

# Heat Capacity, Heats of Fusion and Vaporization, and Vapor Pressure of Decaborane ( $B_{10}H_{14}$ )

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Measurements of the heat capacity of decaborane ( $B_{10}H_{14}$ ) were made from 55° to 380° K by means of an adiabatic calorimeter. The data were used to obtain a table of smoothed values of heat capacity, enthalpy, entropy, and Gibbs free energy from 60° to 380° K. The heat of fusion, the triple-point temperature, the heat of vaporization at 378° K (23.96 mm Hg), and their corresponding estimated uncertainties were determined to be, respectively,  $21,965 \pm 40$  abs j mole<sup>-1</sup>,  $371.93 \pm 0.02^\circ$  K, and  $50,759 \pm 100$  abs j mole<sup>-1</sup>. The vapor pressure was measured from 345° to 395° K by means of an isoteniscope and the results above the triple-point temperature were found to be representable by the equation:

$$\log_{10} p_{\text{mmHg}} = -4225.345/T - 0.0107975T + 16.63911.$$

The entropy of decaborane in the ideal gas state at 378° K and 1-atmosphere pressure was computed from the data to be 402.18 abs j deg<sup>-1</sup> mole<sup>-1</sup> (96.12 cal deg<sup>-1</sup> mole<sup>-1</sup>) with an estimated uncertainty of  $\pm 0.87$  abs j deg<sup>-1</sup> mole<sup>-1</sup>.

## 1. Introduction

Kerr et al. [1]<sup>1</sup> have reported heat-capacity values of decaborane ( $B_{10}H_{14}$ ) from 14° to 305° K. The present paper contains results of the measurements of the heat capacity from 55° to 380° K, of the heats of fusion and vaporization, and of the vapor pressure from 345° to 395° K. The data were used to obtain a table of smoothed values of heat capacity, enthalpy, entropy, and Gibbs free energy of solid and liquid decaborane from 60° to 380° K. The entropy of decaborane in the ideal gas state at 378° K and 1-atmosphere pressure was computed from the data.

## 2. Apparatus and Method

Measurements of the heat capacity and heat of fusion were made in an adiabatic calorimeter, similar in design to that described by Southard and Brickwedde [2]. The details of the design and operation of the calorimeter and the procedures used in the analysis of the data have been given previously [3, 4].

The heat-of-vaporization measurements were made in another adiabatic calorimeter of a design similar to those described by Osborne and Ginnings [5] and by Aston et al. [6], in which, while electric energy is added continuously, the vapor is removed isothermally by controlling a throttle valve. Details of the calorimetric apparatus and procedures are given in the references cited [5, 6].

The vapor-pressure measurements were made by means of an isoteniscope contained in an oil bath the temperature of which was controlled to  $\pm 0.001^\circ$  C. All pressure readings were made to 0.01 mm Hg by means of a cathetometer and were converted to

standard mm Hg ( $g = 980.665$  cm sec<sup>-2</sup>, temperature = 0° C) on the basis that the acceleration due to the local gravity is 980.076 cm sec<sup>-2</sup>.

The temperatures were measured by means of platinum-resistance thermometers and a high-precision Mueller bridge. The platinum-resistance thermometers were calibrated above 90° K, in accordance with the 1948 International Temperature Scale [7], and between 10° and 90° K with a provisional scale [8], which consists of a set of platinum-resistance thermometers calibrated against a helium-gas thermometer. The provisional scale, as used in the calibration of the thermometers, was based upon the value 273.16° K for the temperature of the ice point and 90.19° K for the temperature of the oxygen point. Above 90° K, the temperatures in degrees Kelvin (° K) were obtained by adding 273.16 deg to the temperatures in degrees Celsius (° C).

## 3. Sample

A sample of decaborane was purified by two sublimations in a high vacuum apparatus. The material was collected in a trap held at dry-ice temperature while the apparatus was pumped continuously. The sample was not exposed to the atmosphere at any time following this purification. For the heat-capacity measurements the purified sample was transferred into the calorimeter container in the molten state under vacuum. The purity of this material was determined from its equilibrium melting temperatures in the manner previously described [4]. The results of the measurements are summarized in table 1.

