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SRI Report 951581-4A *September 30, 1969*

PHYSICAL PROPERTIES OF LIQUID OXYGEN DIFLUORIDE AND LIQUID DIBORANE: A CRITICAL REVIEW UPDATED

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CONTRACT 951581
UNDER NAS7-100

SRI Project PRD 6063

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ABSTRACT

This report includes a complete survey of the available literature on the physical and thermodynamic properties of liquid oxygen difluoride and liquid diborane up to September 1, 1969. The report contains all information previously published July 1, 1967, in SRI Report No. 951581-4 with very minor changes in literature references. The survey has shown that no new work on physical properties has been reported from July 1, 1967 to the present. Recommendations for further work were included in the earlier report and these have been restated along with recommendations for more studies on liquid OF_2 and liquid B_2H_6.

FOREWORD

This literature survey was carried out under TDM No. 66X08900, which completed a critical review of published information on the physical properties of liquid OF_2 and liquid B_2H_6 up to July 1, 1967, and under TDM 69X03200 where it was updated to September 1, 1969. Under the earlier TDM, the work was carried out by R. F. Muraca, Director, Analyses and Instrumentation, J. S. Whittick, and J. Neff. The more recent survey was performed under the Synthesis Research Program of Stanford Research Institute, M. E. Hill, Director, W. E. Tolberg, Project Leader, and Derek Tegg. The Technical Cognizance for JPL for the present report was the responsibility of L. R. Toth.

I INTRODUCTION

This report is an updated version of SRI Report No. 951581-4 entitled "Physical Properties of Liquid Oxygen Difluoride and Liquid Diborane: A Critical Review" by J. S. Whittick, J. Neff, and R. F. Muraca. The report was brought up to date by a survey of the literature from July 1, 1967, the date of Report No. 951581-4, to September 1, 1969.

Literature sources reviewed were Chemical Abstracts and abstracts of Scientific and Technical Aerospace Reports, U. S. Government R&D reports, and International Aerospace Abstracts. Also reviewed were CPIA reports, as well as data from manufacturers. The literature search and critical review resulted in several recommendations for further work.

The recommendations made in SRI Report 951581-4 are still valid and some new recommendations have been generated during the latest review. These are discussed in Section II.

A summary and evaluation of available data for the properties of OF_2 are given in Section III and for the properties of B_2H_6 in Section IV. Original data have been tabulated and are accompanied by graphs (where practical) to illustrate concordance or conflicts in the measurements and calculations of various workers. A brief discussion is given of the values for each property, and suggestions are made regarding the validity of the data. A summary of selected data for OF_2 and B_2H_6 is provided in Section V.

II DISCUSSION

Although there are numerous papers in the literature on oxygen difluoride and diborane, there are no published reports of experimental work on the physical properties of these two compounds from July 1, 1967 to the date of this report. Consequently, all references used for deriving the data included in this report are prior to July 1, 1967. One paper was found that discussed significant structure theory and the properties of liquid B_2H_6.[1] The authors calculated vapor pressure, entropy of vaporization, thermal expansion, heat capacity, critical constants, and surface tension. Their calculated results agreed very well with experimental values. Such agreement in turn lends credence to the values reviewed in SRI Report No. 951581-4.

The recommendations of this SRI report for further work on the physical properties of liquid OF_2 and B_2H_6 are still valid. It was recommended that work be done on OF_2 to determine (1) the density matrix or compressibility between 146 and 800 psia at temperatures between 125 and 210°K, (2) the surface tension between 50 and 125°K, and (3) the heat capacity between 50 and 125°K. The missing data for B_2H_6 are (1) the compressibility at very high and very low temperatures, and (2) the thermal conductivity.

In addition to the above recommendations, and in view of the intended use of the OF_2/B_2H_6 system as a space-storable propellant, it seems advisable at this time to point out two more areas of interest for further work. Significantly, there exist no specifications for either OF_2 or B_2H_6 as propellant ingredients. It seems appropriate at this time that propellant specifications be worked out. The second added recommendation involves physical properties of the pure materials as compared with those of specified propellant grade. It would seem desirable to determine the presently unavailable properties of pure compounds and to compare these properties with those of propellant grade materials at a few check points when propellant specifications become available.

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[1] M. S. Jhon, J. Grosh, and H. Eyring, J. Phys. Chem., 71, 2253 (1967).

III . PHYSICAL PROPERTIES OF LIQUID OXYGEN DIFLUORIDE

Reviews and/or compilations of data on the physical properties of OF_2 are provided by Allied Chemical (1), George (5), Thiokol Chemical (17), and Streng (15). The discussions given below are the result of examination and comparison of procedures and values as described in primary references.

General Properties

The general properties of liquid OF_2 are summarized in Table 1.

Melting Point. - The melting point of 98.5% OF_2 (assayed iodometrically) was determined by Ruff and Clusius in 1930 (10). The melting point curve published by the authors shows a small inflection, probably due to impurities. Although these workers eventually obtained purer material by fractionation in order to enable accurate determination of other properties, the melting point apparently was not re-determined. The determination has not been repeated by other workers; the value originally reported by Ruff and Clusius (-223.8°C) is quoted by a manufacturer (1) and appears in the Handbook of Chemistry and Physics (6).

Boiling Point. - Several values have been reported for the boiling point of OF_2; however, the earliest reported value (11) of -146.5°C (98.5% OF_2) must be discarded, since the authors later revised the value to -144.8°C, based on 99.8% material (12). The values of -144.8°C (obtained in 1931) and -145.3°C (14, 1951) are generally reported together, except in the case of the Handbook of Chemistry and Physics (6), where only -144.8°C is cited. The most recent reported value is -145.2°C (17, 1962). The three values are obtained by extrapolation of quite similar vapor pressure data, and purity is reported to be 99.6% or better. The authors reporting -145.3°C (14) employed infrared spectroscopy to show that their material contained only 0.1% SiF_4 and 0.002% CF_4 as impurities. The material used to obtain the -145.2°C value (17) was reported to have been assayed by mass spectroscopy, infrared analyses, and gas chromatography, indicating a purity of not less than 98.6%. However, trace impurities such as oxygen and fluoride are not detectable by infrared analyses and fluorine is difficult to assay by mass spectroscopy or gas chromatography unless the systems are carefully passivated.

The accuracy of temperature measurements in the above determinations was estimated to be within ±0.2°C of the reported values. For the latest determination (-145.2°C), a Mueller bridge-platinum resistance thermometer was used to measure temperature. Other temperature values were determined by calibrated thermocouples.

Critical Constants. - Two independent values of the critical temperature are reported by Anderson *et al*. (2) as -58.0 ± 0.1°C and by Thiokol-Denville (17) as -59.7 ± 0.3°C. The first method of determination is straightforward; the authors observed the temperature of appearance and disappearance of a meniscus in a sealed glass capillary. The latter method employed a small stainless steel bomb to contain the liquid and gaseous OF_2. These authors determined temperatures at which discontinuities occurred in the vapor pressure-temperature curve as a function of liquid mass in the constant-volume apparatus. Low mass loading results in complete evaporation of the liquid sample below the critical temperature and a break in the standard vapor pressure curve; this break is toward the low pressure side. At high mass loading, the liquid does not evaporate and, as the critical temperature is approached, a sharp break toward extreme pressure is noted in the constant volume. Several values of temperature at which these breaks occurred were measured; these temperatures were plotted as a function of mass loading $\left(\frac{\text{volume = constant}}{\text{mass}}\right)$. This produced a parabolic curve from which a maximum $T = T_c$, the critical temperature, was obtained. At the critical temperature, the volume/mass was found to be 2.33 cc/g. The pressure was 725 ± 5 psia (49.5 atm). (This report did not include actual measured values; it reproduced only the graphs used to obtain these values.) These are the best values available because Anderson (2) estimated the critical volume (1.81 cc/g) by extrapolation of his measured densities to T_c, applying the law of rectilinear diameter. Using the Dieterici equation, he also calculated $P_c = 48.9$ atm.

Heat of Vaporization. - The calculated value (Clausius-Clapeyron-vapor pressure) of 2.650 kcal/mole at -144.8°C (12) for the heat of vaporization of OF_2 is widely accepted and compares favorably with the most recent value of 2.66 kcal/mole (17). The values reported in References 10 and 15 are in a sense verified by the similarity of the vapor pressure curves determined by Schnizlein (14). The value of 2.680 kcal/mole in a product bulletin (1) is apparently a misprint which has been perpetuated by at least one reviewer (5).

Heat of Formation. - A lively interchange of chemical data and discussions between Ruff and Menzel (11) and Wartenburg and Klinkott (19) led to a re-determination and re-calculation of values for the heat of formation of OF_2 (18, 13) and a final average of their values as 7 ± 2 kcal/mole (13). Subsequently, the same chemical data were analyzed at the National Bureau of Standards with the published result of 7.6 ± 2 kcal/mole (4). Thiokol (17) reported that an experimental ΔH_f would be determined from the hypergolic reaction of OF_2 and H_2; if this determination has been made, it is not found in the open literature.

A report by Bisbee (3) indicates that a new value of the heat of formation, -4.06 kcal/mole was obtained from the reaction:

$$OF_2 + 2H_2 \rightarrow H_2O + 2HF \text{ (infinite dilution)}$$

This value was probably accepted for several months as the best value because a similar value of -5.2 kcal/mole appeared as a technical note (Z70-1) in the National Bureau of Standards' "Selected Values of Chemical Thermodynamic Properties." However, the Bureau of Standards (8) re-determined the value and found it to be 5.86 ± 0.03 kcal/mole. The new value was obtained by determining the heat of reaction of H_2 with OF_2, F_2, and O_2, and agrees with the earlier plus values. A critical review of earlier work is included in this report.

