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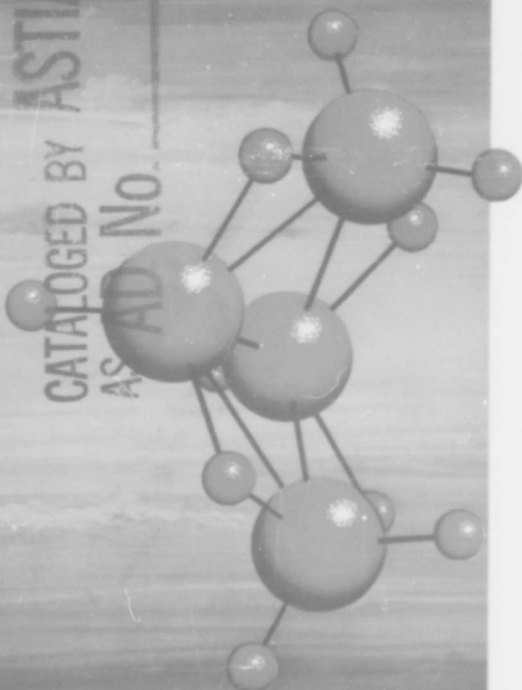
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PHYSICAL PROPERTIES OF
LIQUID PENTABORANE

Navy Contract NOa(s) 58-454 d

September, 1961



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PHYSICAL PROPERTIES OF LIQUID PENTABORANE

Navy Contract NOa(s) 58-454-d

IV - Vapor Pressure

by

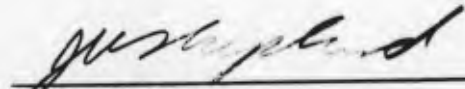
C. F. Boynton and D. E. Terpko

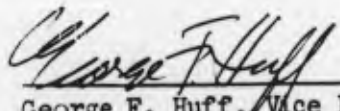
V - Thermal Conductivity

by

J. R. Ludwig and C. F. Boynton

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Research and Development Division



PHYSICAL PROPERTIES OF LIQUID PENTABORANE

Summary

The vapor pressure of liquid pentaborane from 25 to 75°C is described by the equation:

$$\log P = 5.6352 - \frac{1384}{t + 250} ,$$

where P = Vapor pressure in P.S.I.A., and

t = temperature in degrees centigrade.

From 75 to 125°C, the vapor pressure is described by the equation

$$\log P = 4.4803 - \frac{730}{t + 160}$$

The thermal conductivity of liquid pentaborane from 70°F to 105°F is described by the following equation

$$k_L = 0.0874 - 6.84 \times 10^{-5} t$$

where k_L = thermal conductivity, BTU/hr-ft² - (°F/ft)

t = temperature, °F.

The experimental work was done at the Research and Development Laboratories of Callery Chemical Company under Navy Contract NOa(s) 58-454 d with funds supplied by the Air Force (MIPR 60-21).

PHYSICAL PROPERTIES OF LIQUID PENTABORANE

Introduction

Although pentaborane, B_5H_9 , has been known and studied for several decades, no measured physical property data are available for temperatures above its normal boiling point of $60.0^\circ C$, and no data are available on its thermal conductivity. Two characteristics of pentaborane present problems in any program designed to extend data to higher temperatures. First its extreme sensitivity to air and moisture require that pentaborane be protected from the atmosphere during all handling and measurement operations. Second, the decomposition rate of pentaborane becomes significant at these higher temperatures, and special precautions must be observed to obtain reliable data.

High temperature values for density and viscosity of liquid pentaborane as well as solubilities of nitrogen, hydrogen and helium in liquid pentaborane at high pressures were reported in the first volume. High temperature values for vapor pressure, and low temperature values for thermal conductivity of liquid pentaborane are presented in this volume. The experimental program is being supported by funds from MIPR 60-21 (issued by the Air Force) through Contract NOa(s) 58-454 d of the Bureau of Naval Weapons.

PHYSICAL PROPERTIES OF LIQUID PENTABORANE

IV - VAPOR PRESSURE

Results

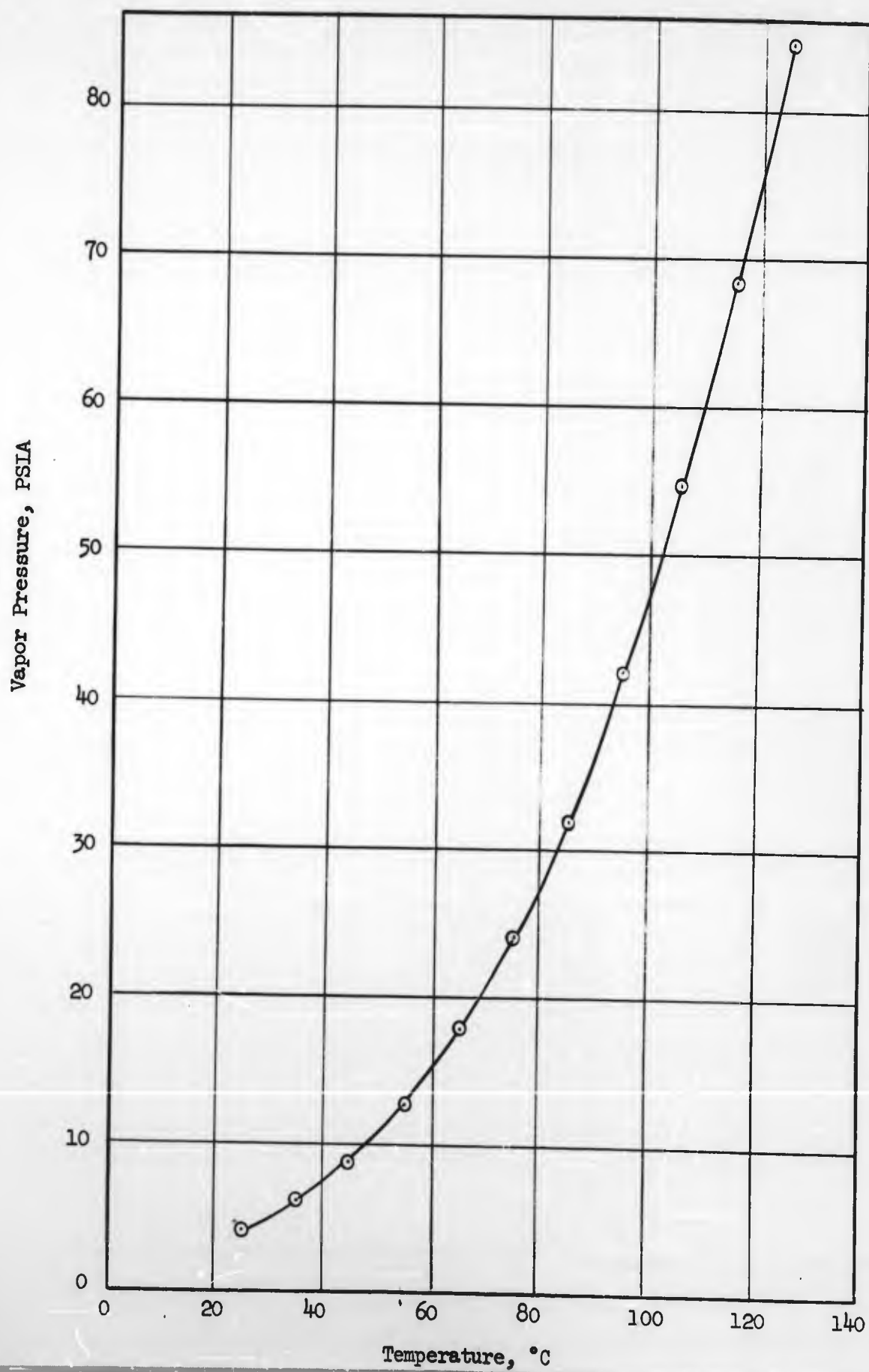
A graphical presentation of the results is given in Figure 1. Tabulated data are given in Table A - 1 (Appendix A).