For the heat-of-vaporization measurements the purified sample of decaborane was transferred into the calorimeter by two procedures. Part of the

<sup>1</sup> Figures in brackets indicate the literature references at the end of this paper.

sample was transferred in the molten state by a method similar to that used for the heat-capacity experiments. The remainder was poured as a saturated benzene solution into the calorimeter. Most of the benzene was removed from the sample by cooling the calorimeter and its contents to 0° C and pumping at high vacuum. The decaborane which remained in the calorimeter was subsequently melted, slowly crystallized, and pumped at about 50° C three times. The high precision obtained in the heat-of-vaporization measurements (see section 6) indicates that very little benzene, if any, remained in the sample.

TABLE 1. *Equilibrium melting temperatures of decaborane*

$$N_2 = 0.0191 \Delta T^a, \text{ } ^\circ\text{K} = 273.16^\circ + ^\circ\text{C}$$

$1/F^b$	$T_{\text{obs}}$
	$^\circ\text{K}$
13.58	<sup>c</sup> 371.8157
8.81	371.8556
6.49	371.8721
5.13	371.8844
3.90	371.8987
2.64	371.9114
1.99	371.9133
1.60	371.9146
1.16	371.9159
0.00	<sup>d</sup> 371.93

Triple-point temperature, 371.93° K  
with an estimated uncertainty  
of  $\pm 0.02^\circ\text{K}$ .<sup>e</sup>  
Impurity, 0.016 mole percent  
with an estimated uncertainty  
 $\pm 0.004$  mole percent.<sup>e</sup>

<sup>a</sup>  $N_2$  is the mole fraction impurity;  $\Delta T = T_{\text{triple point}} - T_{\text{obs}}$ .

<sup>b</sup>  $F$  is the fraction of sample melted.

<sup>c</sup> The temperatures given are believed accurate within  $\pm 0.01^\circ\text{K}$ . Wherever temperatures are given to the fourth decimal, the last two figures are significant only insofar as small temperature differences are concerned.

<sup>d</sup> This temperature, taken to be the triple-point temperature of pure decaborane, was obtained by the extrapolation of a linear equation fitted to the data by the method of least squares.

<sup>e</sup> The uncertainty was estimated by examining the imprecision of the measurements and all known sources of systematic error. The system was assumed to follow the ideal solution law and to form no solid solution with the impurities.

## 4. Heat Capacity

The heat-capacity experiments were made on a 15.8193-g sample from about 55° to 380° K. The observed values of heat capacity are summarized in table 2 and shown graphically in figure 1. The recently published values of Kerr et al. [1], in the range from 14° to 305° K, are also plotted for comparison. The region below 100° K is plotted on a larger scale to show more clearly how the two measurements compare in the region from 50° to 100° K. The results of the present measurements are generally somewhat lower. With the exception of the range from 50° to 100° K, where the present results are lower by about 2 percent, the maximum difference between the two results is about 1 percent (around 175° to 200° K).

Empirical equations, which approximately fit the experimental data, and large scale deviation curves were used to obtain the smoothed values of heat capacity given in table 3. The uncertainty in the values below 350° K was estimated to be  $\pm 0.2$  percent and above this temperature  $\pm 0.3$  percent. A larger uncertainty was assigned to the results at the higher temperature because of the probable inaccuracies in the values of the density used in making vaporization corrections, and because of the greater difficulties encountered in the operation of the calorimeter at its upper temperature limit. The uncertainties given were estimated by considering the imprecision of the measurements and all known sources of systematic error. In the region immediately below the triple-point temperature, greater tolerances than the value given must be allowed because of the inaccuracies in the premelting corrections which were based on the ideal solution law and liquid soluble-solid insoluble impurities.