Thermal Conductivity. - The thermal conductivity was measured at -195.8°C and at -183°C (17) and was found to be an average (3 determinations) of 0.00058 and 0.0006 cal/sec/cm^2/°C/cm, respectively, at the two temperatures.

Vapor Pressure

Original data for vapor pressure *vs* temperature are summarized in Tables 2, 3, 4, and 5. For comparative purposes, the data are plotted in Figure 1. It is immediately evident that the curves for 98.5% or 99.8% OF_2 (11, 12) do not vary significantly. It is interesting to note that the more recent data for 99.8% OF_2 (14) and 99.6% OF_2 (17) falls on either of the two curves in the region 78°-110°K, finally overlapping the prior 99.8% curve in the region 110°-130°K. All determinations of vapor pressure were repeated on re-distilled portions of OF_2; hence, the authors insist that all errors are largely attributable to calibration of thermocouple and other measuring devices, but the possibility of oxygen impurities still cannot be eliminated. The determinations by Thiokol (17) were made with platinum resistance thermometers and are reported to be within ±0.2°C accuracy.

Density

Original data on the variation of the density of liquid OF_2 with temperature are given in Tables 6, 7, and 8 and are plotted in Figure 2. Although the data of Anderson *et al*. (2) are more recent than that of Ruff and Menzel (12), the range covered is extremely narrow and not as amenable to extrapolation. Further, the data of Ruff and Menzel were obtained directly by pycnometer readings, whereas that of Anderson *et al*. were obtained by an indirect flotation method. The data reported by Thiokol (17) were also obtained by a pycnometric method and are in excellent agreement with that of Ruff and Menzel (12) over the range 130°-110°K, deviating to about 0.5% higher values at 80°K.

A value of 1.65 g/cc at -190°C (83°K) for OF_2 (Table 7) was selected by the editors of the Handbook (6); this value was first reported in 1930 for 98.5% OF_2 by Ruff and Menzel (11), but revised in 1931 by these authors (12). Thus, the Handbook figures must be ignored.

No information was found on the determination of densities under pressure. It is recommended that PVT measurements be made to obtain the density matrix between 14.6 and 800 psia at temperatures between -144.9° and -62°C.

Viscosity

The first reference on the viscosity of OF_2 (2) covers a very narrow range of temperature near the boiling point. These data are summarized in Table 10 and plotted in Figure 3. The data have been extrapolated by Streng (15) to 300°K (as 168 µp). More recent data (17) are summarized in Table 11 and also plotted in Figure 3.

Surface Tension

No data were found on the surface tension of liquid OF_2. The importance of this value in estimating expulsion and storage parameters in a gravity-free environment dictates the need for its determination.

Thermodynamic Functions

The behavior of various thermodynamic functions of liquid OF_2 with temperature, calculated from spectroscopic data, is illustrated in Tables 12 and 13. The data in Table 12 (4, 7) have been widely circulated and are considered the most acceptable until actual experimental data may be derived; the data in Table 13 (8) illustrate agreement of calculations from spectroscopic data. The values in Table 14 for experimentally-determined heat capacities of OF_2 over the region 82°-198°K(16) are at wide variance with that formerly accepted. Thus, it is suggested that more experimental work be performed on the determination of the heat capacity of OF_2.

Table 1

SUMMARY OF GENERAL PROPERTIES OF LIQUID OF_2

PROPERTY	VALUES	REMARKS	REF.
Melting Point	-223.8°C, 49.4°K	98.5% purity	(10)
Boiling Point	-144.8°C, 128.4°K		(12)
	-145.3°C, 127.9°K		(14)
	-146.5°C, 126.7°K	98.5% purity	(11)
	-145.2°C, 128.0°K		(17)
Critical Temperature	-58.0°C, 215.2°K	experimental	(2)
	-81°C, 192°K	calculated	(12)
	-59.7°C, 213.5°K	experimental	(17)
Critical Pressure	48.9 atm, 719 psia	calculated	(2)
	49.5 atm, 725 psia	experimental	(17)
Critical Density	0.553 g/cc	extrapolated	(2)
	0.425 g/cc	experimental	(17)
Critical Volume	97.6 cc/mole	calculated	(2)
	127.0 cc/mole	experimental	(17)
Heat of Vaporization	2.650 kcal/mole		(12)
	2.66 kcal/mole		(17)
	2.680 kcal/mole	(apparent misprint)	(1)
Trouton's Constant	20.65		(12)
Heat of Formation	4.6 kcal/mole	chemical data	(11)
	11 kcal/mole	chemical data	(19)
	9.0 kcal/mole	chemical data	(18)
	7 ± 2 kcal/mole	chemical data	(13)
	7.6 ± 2 kcal/mole	computed from (9) and (16)	(4)
	-4.06 kcal/mole	computed	(3)
	5.86 ± 0.03 kcal/mole	chemical data	(8)

Table 2

VAPOR PRESSURE OF LIQUID OF_2 (Ref. 11)*

TEMPERATURE		VAPOR PRESSURE mm	TEMPERATURE		VAPOR PRESSURE mm
°K	°C		°K	°C	
81.6	-191.5	3.2	112.1	-161	211.9
89.6	-183.5	12.4	116.1	-157	289.4
105.1	-168	93.2	122.6	-150.5	546.0
110.1	-163	169.7	125.1	-148	692.6

* 98.5% purity

Table 3

VAPOR PRESSURE OF LIQUID OF_2 (Ref. 12)

$$\left(\log P_{mm} = 7.3892 - \frac{578.64}{T°K}\right)$$

TEMPERATURE		VAPOR PRESSURE mm	TEMPERATURE		VAPOR PRESSURE mm
°K	°C		°K	°C	
80.9	-192.2	1.6	110.6	-162.5	145.0
82.9	-190.2	2.6	112.7	-160.4	180.4
84.3	-188.8	3.3	116.7	-156.4	270.2
85.4	-187.7	4.2	118.4	-154.7	316.7
87.9	-185.2	6.3	120.6	-152.5	393.5
91.2	-181.9	10.9	121.9	-151.2	439.5
94.0	-179.1	17.2	123.4	-149.7	501.0
95.9	-177.2	22.9	123.8	-149.3	521.8
98.1	-175.0	30.8	124.3	-148.8	546.4
100.1	-173.0	41.3	125.2	-147.9	598.2
101.8	-171.3	50.4	125.4	-147.7	598.2
103.4	-169.7	62.2	126.3	-146.8	646.9
104.9	-168.2	76.0	127.0	-146.1	680.9
106.3	-166.8	89.6	127.9	-145.2	733.1
109.8	-163.3	134.0	128.1	-145.0	737.9

Table 4

VAPOR PRESSURE OF LIQUID OF_2 (Ref. 14)

$$\left(\log P_{mm} = 7.2242 - \frac{555.42}{T^{\circ}K}\right)$$

TEMPERATURE		VAPOR PRESSURE mm	TEMPERATURE		VAPOR PRESSURE mm
°K	°C		°K	°C	
77.8	-195.4	1.4	103.6	-169.6	73.0
77.9	-195.3	2.1	103.7	-169.5	73.5
79.3	-193.9	1.7	103.7	-169.5	73.9
81.1	-192.1	2.9	103.7	-169.5	75.0
83.1	-190.1	4.7	110.0	-163.2	140.2
87.5	-185.7	7.6	110.0	-163.2	142.8
87.6	-185.6	8.0	115.9	-157.3	273.0
88.2	-184.0	11.6	116.2	-157.3	279.1
91.5	-181.7	14.3	121.2	-152.0	422.7
91.9	-181.3	15.4	121.2	-152.0	424.4
92.3	-180.9	16.3	121.8	-151.4	471.9
93.3	-179.9	18.3	121.8	-151.4	472.9
93.2	-179.8	18.5	122.0	-151.2	471.3
93.9	-179.3	19.4	124.8	-148.4	577.0
94.1	-178.1	20.6	125.3	-147.9	618.8
95.1	-178.1	24.1	127.2	-146.0	735.0
100.5	-172.7	47.3	127.9	-145.3	740.0
103.6	-169.6	72.2			

Table 5

VAPOR PRESSURE OF LIQUID OF_2 (Ref. 17)

$$\left(\log P_{mm} = 7.4199 - \frac{581.19}{T^{\circ}K}\right)$$

TEMPERATURE		VAPOR PRESSURE mm	TEMPERATURE		VAPOR PRESSURE mm
°K	°C		°K	°C	
90.80	-182.28	10.4	115.34	-157.72	247.1
93.58	-179.58	15.5	118.19	-154.97	321.7
94.48	-178.68	18.5	120.03	-153.13	381.6
96.30	-176.86	25.0	120.48	-152.68	396.3
98.11	-175.05	31.7	120.94	-152.22	414.5
100.82	-172.34	46.6	123.71	-149.45	526.3
103.55	-169.61	65.0	124.64	-148.52	569.8
107.20	-165.96	101.0	126.48	-146.68	662.1
109.94	-163.22	138.9	127.40	-145.76	710.3
110.85	-162.31	154.4	127.86	-145.30	735.9
114.51	-158.65	243.3			

Table 6

DENSITY OF LIQUID OF_2 (Ref. 12)

TEMPERATURE		DENSITY*, g/cc	EQUATION
°K	°C		
126	-147	1.54	$d_{liq} = 2.135 - 0.004695T°K$
83	-190	1.74	
81	-192	1.75	
75	-198	1.78	
73	-200	1.79	
70	-203	1.80	
49.4	-223.8	1.90 (ext.)	