Discussion of Results

Eleven series of measurements were made, each consisting of one to seven runs. Because many of the runs were unsuccessful for some reason, those are being omitted. Table A-II lists the absolute pressures exerted by samples of pentaborane heated to various temperatures in a modified Reid vapor pressure apparatus. The observed pressure rose rapidly at first as the sample warmed to the bath temperature. Then the pressure increased relatively slowly at a constant rate, due to thermal decomposition of the pentaborane. A pressure-time curve was plotted, and a straight line through the linear portion of the curve was extrapolated to zero time to obtain the experimental vapor pressure. This corresponds to the pressure of liquid pentaborane in equilibrium with its vapor at a given temperature, when no decomposition gases are present.

Three different range compound gauges were used to measure the pressure of pentaborane. Each gauge was calibrated against a standard, and the observed pressures were all converted to absolute pressure in pounds per square inch. Included in Table A-II are five duplicate runs to indicate the reproducibility of the results. Except when some obvious trouble such as poor bath temperature regulation or contamination of the sample was present, reproducibility was good.

FIGURE 1

VAPOR PRESSURE OF
LIQUID PENTABORANE

No attempt was made in these experiments to make a comprehensive study of the thermal stability of pentaborane. Incidental to the experiments, however, an indication of the relative rate of decomposition of pentaborane at various temperatures was observed. The rate of pressure increase in the apparatus, in PSI per hour, over the linear portion of the pressure-time curve was calculated for each run. They are listed in Table A-II and presented graphically in Figure 2 on semi-logarithmic paper. Despite the scattering of the points, the expected trend of increased rate of decomposition as the temperature is increased, is quite evident. Calculations based on the decomposition of the pentaborane into hydrogen have indicated that none of the experiments went beyond 0.4 percent decomposition. Although the conditions of these experiments are such that the rates of decomposition cannot be compared directly, the results generally confirm those of McDonald.⁽¹⁾

The observed vapor pressure values agree, within experimental error, with the results published by Wirth and Palmer⁽³⁾, and Shapiro and Ditter⁽²⁾, up to 55°C. The normal boiling point, calculated from the equation