TABLE 2. *Observed heat capacity of decaborane*

$$\text{Molecular weight} = 122.312, \text{ } ^\circ\text{K} = 273.16^\circ + ^\circ\text{C}$$

$T_m^a$	$C_{\text{satd.}}^b$	$\Delta T$
RUN 1		
$^\circ\text{K}$	$\text{abs } j \text{ deg}^{-1}$ $\text{mole}^{-1}$	$^\circ\text{K}$
<sup>c</sup> 57.1400	38.038	<sup>c</sup> 3.6854
60.6736	40.328	3.3818
63.9485	41.295	3.1680
67.0275	42.580	2.9900
69.9471	43.523	2.8492
72.7351	44.784	2.7267
75.4094	45.837	2.6220
RUN 2		
255.2526	176.83	7.6981
264.1192	185.36	10.0353
274.0325	195.33	9.7913
283.7156	204.21	9.5747
293.1851	213.49	9.3643
303.0575	222.68	10.3805
313.3276	232.52	10.1597
323.3792	242.14	9.9436
333.2167	252.03	9.7314
343.8138	262.79	11.4627
355.1360	275.12	11.1818
RUN 3		
297.9518	217.65	6.9681
307.2019	226.61	11.5321
317.9793	236.71	10.0228
327.9017	246.35	9.8220
337.6386	256.02	9.6517
348.1243	266.95	11.3198
359.2964	278.10	11.0243
RUN 4		
249.2684	171.14	10.4227
259.5611	180.67	10.1627
268.6188	189.89	7.9528
273.5818	195.84	1.9731
359.7636	279.76	8.1019
365.4041	286.81	3.1791
368.5344	291.33	3.0815
373.0461	308.38	1.3738
374.4134	315.06	1.3607
375.7728	316.04	1.3582

TABLE 2. Observed heat capacity of decaborane—Continued

Molecular weight = 122.312, °K = 273.16° + °C

$T_m^a$	$C_{\text{satd.}}^b$	$\Delta T$
RUN 5		
°K	abs j deg <sup>-1</sup> mole <sup>-1</sup>	°K
365.1724	286.11	4.8000
369.0933	291.13	3.0418
373.8486	312.90	3.0770
376.9166	317.64	3.0590
379.9692	317.91	3.0462
RUN 6		
85.1410	49.779	7.3043
92.1968	52.387	6.8073
98.8188	55.154	6.4367
106.1033	58.043	8.1324
114.7995	62.389	9.2600
123.8154	67.164	8.7717
132.4032	72.073	8.4040
140.6468	77.130	8.0832
148.5910	82.406	7.8052
157.5087	88.823	10.0301
167.3644	96.326	9.6813
RUN 7		
89.9401	51.444	10.7465
99.8907	55.595	9.1547
108.7621	59.439	8.5881
117.1264	63.197	8.1405
125.0860	67.886	7.7788
132.5417	72.184	7.4834
139.8954	76.787	7.2240
147.0065	81.439	6.9981
153.9032	86.312	6.7953
160.6105	91.307	6.6194
169.6244	98.285	11.4083
180.8098	107.21	10.9625
RUN 8		
181.4863	107.98	10.8747
192.1696	117.27	10.4919
202.4977	126.60	10.1644
212.5120	135.93	9.8642
222.2401	145.05	9.5920
231.7077	154.16	9.3431
240.4659	162.60	9.1235
249.4843	171.35	8.9133
258.3025	179.71	8.7231
266.9307	188.39	8.5333

<sup>a</sup>  $T_m$  is the mean temperature of the heating interval  $\Delta T$ .<sup>b</sup>  $C_{\text{satd.}}$  is the heat capacity along the saturation-pressure curve.<sup>c</sup> The temperatures given are believed accurate within  $\pm 0.01^\circ\text{K}$ . Wherever temperatures are given to the fourth decimal, the last two figures are significant only insofar as small temperature differences are concerned.