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* Estimated from published curve.

Table 7

DENSITY OF LIQUID OF_2 (Ref. 2)

TEMPERATURE		DENSITY, g/cc	EQUATION
°K	°C		
127.9	-145.3	1.521 (ext.)	$d_{liq} = 2.190 - 0.00523T°K$ (Ref. 1,13)
125.9	-147.3	1.528	
125.5	-147.7	1.531	
123.1	-150.1	1.538	
122.7	-150.5	1.547	
117.9	-155.3	1.569	
117.3	-155.9	1.573	

Table 8

DENSITY OF LIQUID OF_2 (Ref. 6)

TEMPERATURE		DENSITY, g/cc	REMARKS
°K	°C		
83	-190	1.65	(from Ref. 9; 98.5% purity)

Table 9

DENSITY OF LIQUID OF_2 (Ref. 17)

TEMPERATURE		DENSITY, g/cc	EQUATION
°K	°C		
77.46	-195.70	1.776	$d_{liq} = 0.8225 - 0.004873T°C$
90.88	-182.28	1.709	
91.78	-181.38	1.706	
94.49	-178.67	1.695	
95.40	-177.76	1.688	
97.21	-175.95	1.681	
99.02	-174.14	1.672	
101.74	-171.42	1.659	
105.38	-167.78	1.642	
106.29	-166.87	1.635	
109.94	-163.22	1.619	
110.86	-162.30	1.611	
114.42	-158.64	1.598	
115.44	-157.72	1.592	
119.11	-154.05	1.576	
120.03	-143.13	1.570	
124.64	-148.52	1.546	

Table 10

VISCOSITY OF LIQUID OF_2 (Ref. 2)

TEMPERATURE		VISCOSITY, cp	EQUATION
°K	°C		
127.9	-145.3	0.2826 (ext.)	$\log \eta = \frac{131.5}{T°K} - 1.5768$
127.4	-145.8	0.2852	
126.0	-147.2	0.2937	
125.8	-147.4	0.2933	
125.7	-147.5	0.2962	
125.3	-147.9	0.2998	
124.5	-148.7	0.3014	
124.4	-148.8	0.3024	
122.6	-150.6	0.3140	
122.4	-150.8	0.3129	
122.2	-151.0	0.3134	
122.1	-151.1	0.3176	
121.9	-151.3	0.3171	
121.7	-151.5	0.3188	
121.2	-152.0	0.3227	
120.4	-152.8	0.3259	

Table 11

VISCOSITY OF LIQUID OF_2 (Ref. 17)

TEMPERATURE		DENSITY, g/cc	VISCOSITY, cp	EQUATION
°K	°C			
77.5	-195.7	1.776	1.004	$\log \eta = \frac{112.4}{T°K} - 1.4508$
90.2	-183.0	1.711	0.654	
93.0	-180.2	1.700	0.572	
95.4	-177.8	1.688	0.522	
99.1	-174.1	1.669	0.470	
103.6	-169.6	1.647	0.422	
108.2	-165.0	1.625	0.376	
112.7	-160.5	1.604	0.344	
117.4	-155.8	1.581	0.323	

Table 12

THERMODYNAMIC FUNCTIONS OF LIQUID OF_2 (Ref. 4,7)

TEMPERATURE		Cal. $mole^{-1}deg^{-1}$			Kcal. $mole^{-1}$			
°K	°C	C_p^o	S^o	$-(F^o - H^o_{298})/T$	$H^o - H^o_{298}$	ΔH_{f^o}	F_{f^o}	log K_p
0	-273.2	0.000	0.000	Infinite	-2.604	8.143	8.143	Infinite
100	-173.2	8.067	19.409	67.487	-1.808	7.888	9.093	-19.872
200	-73.2	9.098	55.275	60.053	-0.956	7.700	10.377	-11.399

Table 13

SOME THERMODYNAMIC FUNCTIONS OF LIQUID OF_2 (Ref. 8)

TEMPERATURE		Cal $mole^{-1}deg^{-1}$			
°K	°C	C_p^o	S^o	$-(F^o - E^o_0)/T$	$(H_0 - E^o_0)/T$
100	-173.2	8.066	49.231	41.267	7.964
150	-123.2	8.494	52.573	44.511	8.062
200	-73.2	9.087	55.092	46.852	8.240

E^o_0 = energy of one mole of perfect gas at absolute temperature

Table 14

HEAT CAPACITY OF LIQUID OF_2 (Ref. 16)

TEMPERATURE		C_p, cal/mole/deg
°K	°C	
82.1	-191.1	17.820
82.2	-191.0	17.496
82.2	-191.0	17.658
83.9	-189.3	17.658
100.0	-173.2	18.090
101.7	-171.5	17.928
101.8	-171.4	18.144
102.9	-170.3	18.090
117.1	-156.1	18.198
117.4	-155.8	18.198
118.2	-155.0	18.360
125.7	-147.5	18.414
126.1	-147.1	18.738
126.5	-146.7	18.522
128.9	-148.3	18.630
138.5	-134.7	18.684
139.2	-134.0	18.846
140.9	-132.3	18.738
143.1	-130.1	18.630
159.2	-114.0	19.062
160.3	-112.9	19.440
163.1	-110.1	19.278
195.2	-78.0	19.818
195.9	-77.3	19.926
197.1	-76.1	19.980
197.9	-75.3	20.142