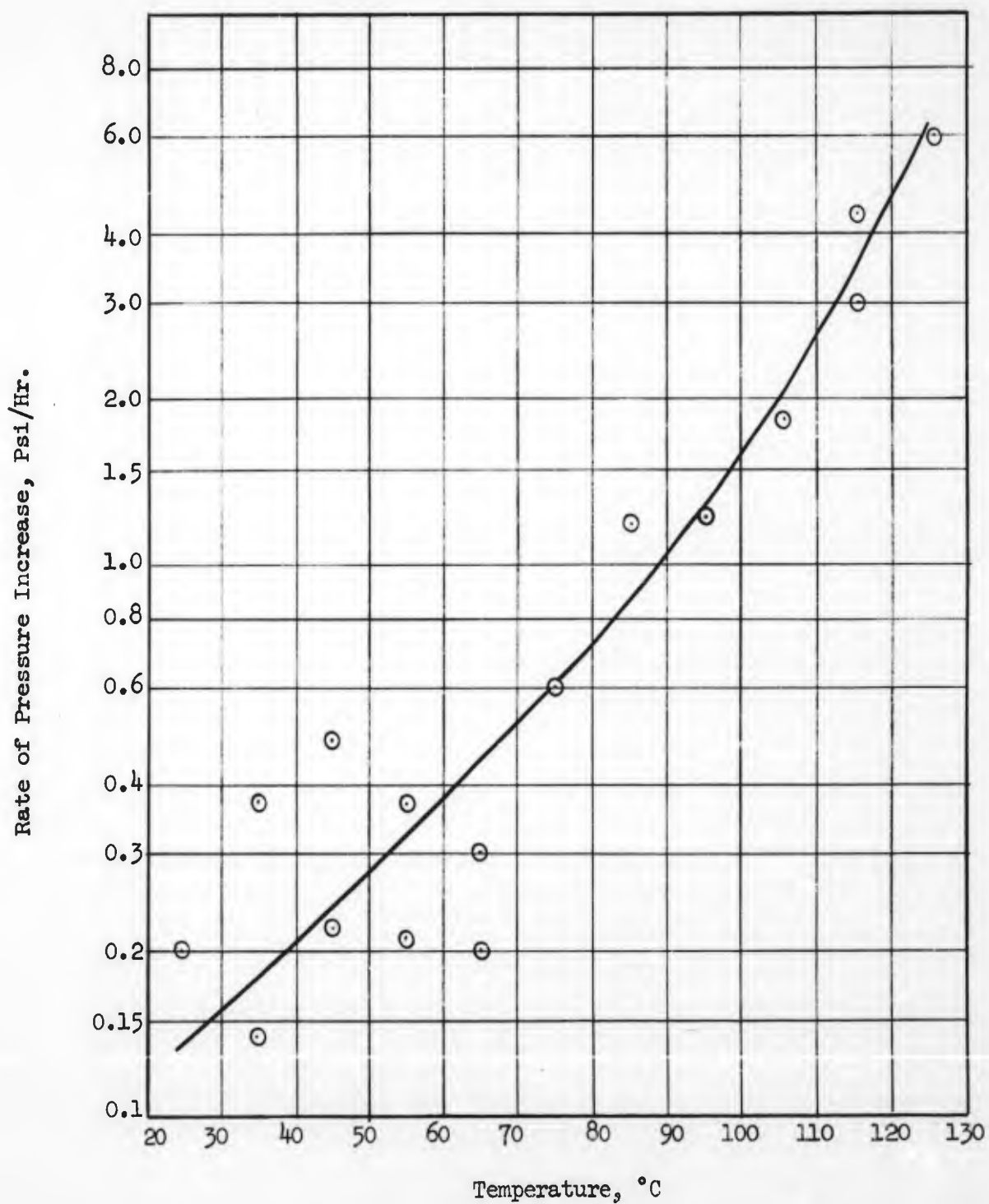
$$\log P = 5.6352 - 1384/(t + 250)$$

is 59.8°C, and that from the equation

$$\log P = 4.4803 - 730/(t + 160)$$

is 60.3°C. Thus the average calculated boiling point is close to Shapiro and Ditter's value of 60.0°C.

FIGURE 2

RELATIVE RATE OF DECOMPOSITION
OF PENTABORANE

It should be noted that Zhigach, Kazakova, and Kigel⁽⁴⁾ found that pentaborane boiled at 60 - 60.2°C. Thus, the previously accepted value of 58.4°C appears to be incorrect.

The color of the pentaborane turned pale yellow as its temperature was increased above room temperature. The depth of yellow color was approximately proportional to the temperature to which the sample was heated. No solids were observed as a result of the heating except a trace of yellow solids at 125°C.

Discussion of Experimental Method

The modified Reid vapor pressure apparatus is shown in Figure 3. The brass body including the valve and O-ring connector was purchased from Research Appliance Company, Allison Park, Pennsylvania. Three different gauges were used: one was a 4-inch, 0 to 30 inches of vacuum gauge; one was a 4-inch, 30 inches vacuum - 60 PSI compound gauge, and one was a 3-inch 30 inches vacuum - 200 PSI compound gauge. With each gauge in place, the volumes of the assembled apparatus were 20.8, 26.8, and 11.8 ml. respectively. The gauges were calibrated against secondary standard gauges which had been calibrated with dead weight gauges. The vacuum regions were calibrated against a mercury manometer. Calibration curves were then drawn for each gauge so that observed pressure readings could be converted directly to absolute pressure in pounds per square inch.

It is assumed that the sample attained the temperature of the bath instantaneously, although actually a finite length of time was required. Thus a small error is introduced because this time is not known exactly, and all experimental vapor pressure values are slightly low. This error is within the experimental error of the pressure readings except possibly at 115 and 125°C.

