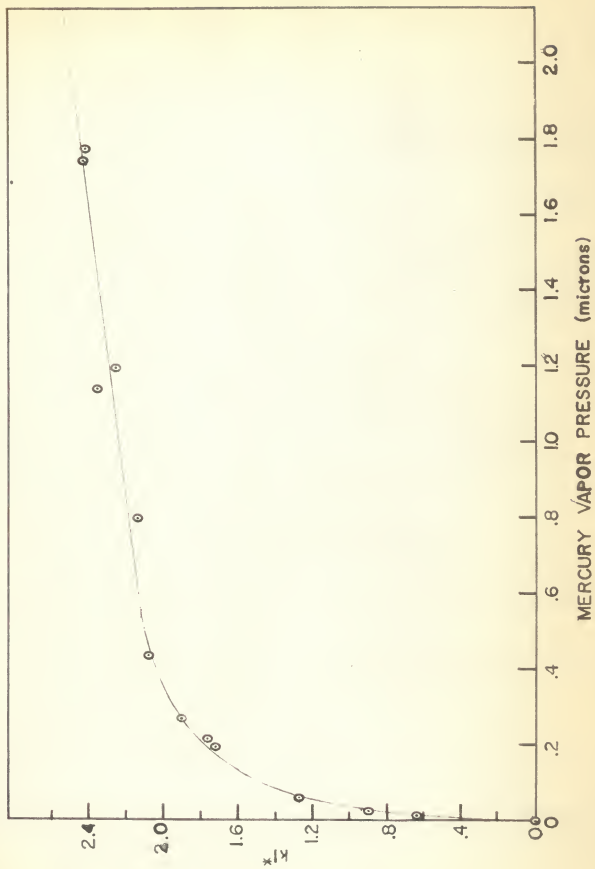
EXPLANATION OF PLATE V

Trial 1 Points ⊙ ; Curve – – – ;
Trial 2 Points ⊡ ; Curve ⋯⋯⋯

Trials 1 and 2 are the mercury vapor pressure
in absorption tube versus time after removal of
liquid nitrogen from trap.

The solid curve (points ▽) refers to mercury
vapor pressure in the liquid nitrogen trap after
removal of liquid nitrogen from trap as determined
by temperature of trap.

PLATE V

MERCURY VAPOR PRESSURE
AT ROOM TEMPERATURE
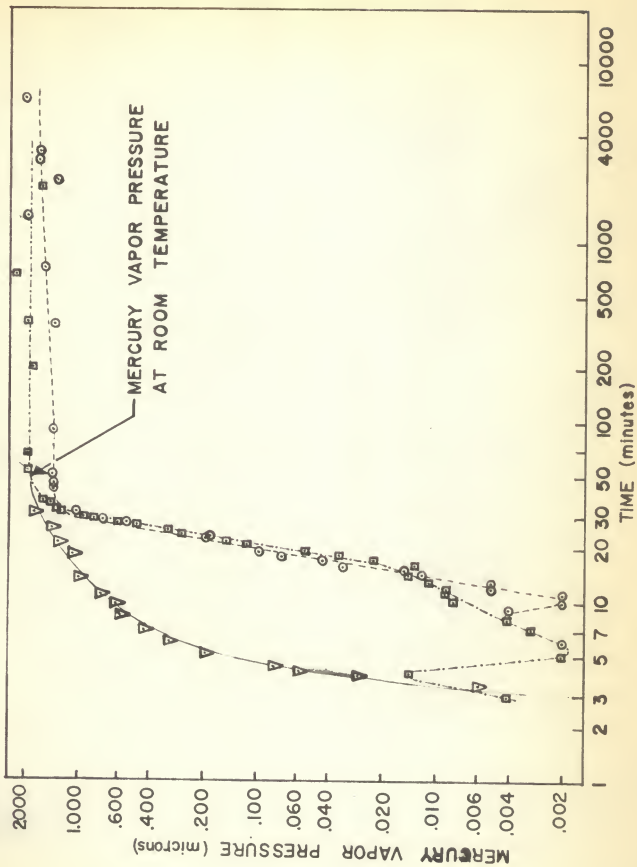
EXPLANATION OF PLATE VI

Spectrogram from low pressure mercury
discharge tube (bottom) compared to spectrum
from externally excited absorption tube when
it reached equilibrium pressure after removal
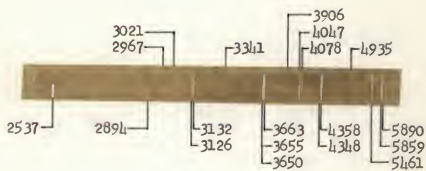of liquid nitrogen from trap.

PLATE VI

Table 3. Mercury vapor pressure in liquid nitrogen trap as function of time.

Time Min.	:	Liquid mercury temperature (°C.)	:	Mercury vapor pressure (μ)
2.95		-38		0.00147
3.43		-29		0.0056
4.17		-11		0.056
5.25		0		0.185
6.13		5		0.305
7.00		8		0.41
8.45		11		0.55
9.72		12		0.60
11.08		14		0.72
12.67		16		0.86
14.00		17		0.95
18.67		18		1.03
21.67		20		1.24
25.83		21		1.35
31.67		24		1.72
39.75		25		1.84
44.75		25		1.84

low pressure mercury lamp used as a source in the absorption measurements.