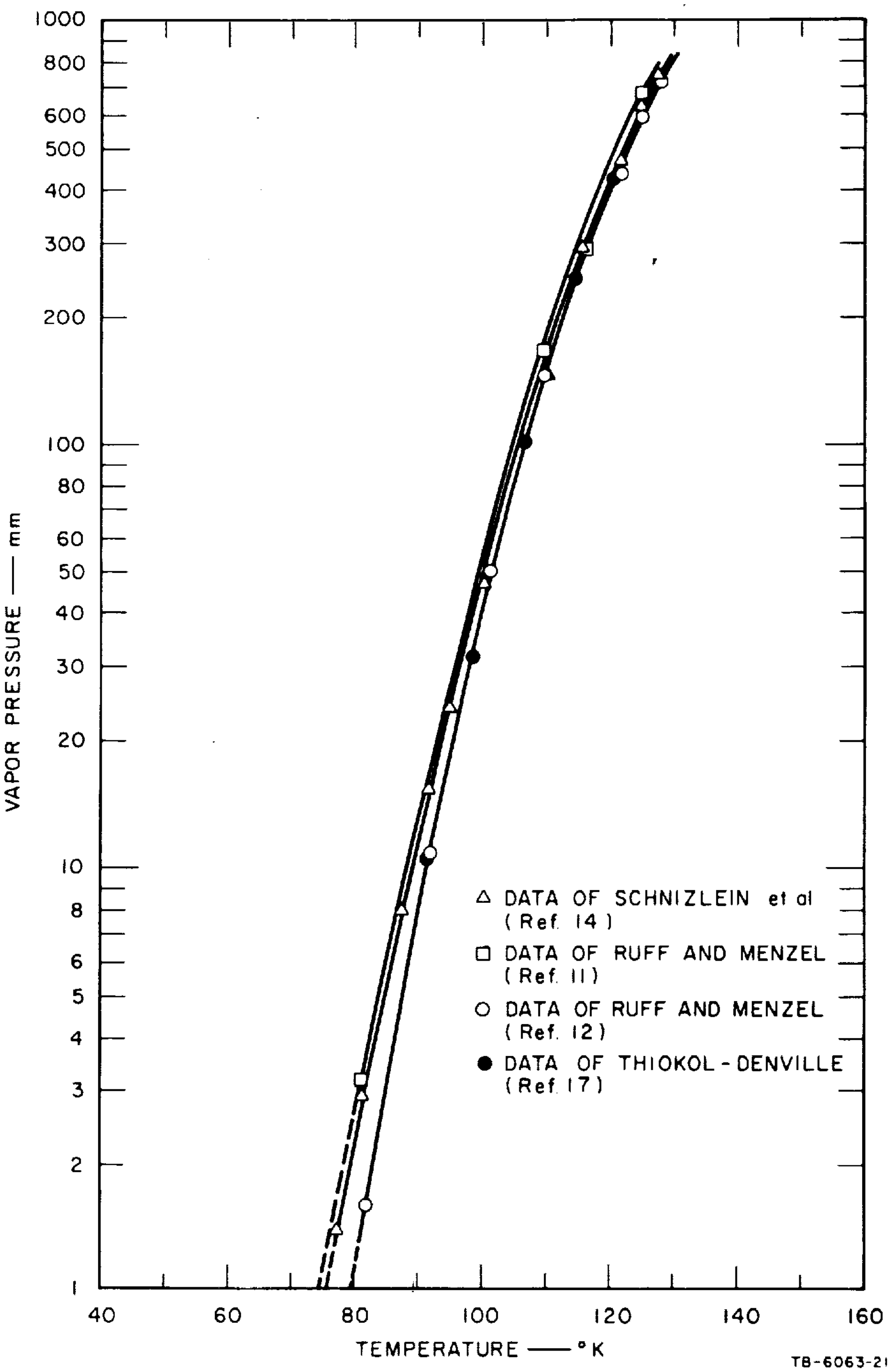

FIG. 1 VAPOR PRESSURE OF LIQUID OF_2

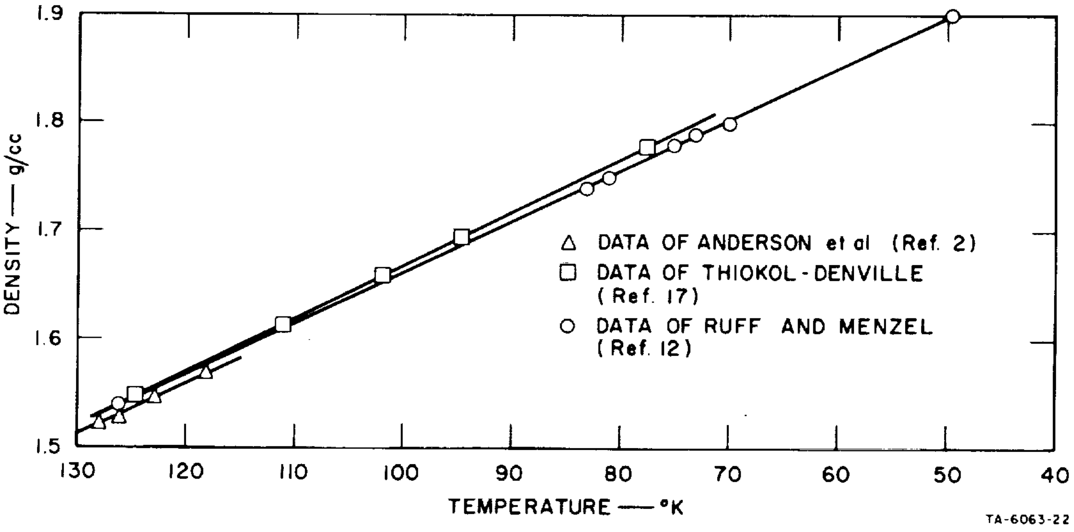

FIG. 2 DENSITY OF LIQUID OF_2

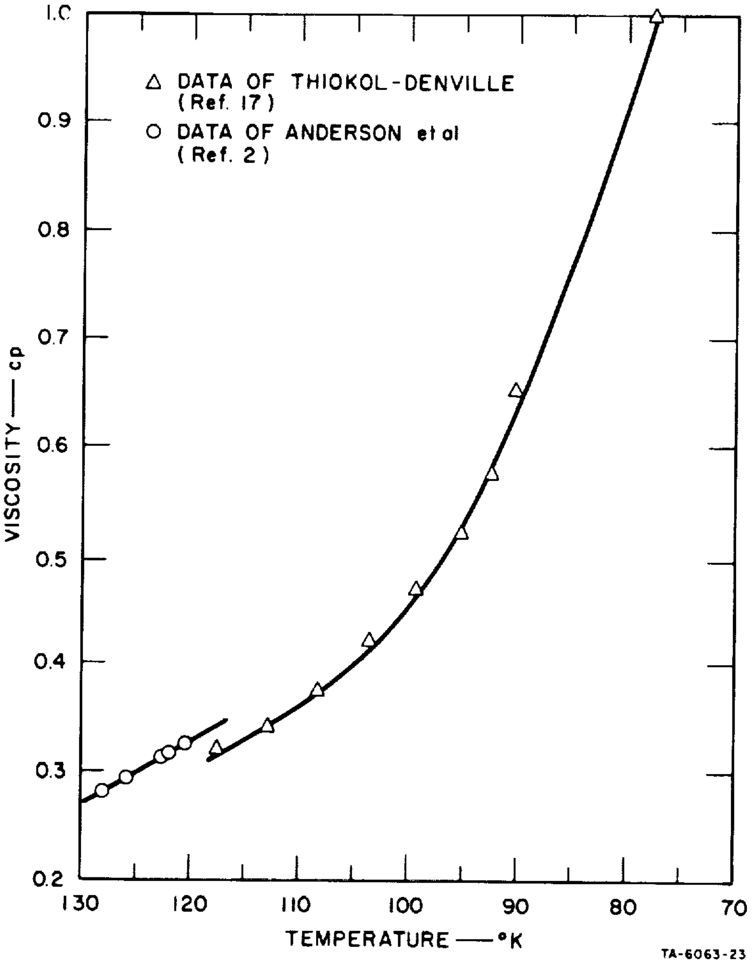

FIG. 3 VISCOSITY OF LIQUID OF_2

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IV . PHYSICAL PROPERTIES OF LIQUID DIBORANE

Reviews and/or compilations of data on the physical properties of B_2H_6 are provided by Callery Chemical (1), Mook (8), Smith (15), and Stock and Kuss (17). The discussions given below are the result of examination and comparison of procedures and values as described in primary references.

General Properties

Summarized in Table 15 are published values for various general properties of liquid diborane (B_2H_6).

Melting Point. - The melting point value of 108.30°K (-164.86°C) is considered to be valid since it was measured independently in 1953 and in 1956 by different workers (2, 19) on material determined to be 99.94% purity, using rigidly-calibrated thermocouples.

The melting point of -165.5°C (107.7°K) published in a Handbook (6), a supplier's brochure (1), and a text (16) undoubtedly is the value obtained by Stock and Kuss in 1923 (17) on material of "regal" purity, using a methane thermometer. The purity of material is not questioned as much as the calibration of the thermometer.

Boiling Point. - As shown in Table 15, there is only about 0.2% deviation in the experimentally-determined values for the boiling point of diborane. An average of the four most comparable values (10, 14, 17, 19) is 180.65°K (-92.53°C), in agreement with the value determined by Wirth and Palmer (19).

Heat of Vaporization. - The heat of vaporization of diborane was calculated from vapor pressure data to be 3.405 and 3.413 kcal/mol by independent workers (12, 19); an average value of 3.422 kcal/mol was obtained by calorimetric determinations (2). In view of the small differences in reported values, an average figure of 3.413 kcal/mol seems appropriate.

Heat of Formation. - The heat of formation of diborane selected by a supplier as 7.53 kcal/mol (1) is based on a report issued in 1955 and concerned with the estimation of heats of combustion of organoboranes. At this time, the experimentally-determined value of 6.73 kcal/mol published in 1958 (13) appears to be more accurate. [Earlier values of +44 kcal/mol (in 1937) and -26 kcal/mol (in 1949) have not been included in the tablulated summary in view of the near-agreement of more recent values.]

Critical Constants. - The critical temperature and pressure of diborane were determined experimentally (9) and (21) to be 16.7°C, and (9) 580 psia, and have been accepted widely. The critical volume of 170 cc was estimated in order to complete a heat-capacity curve for liquid diborane, and was shown to be justified by the smoothness of the heat-capacity curve (14).

Density

Density values for liquid diborane as determined experimentally by several workers are summarized in Tables 16, 17, and 18. The plot of their combined data shown in Figure 4 indicates excellent agreement and a smooth curve. It is suggested that the data of Smith and Miller (15) be used for computations since it covers the entire range from 140° to 260°K.

The data given in Table 19 were prepared from a computer program (5) based on densities calculated from pressure-volume-temperature relationships (11). Comparison of this data plot in Figure 5 with that in Figure 4 reveals that the only comparable point is around 200°K. At the lower temperatures, density values are much lower than shown in Figure 4 and at the higher temperatures, much higher. Thus, this computed data must be disregarded at this time.

Compressibility

Compressibility of liquid diborane has been derived (Table 20 and Figure 6) from PVT data by Smith (15) and determined directly (Table 21 and Figure 7) by Paridon (11). Compressibilities from the two sources are in sharp disagreement and it appears at first that Paridon's work should be selected as more complete and precise. Galbraith (5) actually chose Paridon's data (and other selected physical properties) to prepare a general equation of state for liquid and gaseous diborane. However, the density data of Smith agrees with other reported densities and the saturated density values of Paridon (obtained by extrapolation down to the vapor pressure) do not. Paridon used much larger quantities of diborane for his studies than Smith and his values are reported with greater precision. He also had the advantage of more modern handling techniques for diborane. Other physical property data by Paridon and co-workers (11, 12) published at about the same time are in general agreement with other accepted published values. It is surprising that Paridon's compressibility data have not been published in the technical journals. Since this discrepancy exists, some independent checks, especially at very low and very high pressures should be made.

Viscosity

A log-plot of the viscosity of liquid diborane over the range 145° to 204°K (15) follows a straight line, as shown in Figure 8, suggesting that extrapolation can be performed over a short range. The raw data and the resulting equation are given in Table 22.

Surface Tension

The surface tension data given in Table 23 (7) form a smooth straight-line plot (Figure 9) for liquid diborane over the region 140° to 165°K, and should certainly be amenable to extrapolation over the liquid range. Of the data given in Table 24 (15), one point lies exactly on this curve (at 151.6°K), but a point beyond the boiling point is well away from a straight line (at 203.5°K).

Vapor Pressure

The most common ground for agreement in the measurement of the physical properties of liquid diborane lies in the determination of the vapor pressure curve. Any of the data summarized in Tables 25 to 30 can be fitted with excellent agreement to the curve plotted in Figure 10.

The data of Wirth and Palmer (19) were selected for presentation in graphical form because they are representative of all experimenter's work and because they cover completely the range of liquid diborane from 108° to 180°K, and also agree closely with the calculated values (20).

Heat Capacity, Molal Entropy, and Molal Heat Content

Values for the heat capacity of liquid diborane below the boiling point are summarized in Table 31 (2) and those obtained above the boiling point are summarized in Table 32 (14). By combining these data for the heat-capacity function (Figure 11), a smooth and valid curve is obtained, with excellent agreement at the overlapping temperature range of 170° to 180°K.