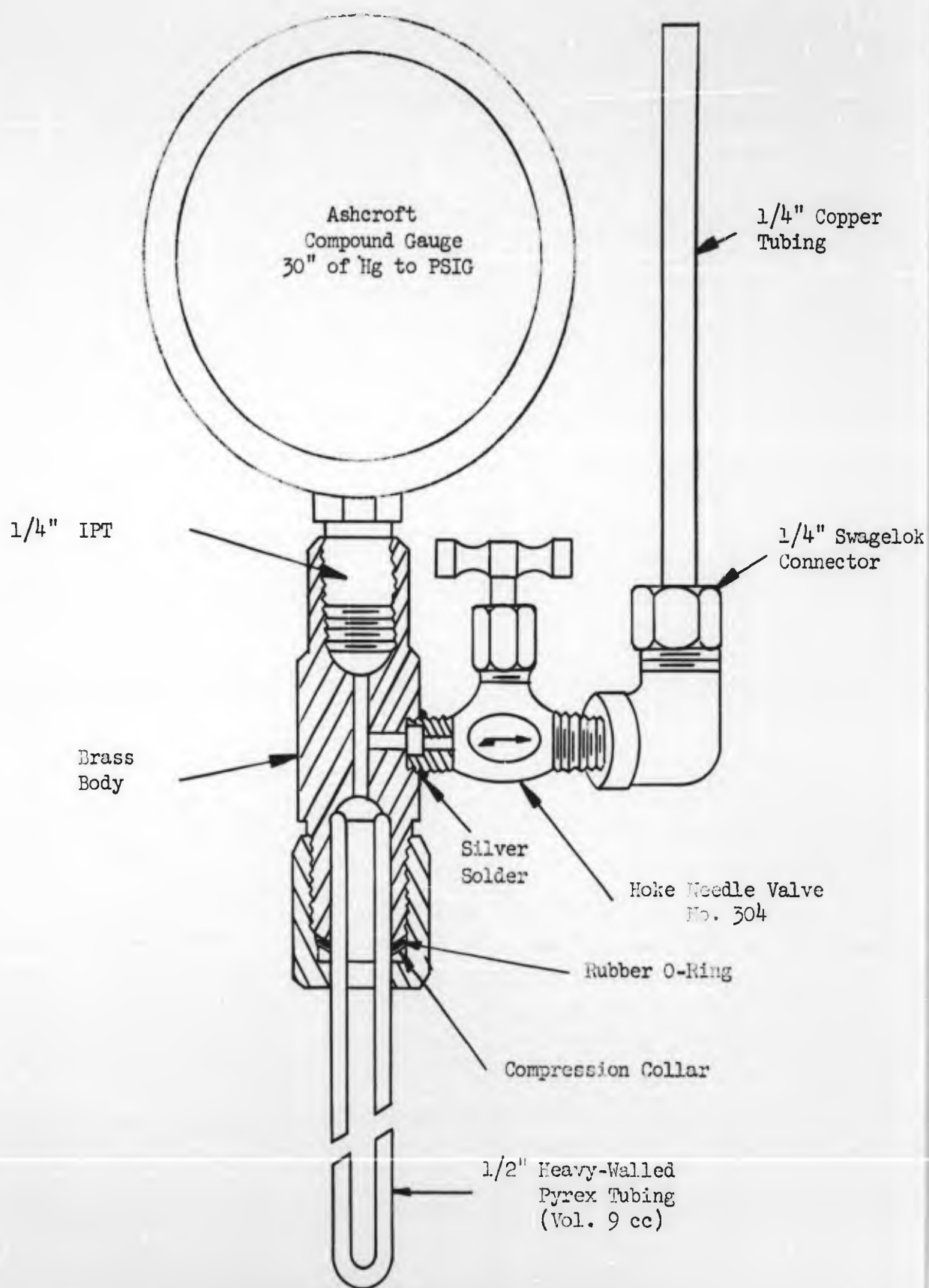


FIGURE 3

MODIFIED REID VAPOR PRESSURE APPARATUS

Several runs were made at each temperature to check the reproducibility of the results. Usually, the first run with a fresh charge of pentaborane gave slightly high vapor pressures--for example, approximately 0.4 PSI higher than normal at 65°C. This is caused principally by hydrolysis of the pentaborane with traces of water in the apparatus. One run in which water was known to be present gave very high apparent vapor pressure. Another contributing factor may be decomposition of traces of other boron hydrides in the pentaborane.

After each run, the sample was frozen at -80°C, and thoroughly degassed. In this way, several runs could be made each time the apparatus was loaded. Any condensable decomposition products remaining in the sample did not affect the experimental vapor pressure. McDonald⁽¹⁾ found that products of the decomposition of pentaborane do not appreciably affect its rate of decomposition.

References

- (1) McDonald, G. E., The Rate of Decomposition of Liquid Pentaborane from 85° to 202°C, RME 55H01, NACA, Lewis Flight Propulsion Laboratory, Cleveland, Ohio.
- (2) Shapiro, I., and Ditter, J. F., J. Chem. Phys. 26, 798 - 801 (1957).
- (3) Wirth, H. E., and Palmer, E. D., J. Phys. Chem. 60, 914 - 16 (1956).
- (4) Zhigach, A. F., Kazakova, E. B., and Kigel, R. A., Doklad Akad. Nauk S.S.S.R., 106, 69 - 71 (1956), CA 50, 13730 i.

PHYSICAL PROPERTIES OF LIQUID PENTABORANE

V - THERMAL CONDUCTIVITY

Results

A graphical presentation of the results is given in Figure 4. Tabulated data are given in Table A-III (Appendix A).

Discussion of Results

Five measurements of the thermal conductivity of liquid pentaborane were made over the temperature range 65 to 105°F. One of the results, however, run number L-24, was inconsistent with the others, and was therefore discarded. The exact reasons for this inconsistency are not known. Because this run was the last in a series of runs, it seems probable that reaction of the pentaborane sample with a rubber gasket in the apparatus caused the apparent thermal conductivity to be relatively high.

The result of the preceding run, number L-23, is also somewhat high, which tends to support the hypothesis that the thermal conductivity of the pentaborane was increasing during that series of runs.

The other three values were found to fit the linear equation

$$k_L = 0.0874 - 6.84 \times 10^{-5} t$$

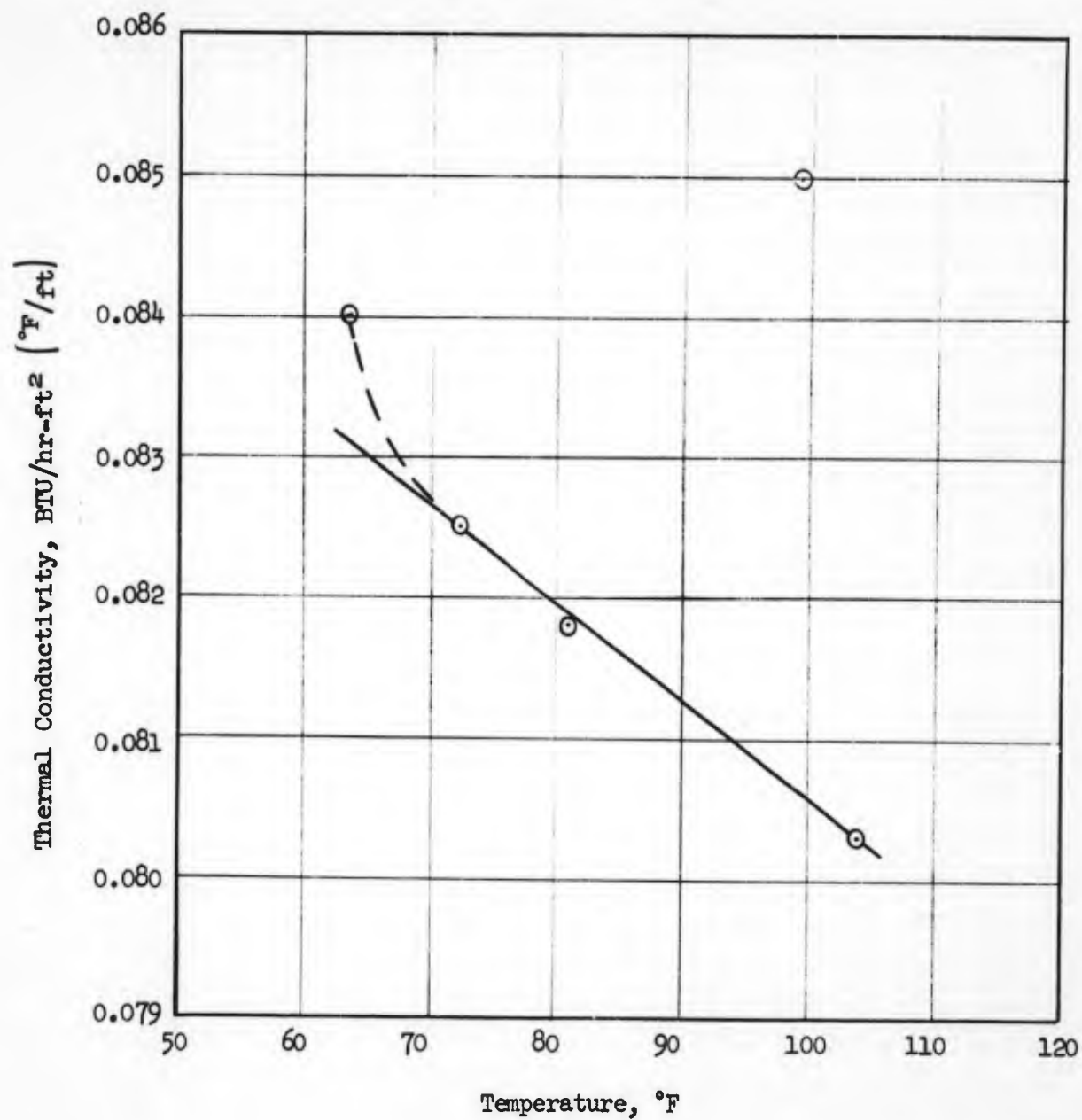
where k_L = thermal conductivity, BTU/hr-ft² - (°F/ft)