TABLE 3. Molal heat capacity, enthalpy, entropy, and Gibbs free energy of decaborane

Molecular weight = 122.312, °K = 273.16° + °C

$T$	$C_{\text{satd.}}^a$	$(H_T - H_0^\circ\text{K})^a$	$(S_T - S_0^\circ\text{K})^a$	$-(F_T - H_0^\circ\text{K})^a$
Crystal				
°K	abs j deg <sup>-1</sup> mole <sup>-1</sup>	abs j mole <sup>-1</sup>	abs j deg <sup>-1</sup> mole <sup>-1</sup>	abs j mole <sup>-1</sup>
60	39.993	1109.7	29.201	642.33
65	41.768	1314.0	32.471	796.56
70	43.703	1527.7	35.637	966.87
75	45.687	1751.1	38.719	1152.8
80	47.686	1984.6	41.732	1353.9
85	49.671	2228.0	44.682	1570.0
90	51.597	2481.2	47.576	1800.7
95	53.544	2744.0	50.418	2045.7
100	55.582	3016.8	53.215	2304.8
105	57.698	3299.9	55.978	2577.8

TABLE 3. Molal heat capacity, enthalpy, entropy, and Gibbs free energy of decaborane—Continued

Molecular weight = 122.312, °K = 273.16° + °C

$T$	$C_{\text{satd.}}^a$	$(H_T - H_0^\circ\text{K})^a$	$(S_T - S_0^\circ\text{K})^a$	$-(F_T - H_0^\circ\text{K})^a$
Crystal—Continued				
°K	abs j deg <sup>-1</sup> mole <sup>-1</sup>	abs j mole <sup>-1</sup>	abs j deg <sup>-1</sup> mole <sup>-1</sup>	abs j mole <sup>-1</sup>
110	59.924	3593.9	58.713	2864.5
115	62.348	3899.5	61.430	3164.9
120	64.945	4217.7	64.137	3478.8
125	67.688	4549.2	66.844	3806.2
130	70.574	4894.8	69.554	4147.2
135	73.595	5255.2	72.274	4501.8
140	76.745	5631.0	75.007	4870.0
145	80.021	6022.8	77.757	5251.9
150	83.414	6431.4	80.526	5647.6
155	86.919	6857.2	83.318	6057.2
160	90.538	7300.8	86.135	6480.8
165	94.271	7762.7	88.978	6918.6
170	98.086	8243.6	91.848	7370.6
175	101.98	8743.7	94.748	7837.1
180	106.01	9263.7	97.677	8318.2
185	110.17	9804.1	100.64	8813.9
190	114.46	10366	103.63	9324.6
195	118.87	10949	106.66	9850.3
200	123.39	11554	109.73	10391
205	128.04	12183	112.83	10948
210	132.74	12835	115.97	11520
215	137.45	13510	119.15	12107
220	142.21	14210	122.37	12711
225	147.00	14933	125.62	13331
230	151.83	15680	128.90	13967
235	156.70	16451	132.22	14620
240	161.61	17247	135.57	15290
245	166.54	18067	138.95	15976
250	171.47	18912	142.37	16679
255	176.38	19782	145.81	17400
260	181.22	20676	149.28	18137
265	186.07	21594	152.78	18893
270	190.90	22536	156.30	19665
273.16	193.95	23144	158.54	20163
275	195.73	23503	159.85	20456
280	200.56	24494	163.42	21264
285	205.37	25508	167.01	22090
290	210.15	26547	170.62	22934
295	214.94	27610	174.26	23796
298.16	217.96	28294	176.57	24351
300	219.71	28697	177.91	24677
305	224.48	29807	181.58	25575
310	229.23	30941	185.27	26492
315	233.98	32099	188.98	27428
320	238.76	33281	192.70	28382
325	243.62	34487	196.44	29355
330	248.60	35718	200.19	30347
335	253.65	36973	203.97	31357
340	258.76	38254	207.77	32386
345	263.91	39561	211.58	33435
350	269.14	40894	215.42	34502
355	274.46	42252	219.27	35589
360	280.04	43639	223.15	36695
365	286.22	45054	227.05	37820
370	292.49	46502	230.99	38966
371	292.88	46795	231.78	39196
371.93	293.49	47068	232.52	39415
Liquid				
371.93	296.73	69033	291.58	39415
372	297.27	69053	291.64	39435
373	308.32	69356	292.45	39727
374	313.11	69667	293.28	40020
375	315.14	69981	294.12	40313
376	316.46	70297	294.96	40608
377	317.31	70614	295.80	40903
378	317.82	70932	296.64	41200
379	317.98	71250	297.48	41497
380	317.99	71568	298.32	41794