Molal entropy and heat content from Rifkin (14) are reproduced in Table 33. More general computerized thermal functions have been evaluated (5) which cover ranges not measurable because of the instability of gaseous diborane above -20°C.

No data were found on thermal conductivity of liquid diborane. This probably should be determined.

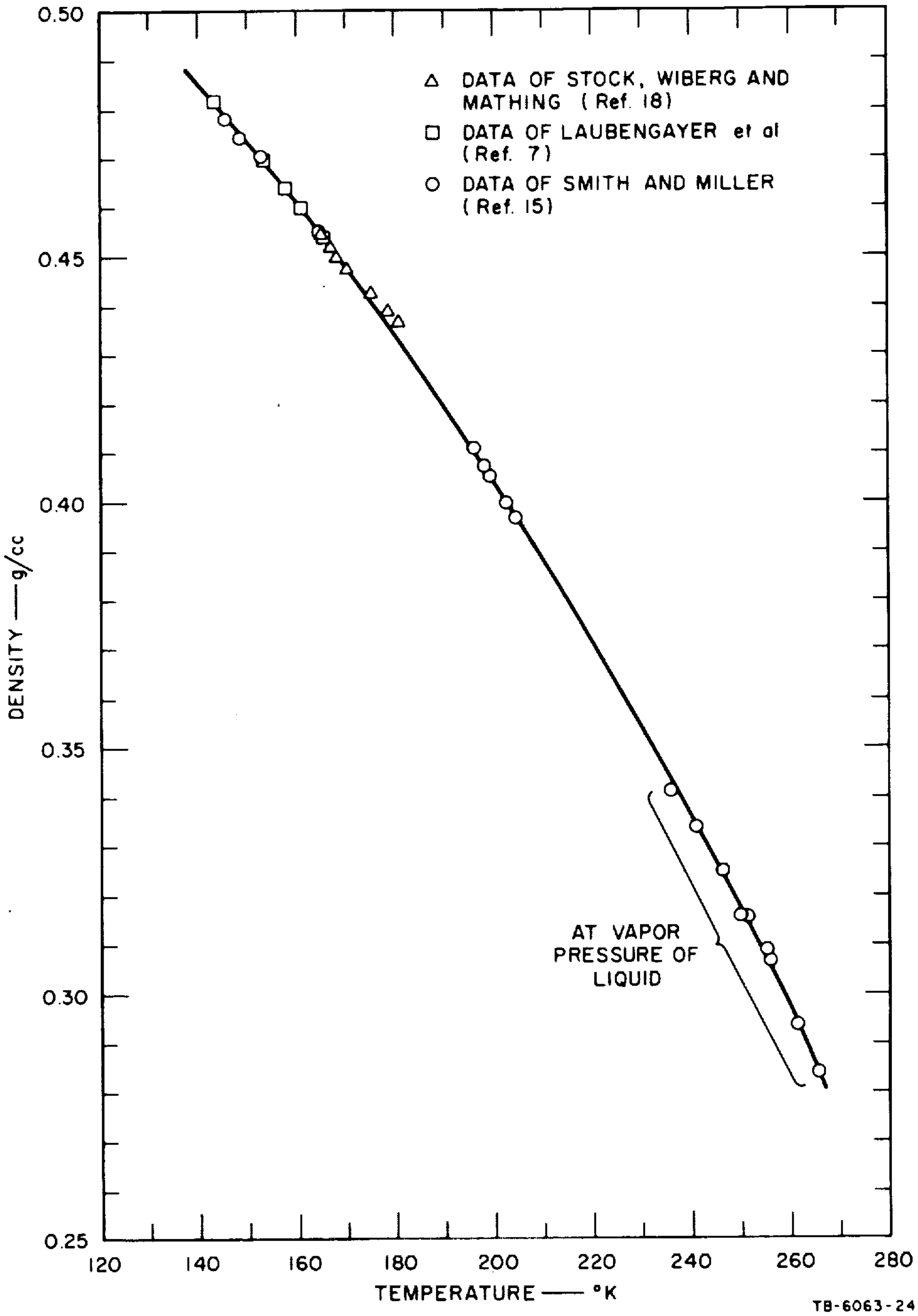

FIG. 4 DENSITY OF LIQUID B_2H_6

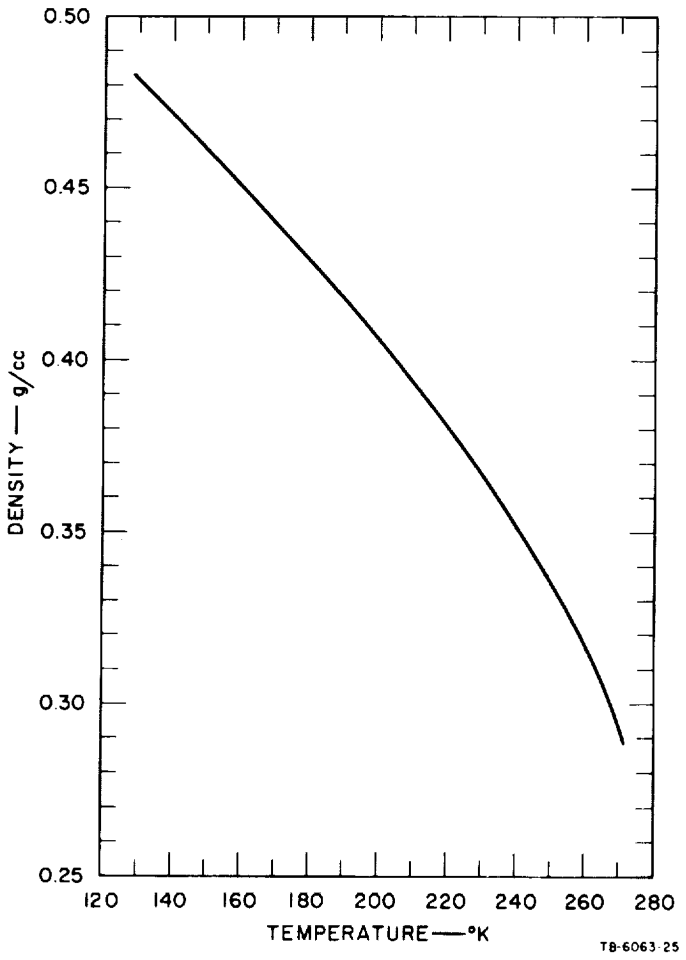

FIG. 5 COMPUTED DENSITY CURVE (Ref. 5, from data of Paridon and McWood, Ref. 11)

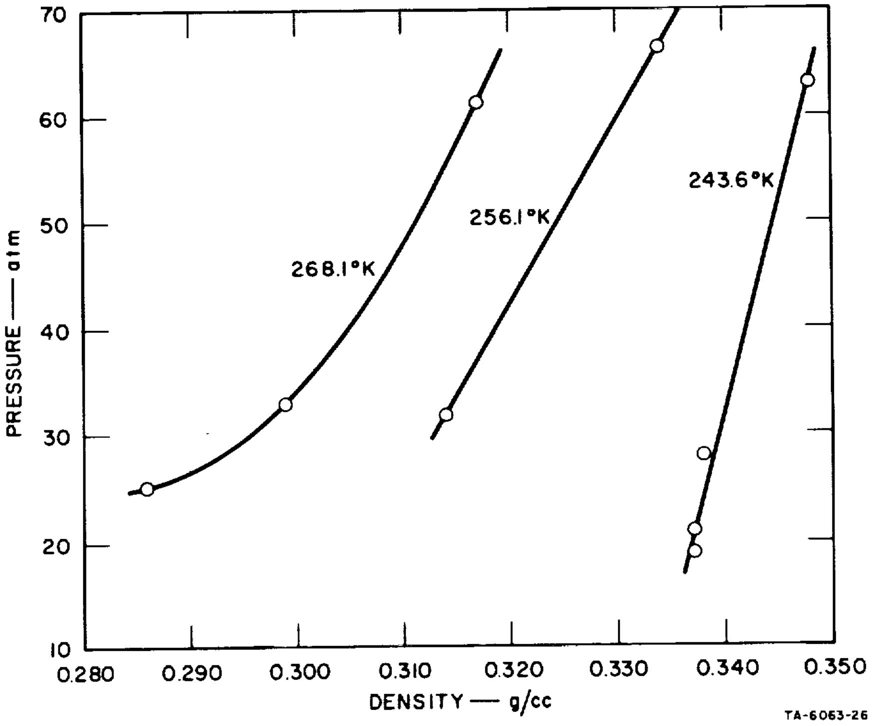

FIG. 6 LIQUID COMPRESSIBILITY ISOTHERMS OF B_2H_6 (Ref. 15)