t = temperature, °F .

FIGURE 4

THERMAL CONDUCTIVITY OF LIQUID PENTABORANE

$$k_L = 0.0874 - 6.84 \times 10^{-5} t$$



The thermal conductivity of liquid pentaborane is of the same order of magnitude as those of many organic liquids, reported by Sakiadis and Coates⁽¹⁾.

The samples gradually turned a pale yellow color, especially at the higher temperatures. This coloration is probably due to two factors. First, liquid pentaborane alone turns pale yellow as its temperature is raised much above room temperature because of slow decomposition. Second, liquid pentaborane slowly attacks the Viton A (duPont) fluororubber O-ring used as a gasket around the top of the cold bar. Unlike ordinary rubber, which is completely unsuitable for use in contact with liquid boron hydrides, fluororubbers are relatively resistant to attack. Over a period of time, however, even fluororubber tends to absorb pentaborane, swell, and impart a yellow color to the liquid. During the runs no gassing, precipitation of solids, or other evidence of decomposition were observed.

Discussion of Experimental Method

The thermal conductivity apparatus was constructed by Research Appliances Company* and was based on a design by Sakiadis and Coates⁽²⁾.

The apparatus is of the steady state type. The liquid layer is enclosed between two 6-inch diameter horizontal parallel steel bars, and heated downward to eliminate convection currents. To establish isothermal surfaces the

* Allison Park, Pennsylvania

top and bottom bars are heated and cooled respectively by large amounts of water circulated from constant-temperature baths. Heat flows in series through the liquid layer and a 4-inch thick steel bar. Eighteen copper constantan thermocouples are embedded in the steel bars in 4 layers at different fixed distances from the surfaces of the bars.

Heat losses are minimized by enclosing the bars in a glass cylinder and providing thermal guarding. The thickness of the liquid layer is measured by three micrometers.

The thermal conductivity of the liquid is calculated by the over-all resistance to heat flow method, which involves calculating the resistance to heat flow due to the steel bars and to the enclosed liquid layer.

By definition

$$R_t = \frac{\sum(\Delta t)}{q} \quad (1)$$

where R_t = total resistance to heat flow (including effects due to surface irregularities and films), °F-hr/BTU

$\sum(\Delta t)$ = over-all temperature drop due to liquid layer and a known thickness of steel bars, °F.

q = heat flow across the liquid layer and steel bars, BTU/hr.

$$R_t = R_s + R_L \quad (2)$$

where R_s = resistance to heat flow due to steel bars alone, °F-hr/BTU

R_L = resistance to heat flow due to liquid layer, °F-hr/BTU

To account for the resistance due to liquid trapped in the surface irregularities, the actual resistance to heat flow is measured when the steel bars are in direct contact and liquid is present. Equation (2) then becomes

$$R_T = R_O + R_L \quad (3)$$

where R_O = resistance to heat flow at zero liquid
film thickness, °F-hr/BTU

The net resistance due to the liquid layer can be used to calculate the thermal conductivity of the liquid.

$$R_L = R_T - R_O = \frac{X_L}{k_L A} \quad (4)$$

$$\text{and } k_L = \frac{X_L}{A(R_T - R_O)} \quad (5)$$

where X_L = thickness of liquid layer, feet

k_L = thermal conductivity of liquid, BTU/hr-ft²
(°F/ft)

A = arbitrary area of steel bars and liquid
layer through which heat flows, ft².

Further details of the calculation method are given in Appendix C.

Because the top of the apparatus is open to the atmosphere, an attempt was made to enclose the entire top section with a polyethylene bag. The air inside was displaced by argon gas, and an argon blanket maintained at all times. However, enough air leaked in during adjustment of the liquid film thickness to ignite the pentaborane and destroy the enclosure several times. Finally, a box was built of half inch sheet Plexiglas around the top of the apparatus. This proved to be sufficiently rugged and leak-proof to enable the measurements to be made without further difficulty.

The top and sides of the box were sealed together with Plexiglas-acetone cement and fastened to the hot steel bar as one piece. The bottom was sealed to the glass pipe surrounding the steel bars. Thus the hot steel bar could be removed for cleaning. During a run, a rubber gasket was placed between the sides and bottom of the box, which were held together at the corners by four large C-clamps. Rubber gaskets were used around the micrometers, adjusting screws, hot water line, and thermocouple leads on the top of the box to allow adjustment of the liquid film thickness. Rubber gaskets were fastened to the Plexiglas with Armstrong adhesive 520. Over a period of time the rubber gaskets, adhesive (and Plexiglas to a lesser extent) absorbed pentaborane vapors and deteriorated.