<sup>a</sup> These quantities are at saturation pressures [4].

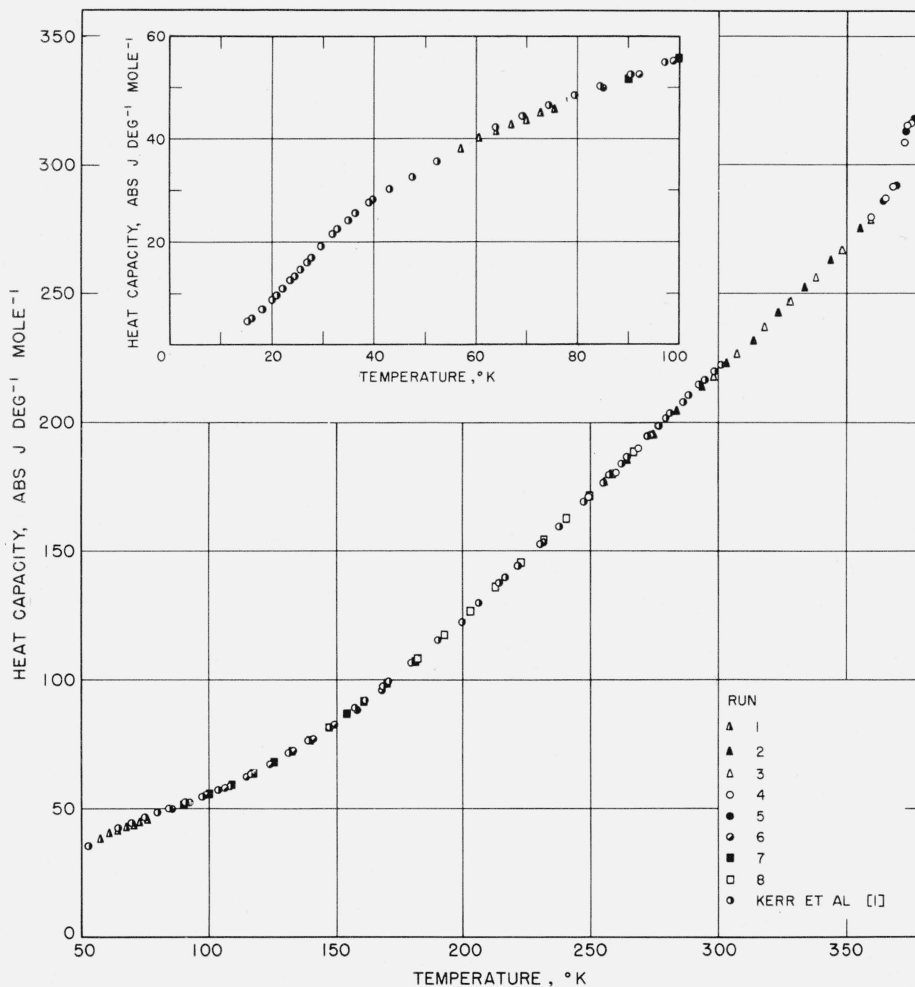


FIGURE 1. Observed heat capacity of decaborane and comparison with other measurements.