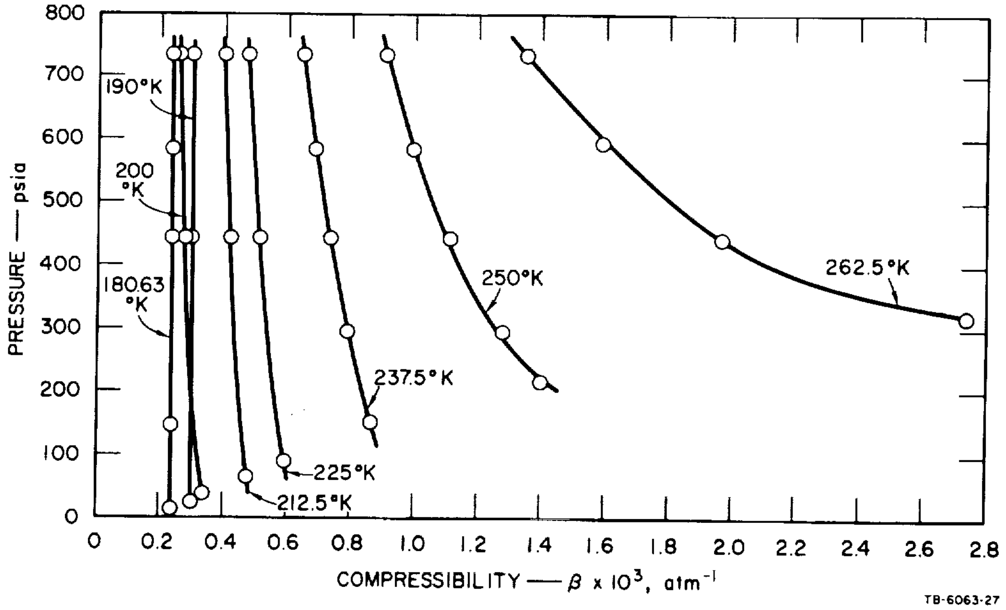

FIG. 7 COMPRESSIBILITY ISOTHERMS FOR LIQUID B_2H_6 (Ref. 11)

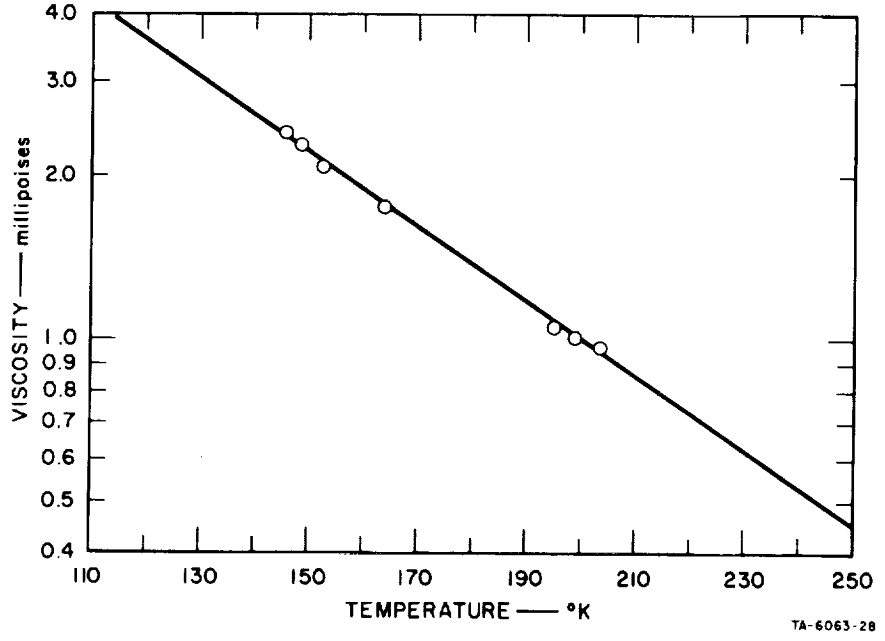

FIG. 8 VISCOSITY OF LIQUID B_2H_6

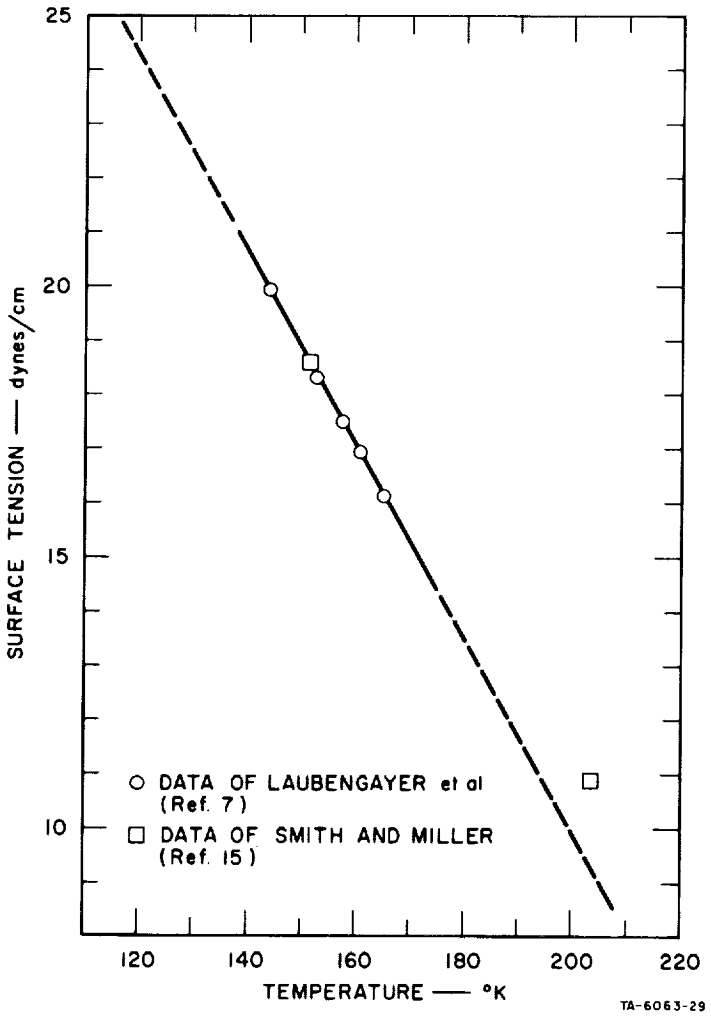

FIG. 9 SURFACE TENSION OF LIQUID B_2H_6

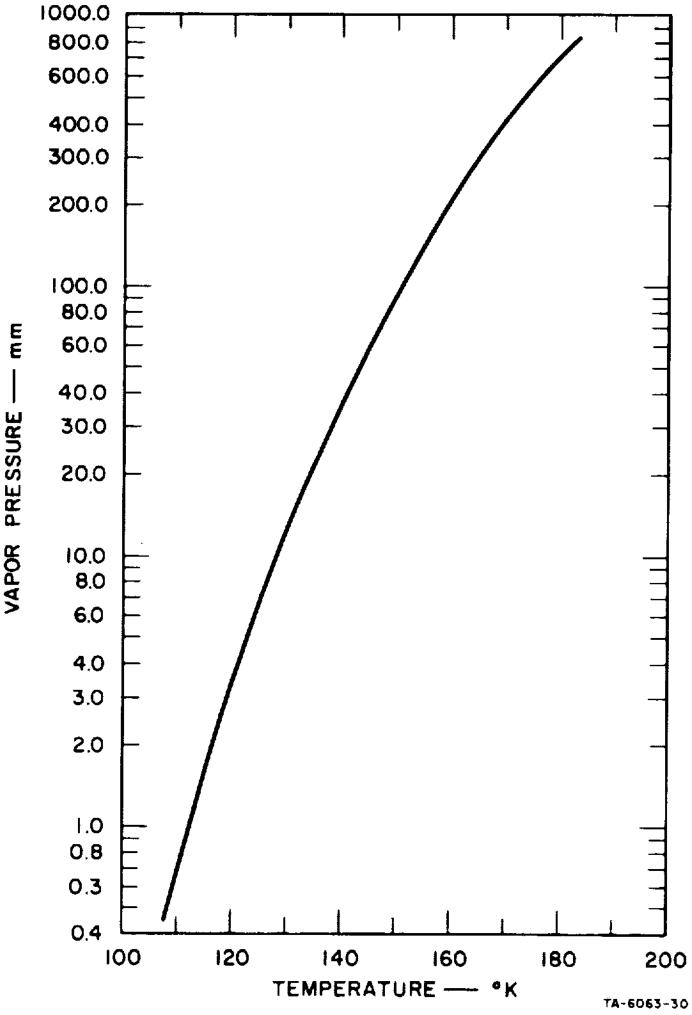

FIG. 10 VAPOR PRESSURE OF LIQUID B_2H_6 (Ref. 19)