Thermal Conductivity of Steel Bars

The thermal conductivity of the steel was determined by running heat flow measurements at several temperatures on n-propyl alcohol as a reference liquid. Sakiadis and Coates^(1,2) have made an extensive study of this liquid in their apparatus, and report the thermal conductivity of n-propyl alcohol is $0.0912 \text{ BTU/hr-ft}^2 (\text{°F/ft})$ at 100°F , and its temperature coefficient is $-0.95 \times 10^{-4} \text{ BTU/hr-ft}^2 (\text{°F/ft})/\text{°F}$.

It was observed that the apparent thermal conductivity of the steel was inversely proportional to the liquid film thickness of the n-propyl alcohol. Sakiadis and Coates⁽²⁾ reported that this effect was due to mechanical vibrations in the apparatus which increase the heat flow. Rather than try to eliminate the mechanical vibrations in the apparatus, due principally to stirrers in the constant temperature baths, it was decided to make all runs at one constant thickness. Therefore all runs with both n-propyl alcohol and pentaborane were made at or close to a film thickness 0.025 inches. Thus the effect of mechanical vibrations was the same during all measurements.

The results of six determinations of the thermal conductivity of steel are listed in Table A-IV (Appendix A). The thermal conductivity of the steel from 52 to 107°F is described by the equation

$$k_s = 29.43 + 0.0558 t$$

where k_s = thermal conductivity, BTU/hr-ft² (°F/ft)

t = temperature, °F.

References

- (1) Sakiadis, B. C., and Coates, J., A. I. Ch. E. Journal 1, 275-88 (1955).
- (2) Sakiadis, B. C., and Coates, J., "Studies of Thermal Conductivity of Liquids, Part III, A Thermoconductimetric Apparatus for Liquids", Louisiana State University, Engineering Experiment Station Bulletin No. 45, (1954).

Appendix A

Tables of Experimental Data

Table A-I	Vapor Pressure of Liquid Pentaborane from 25°C to 125°C
Table A-II	Vapor Pressure of Pentaborane, observed pressure readings
Table A-III	Thermal Conductivity of Liquid Pentaborane from 63°F to 104°F
Table A-IV	Thermal Conductivity of Steel Bars from 52°F to 107°F

TABLE A-I

VAPOR PRESSURE OF LIQUID PENTABORANE

Temperature °C	Vapor Pressure, pounds per square inch, absolute		Deviation obs.-calc.
	Observed	Calculated	
25.0	4.03	4.00 ¹	+ 0.03
35.0	6.01	6.01 ¹	0
45.0	8.65	8.78 ¹	-0.13
55.1	12.56	12.56 ¹	0
65.2	17.7	17.6 ¹	+ 0.1
75.3	23.9	24.0 ¹	-0.1
75.3	23.9	23.9 ²	0
85.4	31.9	32.0 ²	-0.1
95.5	41.9	42.0 ²	-0.1
105.7	54.7	54.1 ²	+ 0.6
115.8	68.3	68.2 ²	+ 0.1
125.8	84.2	84.3 ²	-0.1

¹ Calculated from equation $\log P = 5.6352 - 1384/(t + 250)$
Standard deviation = ± 0.1 PSIA

² Calculated from equation $\log P = 4.4803 - 730/(t + 160)$
Standard deviation = ± 0.3 PSIA

TABLE A-II

VAPOR PRESSURE OF PENTABORANE

<u>Time, Minutes</u>	<u>Observed Total Pressure, PSIA</u>					
0	0	0	0	0	0	0
10	---	5.96	---	---	17.5	22.3
15	---	---	8.67	12.65	---	---
20	---	6.06	---	---	17.8	24.0
30	4.13	6.08	8.89	12.75	17.8	24.1
40	---	6.11	---	---	17.9	24.2
45	---	---	9.02	12.83	---	---
50	---	6.13	---	---	17.9	24.4
60	---	6.16	9.13	12.93	17.95	24.5
70	---	6.18	---	---	18.0	24.6
80	---	6.20	---	---	18.0	24.7
90	---	6.22	---	---	18.05	24.8
100	---	6.25	---	---	18.1	24.9
110	---	6.27	---	---	18.15	25.0
120	---	6.30	---	---	18.15	25.1
130	---	6.32	---	---	18.15	25.2
140	---	6.34	---	---	18.2	25.4
150	4.52	6.37	---	---	18.25	25.5
Run Number*	1-1	4-2	2-3	2-5	10-3	8-4
Temperature, °C	25.0	35.0	45.0	55.1	65.2	75.3
Extrapolated Vapor Pressure at Zero Time, PSIA	4.03	6.01	8.65	12.56	17.7	23.9
Rate of Pressure Increase, PSI per Hour	0.20	0.14	0.48	0.37	0.20	0.60

* Run Number 4-2, for example, indicates fourth charge of pentaborane, second run

Table A-II
(Continued)

<u>Time, Minutes</u>	<u>Observed Total Pressure, PSIA</u>				
0	0	0	0	0	0
10	29.8	39.3	51.6	66.8	82.7
15	---	---	---	---	---
20	32.0	41.8	54.0	67.8	84.7
30	32.0	42.5	55.5	68.8	85.2
40	32.7	42.9	55.9	69.8	86.7
45	---	---	---	---	---
50	32.8	43.1	56.2	70.8	88.2
60	33.0	43.2	56.5	71.3	90.2
70	33.3	43.3	57.1	71.8	91.2
80	33.5	43.5	---	72.3	92.2
90	33.7	43.8	57.4	72.8	93.2
100	33.9	44.2	57.7	73.6	94.2
110	34.1	---	57.8	73.8	95.5
120	(34.2)	---	58.2	74.3	---
130	(34.2)	44.5	58.5	74.8	---
140	(34.3)	44.8	59.0	75.3	---
150	(34.4)	45.0	59.3	75.8	---
Run Number	8-5	8-6	9-2	11-2	11-3
Temperature, °C	85.4	95.5	105.7	115.8	125.8
Extrapolated Vapor Pressure at Zero Time, PSIA	31.9	41.9	54.7	68.3	84.2
Rate of Pressure Increase, PSI per Hour	1.20	1.24	1.84	3.00	6.00