DISCUSSION OF RESULTS

The calibration data in Table 2 corresponding to all temperatures from -20.0° to 20.0° Centigrade inclusive, with the exception of those for 0.5 and 1.7° Centigrade, were taken as the ethyl alcohol-ice mixture warmed in a dewar flask to room temperature. It was assumed in determining the mercury vapor pressures listed in the table that the vapor pressure in the absorption tube corresponded precisely to the liquid mercury temperature at the time the absorption measurement was made even though the temperature was slowly increasing. That this assumption did not introduce important errors is shown by the

fact that the points in Plate IV which correspond to absorption measurements under much more stable temperature conditions (0° and 25° C.) fit smoothly into the sequence taken under dynamic conditions.

The general shape of the calibration curve in Plate IV is in agreement with the theory given in the Introduction. As the pressure increases from zero, the absorption increases very rapidly at first. This corresponds to absorption of the central part of the emission line. As the vapor pressure increases more, the central part of the line is completely absorbed and the slope of the curve decreases greatly, corresponding to the much lower absorption of the edges of the emission line.

The curve in Plate V indicates that oscillations occurred in the mercury vapor pressure in the absorption tube within the first 10 minutes after the liquid nitrogen was removed from the trap. These may be attributed to the fact that the outer tube of the liquid nitrogen trap warms more rapidly than the inner tube thus causing an oscillation in the pressure as the mercury shifts from the outer to the inner surface.

Trials 1 and 2 in Plate V both indicate that the mercury vapor pressure in the absorption tube 50 minutes after the liquid nitrogen was removed was approximately the vapor pressure of mercury at room temperature. Variations in the pressure after reaching this level are of the order of ±15 per cent and may be attributed to error in measuring the pressure and to actual variations in the pressure as room temperature changed. Estimated probable errors in individual measurements were less

than 5 per cent.

After the pressure in the absorption tube leveled off at approximately 2 microns, the Bayard-Alpert gauge indicated a pressure of roughly 0.8 microns. This gauge was calibrated for nitrogen and not mercury vapor, but one would expect the pressure indication to be of the same order of magnitude as the true pressure. The important point here is that the ionization gauge offered a completely independent method of measuring the pressure in the absorption tube, and it was in reasonable agreement with the absorption method.

Plate V also has a curve showing how the mercury vapor pressure in the liquid nitrogen trap increased after the liquid nitrogen was removed. The curve was made by assuming that the mercury vapor pressure at each instant corresponded to the temperature of the mercury in the trap. This assumption is not as reasonable here as it was in the case of the calibration curve since in that case the same temperature change occurred over a period of 22 hours and here it took place in 40 minutes. One might speculate that the pressure was actually somewhat less at each instant of time, particularly when the trap was warming rapidly in the first 5 minutes. The significance of the curve is that it indicates that the vapor pressure in the trap rose to that corresponding to room temperature in very nearly the same length of time that the pressure rose to equilibrium in the absorption tube.

Plate VI, the spectrogram, shows clearly that mercury was

present in the absorption tube after the liquid nitrogen was removed and the pressure reached equilibrium. The 2537 A. line does not appear in the spectrum from the absorption tube since the emission was studied through the pyrex walls of the tube and thus absorbed before it reached the spectrograph. Since all of the lines may be identified as arising from mercury, the spectrogram indicates that mercury was the principal gas in the tube. This fact supports the assumption stated in the Introduction that the only absorber of consequence in the absorption tube was mercury.

CONCLUSION

Two independent methods of detecting mercury vapor and of measuring pressure in the absorption tube were used and gave complementary results. Thus it may be stated that the copper foil used in this study is ineffective as a trap for mercury vapor. Furthermore, since the foil apparently did little more than act as a baffle to the diffusion of the vapor, it is unlikely that copper may be used to absorb mercury vapor in sufficient quantity to be used as a trap regardless of the design of the trap.

ACKNOWLEDGMENTS

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A study of the possibility of using a copper foil trap to replace the liquid nitrogen trap commonly used in vacuum systems employing mercury diffusion pumps was undertaken.

The basic procedure used in the investigation was simply to expose the copper foil trap to mercury vapor from the diffusion pumps and then measure the rate of passage of vapor through the trap into an optical absorption cell. Primarily one is interested in the partial pressure of mercury in the absorption tube as a function of time. This was determined by measuring the absorption of a collimated, monochromatic beam of the resonance radiation for mercury.

A correlation between the absorption of resonance radiation and mercury vapor pressure in the absorption tube was established. This was accomplished by controlling the temperature of a pool of mercury in a flask attached to the absorption tube. By keeping the mercury pool below room temperature, one knew the mercury vapor pressure in the absorption tube since it was precisely the equilibrium vapor pressure for mercury at the temperature of the pool. Tabulations of the vapor pressure of mercury as a function of temperature have been made; so all that remained to be measured was the absorption of resonance radiation at known temperatures of the mercury pool.

A measure of the effectiveness of the copper foil trap was established by removing the liquid nitrogen from the trap which had been isolating the copper foil from the mercury of the diffusion pump and then measuring the absorption in the cell as the mercury vapor passed through the copper foil trap into

the cell. With the aid of the calibration curve, i.e. the correlation between mercury vapor pressure and kl from Lambert's Law for absorption $I = I_o e^{-kl}$, a plot of mercury vapor pressure in the absorption tube versus time was made.

This curve indicated decisively that the copper foil trap was ineffective since the mercury vapor pressure in the absorption tube rose to 2 microns in 50 minutes.