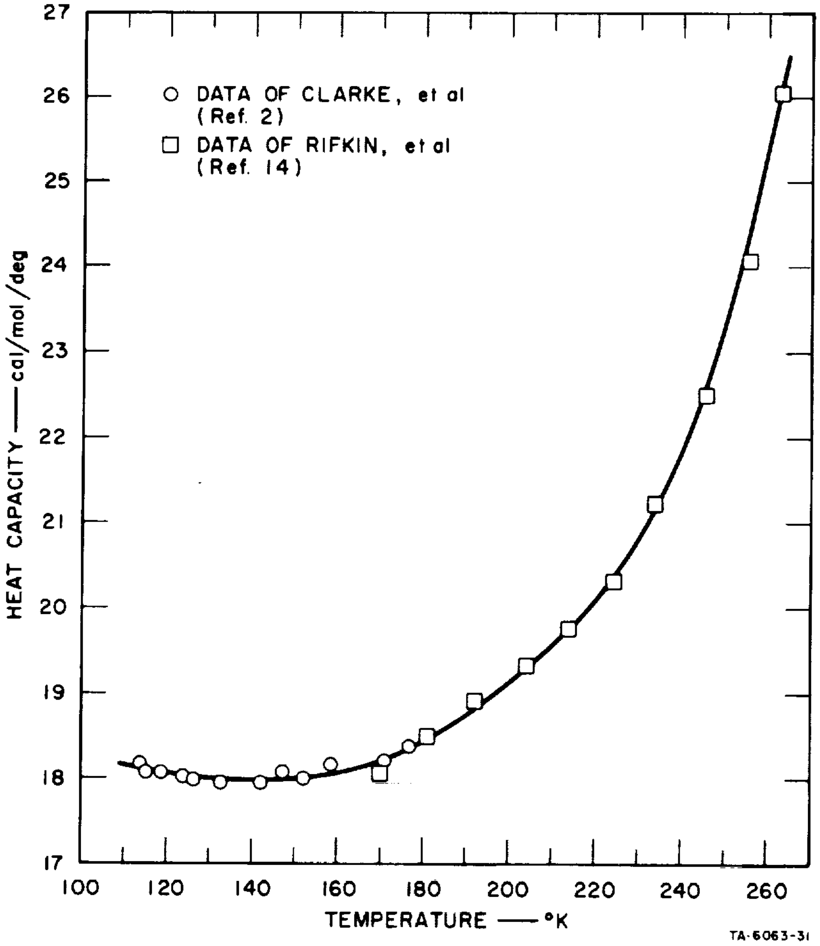

FIG. 11 HEAT CAPACITY OF LIQUID B_2H_6

Table 15

SUMMARY OF GENERAL PROPERTIES OF LIQUID DIBORANE

PROPERTIES	VALUES	REMARKS	REF.
Melting Point	-164.86°C, 108.30°K	experimental	(19)
	-164.86°C, 108.30°K	experimental	(2)
	-165.5°C, 107.7°K	experimental	(1,6,16,17)
Boiling Point	-92.84°C, 180.32°K	experimental	(2)
	-92.6°C, 180.6°K	experimental	(14)
	-92.57°C, 180.59°K	experimental	(10)
	-92.53°C, 180.63°K	experimental	(19)
	-92.5°C, 180.7°K	experimental	(1,6,16,17)
Heat of Vaporization	3.422 kcal/mole @ 180.32°K	experimental	(2)
	3.413 kcal/mole @ 180.63°K	calculated (vapor pressure)	(19)
	3.405 kcal/mole @ 180.60°K	calculated (vapor pressure)	(12)
	3.41 kcal/mole @ -92.5°C	--	(1)
Heat of Formation	6.73 kcal/mole @ 25°C	experimental	(13)
	7.53 kcal/mole @ 25°C	calculated	(1)
Critical Temperature	16.7°C, 289.9°K	experimental	(9)
Critical Pressure	581 psia, 39.5 atm.	experimental	(9)
Critical Volume	170 cc	estimated	(14)

Table 16

DENSITY OF LIQUID DIBORANE (Ref. 15)

TEMPERATURE		DENSITY, g/cc	TEMPERATURE		DENSITY, g/cc
°K	°C		°K	°C	
145.5	-127.7	0.4783	235.4	-37.8	0.341
149.6	-124.6	0.4747	240.3	-32.9	0.334
152.7	-120.5	0.4698	243.6	-29.6	0.333
163.9	-109.2	0.4552	246.1	-27.11	0.325
195.7	- 77.5	0.4110	249.9	-23.3	0.316
197.6	- 75.6	0.4073	251.2	-22.0	0.316
198.9	- 74.4	0.4055	255.3	-17.9	0.309
198.5	- 74.3	0.4052	255.9	-17.3	0.307
202.3	- 70.9	0.4001	260.8	-12.4	0.294
203.9	- 69.3	0.3976	265.8	- 7.4	0.284

Table 17

DENSITY OF LIQUID DIBORANE (Ref. 18)

TEMPERATURE		DENSITY, g/cc	TEMPERATURE		DENSITY, g/cc
°K	°C		°K	°C	
163.2	-110.0	0.4547	172.3	-100.9	0.4454
163.7	-109.5	0.4541	175.1	- 98.1	0.4425
165.3	-107.8	0.4525	176.5	- 96.7	0.4414
167.6	-105.6	0.4503	178.7	- 94.5	0.4393
170.5	-102.7	0.4472	180.6	- 92.6	0.4371

Table 18

DENSITY OF LIQUID DIBORANE (Ref. 7)

TEMPERATURE		DENSITY, g/cc	EQUATION
°K	°C		
143.7	-129.5	0.4818	$d = 0.3140 - 0.001296\ T^{\circ}C$
152.9	-120.3	0.4698	
157.6	-115.6	0.4640	
160.7	-112.5	0.4600	
165.0	-108.2	0.4542	

Table 19

DENSITY OF LIQUID DIBORANE (Ref. 5, 11)

TEMPERATURE		DENSITY, g/cc	TEMPERATURE		DENSITY, g/cc
°K	°C		°K	°C	
130	-143	0.4818	210	-63	0.3938
140	-133	0.4716	220	-53	0.3805
150	-123	0.4612	230	-43	0.3667
160	-113	0.4506	240	-33	0.3518
170	-103	0.4399	250	-23	0.3352
180	- 93	0.4288	260	-13	0.3161
190	- 83	0.4174	270	- 3	0.2926
200	- 73	0.4057			

Table 20

COMPRESSIBILITY OF LIQUID DIBORANE (Ref. 15)

TEMPERATURE		PRESSURE		DENSITY, g/cc
°K	°C	psia	atm	
243.6	-29.6	268	18.4	0.337
		309	21.0	0.337
		410	27.9	0.338
		927	63.1	0.348
250.0	-23.2	393	26.7	0.324
		900	61.2	0.332
256.1	-17.1	469	31.9	0.314
		977	66.4	0.334
268.1	- 5.1	370	25.2	0.286
		484	32.9	0.299
		902	61.3	0.317

Table 21

COMPRESSIBILITY OF LIQUID DIBORANE, β (Ref. 11)

TEMPERATURE		PRESSURE		$\beta \times 10^3$, atm^{-1}
°K	°C	atm	psia	
180.63	-92.53	50	734.80	0.233
		40	587.84	0.234
		30	440.88	0.234
		20	293.92	0.235
		10	146.96	0.235
		1.0000	14.696	0.236
190	-83	50	734.80	0.295
		40	587.84	0.296
		30	440.88	0.297
		20	293.92	0.297
		10	146.96	0.298
		1.6358	24.04	0.299
200	-73	50	734.80	0.255
		40	587.84	0.266
		30	440.88	0.280
		20	293.92	0.295
		10	146.96	0.313
		2.6084	38.33	0.329
212.5	-60.7	50	734.80	0.396
		40	587.84	0.410
		30	440.88	0.425
		20	293.92	0.442
		10	146.96	0.461
		4.3662	64.17	0.473
225	-48	50	734.80	0.471
		40	587.84	0.492
		30	440.88	0.516
		20	293.92	0.543
		10	146.96	0.575
		6.8601	100.82	0.586
237.5	-35.7	50	734.80	0.646
		40	587.84	0.686
		30	440.88	0.734
		20	293.92	0.792
		10.312	151.55	0.862
250	-23	50	734.80	0.905
		40	587.84	0.995
		30	440.88	1.11
		20	293.92	1.28
		14.891	218.84	1.40
262.5	-10.7	50	734.80	1.35
		40	587.84	1.59
		30	440.88	1.97
		20.795	305.60	2.74

Table 22

VISCOSITY OF LIQUID DIBORANE (Ref. 15)

TEMPERATURE		VISCOSITY, millipoises	TEMPERATURE		VISCOSITY, millipoises
°K	°C		°K	°C	
145.6	-127.6	2.45	198.8	-74.4	1.04
148.6	-124.6	2.31	198.9	-74.3	1.04
152.7	-120.5	2.10	198.9	-74.3	1.02
164.0	-109.2	1.77	202.3	-70.9	0.975
195.7	- 77.5	1.06	203.9	- 69.3	0.976
197.6	- 75.6	1.04			

$\eta = 16.4734(10^{-6})dht - 2591.8(10^{-6})dh/t$

η = viscosity, poises

d = density, g/cc

h = mean head, cm

t = time of flow, seconds

Table 23

SURFACE TENSION OF LIQUID DIBORANE (Ref. 7)

TEMPERATURE		SURFACE TENSION, dynes/cm^2
°K	°C	
143.7	-129.5	19.94
152.9	-120.3	18.32
157.6	-115.6	17.51
160.7	-112.5	16.95
165.0	-108.2	16.12

Table 24

SURFACE TENSION OF LIQUID DIBORANE (Ref. 15)

TEMPERATURE		SURFACE TENSION, dynes/cm^2
°K	°C	
151.6	-121.6	18.6
203.5	- 69.7	10.9

Table 25

VAPOR PRESSURE OF LIQUID DIBORANE (Ref. 19)

TEMPERATURE		VAPOR PRESSURE, mm	TEMPERATURE		VAPOR PRESSURE, mm
°K	°C		°K	°C	
108.22	-164.94	0.53	125.02	-148.14	6.76
108.37	-164.79	0.54	130.12	-143.14	12.74
108.76	-164.40	0.58	131.81	-141.35	15.66
113.07	-160.09	1.29	135.12	-138.04	22.39
115.10	-158.06	1.68	140.00	-133.16	37.38
117.89	-155.27	2.59	140.11	-132.89	37.55
119.90	-153.26	3.46	145.03	-128.13	60.04
124.40	-148.76	6.34	147.00	-126.16	71.99
$\log P_{mm} = 6.9681 - 674.82/(T - 15.02)$					
150.03	-123.13	93.28	170.01	-130.15	405.89
154.07	-119.09	130.49	172.90	-100.66	485.47
155.40	-177.16	141.40	175.04	- 98.12	552.05
160.00	-113.16	204.82	178.87	- 94.29	688.25
160.42	-112.74	211.62	179.94	- 97.22	732.04
165.14	-108.02	294.55	180.66	- 92.51	761.83
166.99	-106.26	331.01			
$\log P_{mm} = 6.61885 - 583.120/(T - 24.63)$					