TABLE A-II
(Continued)

DUPLICATE RUNS

<u>Time, Minutes</u>	<u>Observed Total Pressure, PSIA</u>				
0	0	0	0	0	0
10	---	8.52	11.95	16.55	59.1
15	6.01	---	---	---	---
20	---	8.67	12.55	17.65	64.8
30	6.20	8.72	12.65	17.8	69.5
40	---	8.74	12.8	17.9	71.1
45	6.27	---	---	---	---
50	---	8.76	12.8	18.05	71.9
60	6.38	8.80	12.8	18.05	72.4
70	---	8.82	12.9	18.05	73.4
80	---	8.84	12.9	18.15	74.0
90	---	8.98	12.9	18.3	74.7
100	---	9.00	13.0	18.3	---
110	---	9.00	13.0	18.3	---
120	---	9.08	13.05	18.3	---
130	---	9.08	13.05	18.45	---
140	---	9.10	13.15	18.45	---
150	---	9.14	13.15	18.5	---
Run Number	2-2	4-3	8-2	8-3	9-3
Temperature, °C	35.0	45.0	55.1	65.2	115.8
Extrapolated Vapor Pressure at Zero Time, PSIA	6.01	8.60	12.62	17.7	68.2
Rate of Pressure Increase, PSI per Hour	0.37	0.22	0.21	0.30	4.37

TABLE A-III

THERMAL CONDUCTIVITY OF LIQUID PENTABORANE

Expt. No.	Temperature		Thermal Conductivity, BTU/ft ² -hr (°F/ft)	
	°C	°F	Observed	Calculated*
L-23	17.52	63.53	0.0840	0.0831
L-32	22.29	72.12	0.0825	0.0825
L-21	27.14	80.85	0.0818	0.0818
L-24	37.46	99.42	0.0850 (?)	0.0806
L-33	39.93	103.88	0.0803	0.0803

* Calculated from the equation $k_L = 0.0874 - 6.84 \times 10^{-5} t$ (in °F)

TABLE A-IV

THERMAL CONDUCTIVITY OF STEEL BARS

(Based on n-propyl alcohol as reference)

Film thickness 0.025 inch

Temperature		Thermal Conductivity, BTU/hr-ft ² (°F/ft)		Deviation obs.-calc.
<u>°C</u>	<u>°F</u>	<u>Observed</u>	<u>Calculated*</u>	
11.38	52.49	32.33	32.36	-0.03
14.19	57.54	32.57	32.64	-0.07
20.79	69.43	33.47	33.30	0.17
33.18	91.72	34.46	34.55	-0.09
33.30	91.94	34.57	34.56	0.01
41.86	107.34	35.43	35.42	0.01

* Calculated from equation $k_s = 29.43 + 0.0558 t$ (in °F)Standard deviation = ± 0.09 BTU/hr-ft² (°F/ft)

Appendix B
Experimental Procedure

Section 1: Vapor Pressure

Section 2: Thermal Conductivity

Section 1

Experimental Procedure

Determining Vapor Pressure of Pentaborane from 25 to 125°C

The constant temperature bath, a 7-gallon Pyrex jar containing mineral oil, was brought to the desired temperature. Temperature was maintained constant within $\pm 0.1^\circ\text{C}$ with a Brown Pyro-vane controller. Temperatures were determined with mercury thermometers graduated in tenths of a degree and compared with a thermometer certified by the National Bureau of Standards. Stem corrections were made when applicable.

Two ml. of pentaborane was introduced into the glass tube of the modified Reid vapor pressure apparatus, directly from the storage container through a long hypodermic needle. The tube was first flushed out with dry nitrogen gas, and a nitrogen blanket maintained during the transfer. The tubing was quickly inserted into the body of the apparatus and tightened in place.

The sample was frozen at -80°C , and the apparatus was thoroughly evacuated through the side-arm. The Hoke valve was closed and the entire apparatus immersed in the constant temperature bath at zero time. During the early runs, pressure readings were taken every 15 minutes for an hour. During the later runs, readings were taken every 10 minutes for 150 minutes. Before each reading, the gauge was tapped to be sure the needle was free.

After the run, the apparatus was removed from the bath and cooled. The sample was immersed in a dry ice-acetone bath and degassed with a vacuum pump. It was stored at -80°C until the next run.

At the end of a series of runs, the pentaborane was frozen at -80°C and the tube removed from the apparatus. The tube was placed open side down in a beaker located in a safe place outside the laboratory. After the pentaborane had melted, propanol was added to the beaker from a distance. Later, methanol was added to hasten the alcoholysis of the sample. During all handling and disposal operations, the operator wore leather gloves and safety glasses.

Section 2

Experimental Procedure

Determining Thermal Conductivity of Liquid Pentaborane from 63°F to 104°F

The hot and cold constant temperature baths were fixed to the desired temperatures. The hot bath consisted of a 9-1/2 gallon glass jar, and the cold bath was a tall polyethylene bucket holding about 8 gallons. Both containers were set in wooden boxes and insulated with vermiculite. Each bath was stirred with a Fisher "Jumbo" electric stirrer, and maintained at constant temperature within $\pm 0.05^\circ\text{C}$ by an Aminco bimetallic thermoregulator. A small refrigerating unit supplied a slight excess of cooling to the cold bath. Eastern Industries Model D-11 centrifugal pumps circulated the water to the hot and cold steel bars. The sample chamber was thoroughly flushed with argon through two 18 gauge stainless steel tubes. About 50 ml of liquid pentaborane was loaded directly from a storage container under nitrogen pressure into the sample chamber through a hypodermic needle and a rubber serum bottle cap cemented to the top of the Plexiglas box. Any air or gas bubbles present were removed by tilting the apparatus or moving the hot bar. The hot and cold bars were brought into direct contact and the apparatus allowed to come to temperature equilibrium. The current in the heating jacket around the apparatus was adjusted to compensate for radial heat loss.