TABLE 4. Molal heat of fusion of decaborane

Molecular weight=122.312; mass of sample=15.8193g;  
triple-point temperature=371.93° K; ° K=273.16°+° C

Temperature interval	Heat input	Heat capacity and premelting corrections	$\Delta H$	$\Delta H_f$
° K	abs j	abs j	abs j	abs j mole <sup>-1</sup>
364.8085 to 376.7299	3861.2	1018.2	2843.0	21981
370.0751 to 372.3592	3021.3	180.9	2840.4	21962
370.6142 to 372.3101	2965.0	125.7	2839.3	21953
Mean				21965
Standard deviation of the mean <sup>a</sup>				8
Estimated uncertainty of the mean <sup>b</sup>				±40

<sup>a</sup> Standard deviation of the mean, as used here and in table 5, is defined as  $[(\sum d^2)/n(n-1)]^{1/2}$ , where  $d$  is the difference between a single observation and the mean, and  $n$  is the number of observations.

<sup>b</sup> See footnote e, table 1.

## 5. Heat of Fusion

The heat of fusion was determined by supplying electric energy continuously from a temperature just below the triple-point temperature (371.93° K) to

just above it, and by correcting for the heat capacity of the sample and container and for premelting that results from the presence of impurities. The results of the measurements are summarized in table 4. The values given in the fourth column are the total heats required to melt the 15.8193-g sample in the calorimeter. Upon consideration of the precision obtained, the procedures by which the various corrections were applied to the total energy input, the purity of the sample, and other known possible sources of systematic error, the uncertainty in the value obtained for the heat of fusion (21,965 abs j mole<sup>-1</sup>) is considered to be  $\pm 40$  abs j mole<sup>-1</sup>.<sup>2</sup>

## 6. Heat of Vaporization

Measurements of the heat of vaporization were made at 378° K and 23.96 mm Hg pressure. The molal heat of vaporization,  $L_v$ , is related to the experimentally observed quantities  $Q$  and  $m$  by:

$$L_v = QM/m - TVdp/dt, \quad (1)$$

<sup>2</sup> See footnote e, table 1.

where  $Q$  is the energy input necessary to vaporize the  $m$  grams of material collected,  $M$  the molecular weight,  $T$  the absolute temperature of vaporization,  $V$  the molal volume of liquid decaborane, and  $p$  the vapor pressure. The molal volume was taken to be that value obtained from the density of the liquid at 100° C as reported by Stock and Pohland [9]. The vapor pressure used in the calculation was taken from the results of the measurements reported in this paper. The results of the heat-of-vaporization measurements and calculations are summarized in table 5. Considering the imprecision of the measurements and various known possible sources of systematic error, the molal heat of vaporization obtained (50,759 abs j mole<sup>-1</sup>) was estimated to be uncertain by as much as ±100 abs j mole<sup>-1</sup>. The heat of vaporization calculated from the Clausius-Clapeyron equation amounted to 51,800 abs j mole<sup>-1</sup>.

## 7. Vapor Pressure

The vapor-pressure measurements were made from 345° to 395° K. The results above the triple-point temperature can be represented by the following equation:

$$\log_{10} p_{\text{mm Hg}} = -4225.345/T - 0.0107975T \quad (2)$$

$$+ 16.63911.$$

The constants were obtained by the method of least squares. In table 6 are given the observed vapor pressures, the values calculated from eq 2, and their deviation. Stock and Pohland [9] reported the following equation for the vapor pressure of decaborane:

$$\log_{10} p_{\text{mm Hg}} = -3218.5/T - 1.75 \log_{10} T - \quad (3)$$

$$0.005372 + 7.4106.$$

In column 5 are given for comparison the values of the vapor pressure obtained from eq 3. The agreement is fairly good.

## 8. Derived Thermal Properties

As described earlier, the experimental heat-capacity data were used to obtain smoothed values of heat capacity from 60° to 380° K. The values of enthalpy, entropy, and Gibbs free energy given in table 3 were obtained by numerical integration [4] of the smoothed values of heat capacity, using 4-point Lagrangian integration coefficients [10]. The starting values at 60° K were obtained by interpolating in the table of thermal functions given by Kerr et al. [1].