Table 26

VAPOR PRESSURE OF LIQUID DIBORANE (Ref. 5)

TEMPERATURE		VAPOR PRESSURE, mm	TEMPERATURE		VAPOR PRESSURE, mm
°K	°C		°K	°C	
130	-143	12.707	160	-113	204.65
140	-133	37.392	170	-103	404.18
150	-123	93.358	180	- 98	733.32
			182	- 91	818.90

Table 27

VAPOR PRESSURE OF LIQUID DIBORANE (Ref. 18)

TEMPERATURE		VAPOR PRESSURE, mm	TEMPERATURE		VAPOR PRESSURE, mm
°K	°C		°K	°C	
163.0	-110.0	260	172.3	-100.9	472
163.7	-109.5	267	175.1	-98.1	557
165.4	-107.8	298	176.5	-96.7	599
167.6	-105.6	340	178.7	-94.5	674
170.5	-102.7	416	180.6	-92.6	755

Table 28

VAPOR PRESSURE OF LIQUID DIBORANE (Ref. 2)

TEMPERATURE		VAPOR PRESSURE, mm	EQUATION
°K	°C		
111.78	-161.38	1.16	$\log P_m = 8.1110 - \frac{870.95}{T} - 2.221 \times 10^{-3}T$
116.20	-156.96	2.31	
123.50	-149.66	6.11	
140.49	-132.67	39.23	
154.15	-119.01	131.01	
173.15	-100.01	490.61	
175.65	-95.51	569.67	

Table 29

VAPOR PRESSURE OF LIQUID DIBORANE (Ref. 11)

TEMPERATURE		VAPOR PRESSURE, mm	EQUATION
°K	°C		
151.23	-121.93	104.50	$\log P_{atm} = 3.8262 - \frac{598.30}{T} - \frac{1.6733 \times 10^4}{T^2}$
160.39	-112.77	211.81	
169.92	-103.24	404.70	
180.64	-92.52	761.90	

Table 30

VAPOR PRESSURE OF LIQUID DIBORANE (Ref. 3)

TEMPERATURE		VAPOR PRESSURE mm	TEMPERATURE		VAPOR PRESSURE mm
°K	°C		°K	°C	
118.2	-155.2	2.7	148.1	-125.1	80.6
121.0	-152.2	1.1	149.2	-124.0	89.2
124.3	-118.9	6.5	119.7	-123.5	92.8
127.0	-146.2	8.8	150.9	-122.3	102.4
128.8	-144.4	11.0	152.3	-120.9	115.1
130.0	-143.2	13.8	156.2	-117.0	156.5
131.4	-141.8	15.0	158.4	-114.8	185.6
132.0	-141.2	16.0	159.7	-113.5	203.6
133.6	-139.6	18.8	161.4	-111.8	232.5
135.2	-138.0	22.7	163.0	-110.2	259.3
136.7	-136.5	26.8	164.7	-108.5	292.0
138.3	-134.9	31.8	166.0	-107.2	319.3
139.7	-133.5	36.6	167.8	-105.4	357.3
139.8	-133.4	36.9	169.8	-103.4	402.8
141.3	-131.9	42.8	171.1	-102.1	438.8
142.0	-131.2	45.8	173.2	-100.0	497.0
142.7	-130.5	48.9	175.1	- 98.1	554.8
144.8	-128.4	59.6	177.1	- 96.1	622.3
145.2	-128.0	62.1	178.3	- 94.9	663.7
146.6	-126.6	70.8	178.5	- 94.3	670.2
147.0	-126.0	73.5	179.2	- 94.0	699.7
			180.8	- 92.4	760.3

$\log P_{mm} = -974.156/ -0.00653809T + 9.45290$

Table 31

HEAT CAPACITY OF LIQUID DIBORANE (Ref. 2)

TEMPERATURE		HEAT CAPACITY, cal/mole/deg	TEMPERATURE		HEAT CAPACITY, cal/mole/deg
°K	°C		°K	°C	
112.86	-160.30	18.18	142.45	-130.71	17.98
114.00	-159.16	18.18	146.62	-126.54	18.09
115.27	-157.89	18.08	152.08	-121.08	18.02
118.75	-154.41	18.08	158.61	-114.55	18.18
123.93	-149.13	18.02	165.42	-107.74	18.20
126.23	-146.93	18.01	166.23	-106.93	18.21
132.93	-140.23	17.97	170.95	-102.11	18.21
138.32	-134.84	18.01	176.72	-96.44	18.38

Table 32

HEAT CAPACITY OF SATURATED LIQUID DIBORANE (Ref. 14)

TEMPERATURE		HEAT CAPACITY, cal/mole/deg	TEMPERATURE		HEAT CAPACITY, cal/mole/deg
°K	°C		°K	°C	
170.09	-163.07	18.08	224.11	-49.05	20.33
180.86	-92.30	18.51	234.87	-38.29	21.23
192.00	-81.16	18.94	245.64	-27.52	22.52
203.36	-69.80	19.35	255.64	-17.52	24.09
213.63	-59.53	19.76	263.14	-10.02	26.06

Table 33

MOLAL ENTROPY AND MOLAL HEAT CONTENT
FOR LIQUID DIBORANE
UNDER ITS SATURATED VAPOR PRESSURE
(Ref. 14)

TEMPERATURE		$S - S_0$, cal/mole/deg	$H - H_0$, cal/mole
°K	°C		
180.68	-92.48	30.57	3115
190	-83	31.58	3290
200	-73	32.56	3482
210	-63	33.50	3678
220	-53	34.43	3880
230	-43	35.33	4089
240	-33	36.24	4313
250	-23	37.15	4538
260	-13	38.10	4788
270	-3	39.10	5066

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V. RECOMMENDATIONS AND SUMMARY

The review of available data given in Sections II and III suggests that re-determinations, extended determinations, or original determinations be made for several of the properties of liquid oxygen difluoride and liquid diborane in order to provide a sound basis for estimating the storage and performance paramenters of the propellant system. The following list indicates the lack of adequate data for the physical properties of interest:

Oxygen difluoride (OF_2)

(1) Density matrix (compressibility) between 14.6 and 800 psia at temperatures between 125° and 210°K.

(2) Surface tension between 50° and 125°K.

(3) Heat capacity between 50° and 125°K.

Diborane B_2H_6

(1) Compressibility at several check points, especially at very high and very low temperatures.

(2) Thermal conductivity.

A SUMMARY OF SELECTED VALUES FOR THE PROPERTIES OF OF_2 AND B_2H_6 IS GIVEN IN TABLES 34 AND 35, RESPECTIVELY.

Table 34

SELECTED VALUES FOR THE PROPERTIES OF LIQUID OXYGEN DIFLUORIDE

PROPERTY	VALUES OR EQUATIONS	REFERENCE
Melting Point	-223.8°C, 49.4°K	Table 1
Boiling Point	-145.3°C, 127.9°K	Table 1
Critical Temperature	-59.7°C, 213.5°K	Table 1
Critical Pressure	49.5 atm, 725 psia	Table 1
Critical Density	0.425 g/cc	Table 1
Critical Volume	127.0 cc/mole	Table 1
Heat of Vaporization	2.66 kcal/mole	Table 1
Heat of Formation	5.86 ± 0.03 kcal/mole	Table 1
Thermal Conductivity	$0.00058\ \mathrm{cal/sec/cm^2/°C/cm}$ at -195.8°C $0.0006\ \mathrm{cal/sec/cm^2/°C/cm}$ at -183°C	Table 1
Vapor Pressure	$\log P_{mm} = 7.2242 - \frac{555.42}{T°K}$	Table 4, Fig. 1
Viscosity	$\log \eta = \frac{112.4}{T°K} - 1.4508$	Table 11, Fig. 3
Density	$d_{liq} = 0.8225 - 0.004873T°C$	Table 9, Fig. 2

Table 35

SELECTED VALUES FOR THE PROPERTIES OF LIQUID DIBORANE

PROPERTY	VALUES OR EQUATIONS	REFERENCE
Melting Point	-164.86°C, 108.30°K	Table 15
Boiling Point	-92.53°C, 180.63°K	Table 15
Heat of Vaporization	3.413 kcal/mole @ 180.63°K	Table 15
Heat of Formation	6.73 kcal/mole @ 25°C	Table 15
Critical Temperature	16.7°C, 289.9°K	Table 15
Critical Volume	170 cc	Table 15
Density	(145°K to 260°K)	Table 16, Fig. 4
Compressibility	(180°K to 260°K)	Table 21, Fig. 7
Surface Tension	(143°K to 165°K)	Table 23, Fig. 9
Viscosity	$\eta = 16.4734\ (10^{-6})\ dhT - 2591.8\ (10^{-6})\ dh/T$	Table 22, Fig. 8
Vapor Pressure	$\log P_{mm} = 6.9681 - 674.82/T - 15.02$ at 108° - 147°K $\log P_{mm} = 6.61885 - 583.120/T - 24.63$ at 150° - 180°K	Table 26, Fig. 10
Heat Capacity	(112° to 176°K)	Table 32, Fig. 11
	(170° to 263°K)	Table 33, Fig. 11