The emf readings of all the thermocouples were read by means of a Leeds and Northrup Type K potentiometer and reflecting galvanometer. The temperatures were determined in series several times until three successive readings indicated

that the apparatus had reached equilibrium. The film thickness was then adjusted to 0.025 inches and the apparatus again allowed to come to equilibrium. Temperature readings were taken the same as at zero thickness.

In order to remove the pentaborane safely, it was diluted with petroleum naptha and drawn out with a hypodermic syringe.

Appendix C

Calculations for Determining Thermal
Conductivity of Liquid Pentaborane
from 63°F to 104°F

Calculation of Thermal ConductivityNotation

- k_L = thermal conductivity of liquid, BTU/hr-ft² (°F/ft)
- k_s = thermal conductivity of the steel bars, at the average temperatures of layers C and D, BTU/hr-ft² (°F/ft)
- X_L = thickness of liquid layer, feet
- X_s = thickness of steel between thermocouple layers C and D
- X_s = 3.7432 inches or 0.31193 feet
- R_T = total resistance to heat flow at film thickness X_L , including resistance of liquid layer and steel bars, °F-ft/BTU
- R_0 = resistance to heat flow at zero apparent liquid film thickness, °F-ft/BTU
- A = arbitrary area of steel bars and liquid layer normal to direction of heat flow, ft²
- $\sum(\Delta t)$ = over-all temperature drop due to liquid layer and a known thickness of steel bars, °F
- $\sum(\Delta t_0)$ = over-all temperature drop due to a known thickness of steel bars for zero apparent liquid film thickness, °F
- q = heat flow through the liquid layer and through the known thickness of steel bars, BTU/hr
- q_0 = heat flow through the known thickness of steel bars at zero apparent liquid film thickness, BTU/hr

$\bar{B}$, $\bar{C}$, and $\bar{D}$ = average temperatures of B, C, and D layers respectively in the steel bars, °F

$\bar{B}_0$, $\bar{C}_0$, and $\bar{D}_0$ = average temperatures of B, C, and D layers respectively at zero apparent liquid film thickness, °F

From page 13

$$k_L = \frac{X_L}{A(R_T - R_O)} \quad (5)$$

and by definition

$$R_T = \frac{\sum (\Delta t)}{q} \quad (1)$$

where $\sum (\Delta t) = \bar{B} - \bar{D}$

$$q = \frac{k_s A (\bar{C} - \bar{D})}{X_s} \quad (6)$$

Also $R_O = \frac{\sum (\Delta t_o)}{q_o} \quad (7)$

where $\sum (\Delta t_o) = \bar{B}_0 - \bar{D}_0$

$$q_o = \frac{k_s A (\bar{C}_0 - \bar{D}_0)}{X_s} \quad (8)$$

Substituting equations (1), (6), (7), and (8) in equation (5),

$$k_L = \frac{X_L}{A \left[\frac{X_s (\bar{B} - \bar{D})}{k_s A (\bar{C} - \bar{D})} - \frac{X_s (\bar{B}_0 - \bar{D}_0)}{k_s A (\bar{C}_0 - \bar{D}_0)} \right]}$$

$$k_L = \frac{X_L k_S}{X_S \left(\frac{\bar{B} - \bar{D}}{\bar{C} - \bar{D}} - \frac{\bar{B}_O - \bar{D}_O}{\bar{C}_O - \bar{D}_O} \right)} \quad (10)$$

Calculation of Average Temperature of Liquid Film, t_L , °F

The temperature of the liquid film is taken as the arithmetical average of the temperatures of the two liquid-metal interfaces, which are determined by extrapolation from the C and D layers.

0.3954 inches = distance of B layer from hot bar - liquid interface

0.7290 inches = distance of C layer from cold bar - liquid interface

3.7432 inches = distance between C and D layers

t_1 = extrapolated temperature of hot liquid surface, °F

t_2 = extrapolated temperature of cold liquid surface, °F

$$t_1 = \bar{B} - \frac{0.3954}{3.7432} (\bar{C} - \bar{D}) \quad (11)$$

$$t_2 = \bar{C} + \frac{0.7290}{3.7432} (\bar{C} - \bar{D}) \quad (12)$$

and the average temperature of the liquid film

$$t_L = \frac{t_1 + t_2}{2} \quad (13)$$

Sample Calculation (Expt. No. L-32)

Run Conditions

Hot water bath, 77.0°F

Cold water bath, 49.1 °F

Experimental Data

Average thermocouple readings and temperatures

when $X_L = 0.0000$ in.

<u>Layer</u>	<u>Millivolts</u>	<u>°F</u>
$\bar{B}_0$	0.9480	75.14
$\bar{C}_0$	0.9124	73.58
$\bar{D}_0$	0.8464	70.66

when $X_L = 0.0252$ in.

<u>Layer</u>	<u>Millivolts</u>	<u>°F</u>
$\bar{B}$	0.9555	75.48
$\bar{C}$	0.7999	68.57
$\bar{D}$	0.7524	66.44

Calculations

Average temperature, layers C and D at .0252 in. = 67.505°F

$$\therefore k_s = 29.43 \quad 0.0558 (67.505)$$

$$k_s = 33.20 \text{ BTU/hr-ft}^2 (\text{°F/ft})$$

and $k_L =$

$$3.7432 \left[\frac{(33.20) (0.0252)}{\left(\frac{75.48 - 66.44}{68.57 - 66.44} \right) - \left(\frac{75.14 - 70.66}{73.58 - 70.66} \right)} \right]$$

$$k_L = 0.0825 \text{ BTU/hr-ft}^2 (\text{°F/ft})$$

$$t_1 = 75.48 - 0.1056 (68.57 - 66.44)$$

$$t_1 = 75.26 ^\circ\text{F}$$

$$t_2 = 68.57 + 0.1948 (68.57 - 66.44)$$

$$t_2 = 68.98 ^\circ\text{F}$$

and

$$t_L = \frac{75.26 + 68.98}{2} = 72.12 ^\circ\text{F}$$

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