The computation to obtain the entropy of decaborane in the ideal-gas state at 378° K and 1-atm pressure is summarized in table 7. The correction for gas imperfection has not been applied because of the lack of necessary data. However, the correction is believed to be smaller than the uncertainty in the value of the entropy. The value of the entropy of decaborane in the ideal-gas state at 378° K and 1-atm

pressure in calories (1 cal=4.1840 abs j) becomes 96.12 cal deg<sup>-1</sup> mole<sup>-1</sup> with an estimated uncertainty of ±0.21 cal deg<sup>-1</sup> mole<sup>-1</sup>.

TABLE 5. Molal heat of vaporization of decaborane at 378° K  
Pressure=23.96 mm Hg; molecular weight=122.312; °K=273.16°±°C

$QM/m$	$TV \frac{dp}{dT}$	$L_v$
abs j mole <sup>-1</sup>	abs j mole <sup>-1</sup>	abs j mole <sup>-1</sup>
50753	8	50745
50765	8	50748
50781	8	50773
50777	8	50769
Mean.....		50759
Standard deviation of the mean <sup>a</sup> .....		7
Estimated uncertainty of the mean <sup>b</sup> .....		±100

<sup>a</sup> See footnote a, table 4.

<sup>b</sup> The uncertainty was estimated by examining the imprecision of the measurements, and all known sources of systematic error.

TABLE 6. Vapor pressure of decaborane  
°K=273.16°±°C

$T$ , obs	$p$ , obs	$p$ , calc	$\Delta p$ , obs-calc	$p$ , S and $P^a$
Series I (solid)				
° K	mm Hg	mm Hg	mm Hg	mm Hg
345.45	<sup>b</sup> 3.66	-----	-----	-----
355.12	6.05	-----	-----	-----
361.81	10.08	-----	-----	-----
371.53	18.23	-----	-----	-----
Series II (liquid)				
384.91	32.02	32.03	-0.01	32.03
384.90	31.95	32.01	-0.06	32.02
390.08	39.45	39.37	+0.08	39.70
390.08	39.28	39.37	-0.09	39.70
395.08	47.61	47.68	-0.07	48.53
395.06	47.73	47.64	+0.09	48.49
Series III (liquid)				
372.11	18.45	18.46	-0.01	18.24
371.92	18.29	18.30	-0.01	18.08
371.92	18.21	18.30	-0.09	18.08
375.17	21.20	21.17	+0.03	20.96
375.17	21.16	21.17	-0.01	20.96
378.18	24.10	24.15	-0.05	23.96
378.20	24.18	24.17	+0.01	23.98
378.20	24.37	24.17	+0.20	23.98
381.31	27.54	27.59	-0.05	27.47
381.30	27.62	27.58	+0.04	27.46
381.25	27.53	27.52	+0.01	27.40
381.24	27.56	27.51	+0.05	27.38
381.24	27.56	27.51	+0.05	27.38

<sup>a</sup> See reference [9].

<sup>b</sup> The values are listed according to the chronological order in which they were observed.

TABLE 7. Molal entropy of decaborane at 378° K  
Molecular weight=122.312, °K=273.16°±°C

$S$ , solid at 371.93°K.....	abs j deg <sup>-1</sup> mole <sup>-1</sup> 232.52±0.48
$\Delta S$ , fusion, 21,965/371.93.....	59.06±0.11
$\Delta S$ , liquid, 371.93° to 378°K.....	5.06±0.02
$\Delta S$ , vaporization at 378°K, 50,759/378.....	134.28±0.26
$\Delta S$ , compression, $R \ln 23.96/760$ .....	-28.74
$S$ , ideal gas at 378°K and 1 atm.....	402.18±0.87 <sup>a</sup>

<sup>a</sup> The over-all uncertainty was obtained by summing the absolute values of the various uncertainties listed.

## 9. References

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