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3712

HEAT CAPACITY, HEATS OF FUSION AND VAPORIZATION, AND VAPOR PRESSURE OF TRIETHYLBORANE, $(C_2H_5)_3B$

by

George T. Furukawa

Thermodynamics Section
Heat and Power Division

February 1, 1955

U. S. DEPARTMENT OF COMMERCE
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NBS PROJECT | **NBS REPORT**

0302-10-2677 | 3712

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FOREWORD

This report is one of a series on the thermodynamic investigation of boron-containing compounds. The information should aid in the better understanding of these relatively unstable compounds and in the development of technical and scientific applications.

Chas W Beckett

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F. G. Brickwedde, Chief
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HEAT CAPACITY, HEATS OF FUSION AND VAPORIZATION, AND VAPOR PRESSURE OF TRIETHYLBORANE, $(C_2H_5)_3B$

by

George T. Furukawa

Abstract

The heat capacity of triethylborane, $(C_2H_5)_3B$, was determined from 15° to 300°K, and the data were used to construct a table of smoothed heat capacity, enthalpy, entropy, and Gibbs free energy from 0° to 300°K. The heat of fusion was found to be 11,854±100 abs j $mole^{-1}$ and the triple-point temperature 180.3±0.5°K. The heat-of-vaporization experiments at 300°K (56.27 mm Hg) yielded 36,710±36 abs j $mole^{-1}$. The results of the vapor-pressure measurements from 275° to 365°K can be represented by

$$\log_{10} p_{mmHg} = -\frac{2837.8990}{T} - 7.0393186 \log_{10} T + 28.64715.$$

The entropy of the ideal-gas triethylborane at 300°K and 1 atm was computed from the results of the measurements to be 439.1±0.8 abs j deg^{-1} $mole^{-1}$ (104.9±0.2 cal deg^{-1} $mole^{-1}$).

INTRODUCTION

As a part of the program to obtain thermodynamic data on boron-containing compounds, heat-capacity measurements were made on triethylborane, $(C_2H_5)_3B$, from about 15° to 300°K. Heats of fusion and vaporization were also determined. The vapor pressure of the substance was measured from about 275° to 365°K. The data were used to obtain a smoothed table of heat capacity, enthalpy, entropy, and Gibbs free energy from 0° to 300°K and to compute the entropy of the substance in the ideal-gas state at 1 atm pressure and 300°K.

APPARATUS AND METHOD

The heat capacity and the heat of fusion were measured in an adiabatic calorimeter similar to that described by R. B. Scott, et al.

[1][1]. The operation of the calorimeter was similar also to that outlined in this reference. The treatment of the observed data was described earlier [2], which included curvature and vaporization corrections wherever applicable. The heat of vaporization was determined in another adiabatic calorimeter of a design similar to those described by Osborne and Ginnings [3] and by Aston, et al. [4]. The vapor was removed isothermally by controlling a throttle valve while electrical energy was added continuously. The temperature of the vapor was sensed by means of a nickel thermometer wound on the tube between the throttle valve and the calorimeter sample container. The material was collected by condensation and weighed.

Vapor pressures were measured by means of an isoteniscope. All pressure readings were made to 0.01 mm Hg using a Wild cathetometer and were converted to standard mm Hg (g = 980.665 cm sec^{-2}, temperature = 0°C) on the basis that the local gravity is 980.076 cm sec^{-2}.

The International Temperature Scale [5] was used in all measurements, except below 90°K where a provisional scale [6] was used based on a set of platinum resistance thermometers calibrated against a helium-gas thermometer.

SAMPLE AND ITS PURITY AND HEAT OF FUSION

The results given in this report are based on the measurements obtained with a second triethylborane sample. The first sample, although considered highly pure, was found to be approximately 97 mole percent pure according to the results of melting-point studies. The

[1] The figures in brackets indicate literature references at the end of this paper.

second sample was prepared with even greater care, but it was found to be only 98 percent pure. Although this material is considered to be quite impure for a substance generally used in thermodynamic investigations, the urgency for thermodynamic information on this substance prompted the calorimetric measurements in spite of its relatively high impurity content.

About 200 ml of the material was obtained in a "break-seal" ampoule. The middle 100-ml portion of the sample was used in the calorimetric measurements by vaporizing the first 1/4 of the original 200-ml sample into a flask, the next 1/2 into the calorimeter, and the remainder into the flask. After transfer into the calorimeter, the material was not raised above its triple-point temperature (180.3°K) before the purity determination. The purity of the material was computed from its equilibrium melting temperatures. The details of the method are given in an earlier report based on the calorimetric studies of diphenyl ether [7]. The results of the measurements and the computation are summarized in table 1.[2] The cryoscopic constant used in the computation was 0.043 deg^{-1}. The triple-point temperature, T_{tp}, was found to be 180.3±0.5°K.

The heat of fusion was determined in the usual manner by heating continuously through the triple-point temperature from a temperature a few degrees below the triple-point temperature to just above

[2] The temperatures given in this table and subsequent tables were obtained from the relation °K = °C + 273.1600° and are probably accurate to ±0.01°K. Whenever temperatures are expressed to the fourth decimal, the last two figures are significant only in the measurement of small temperature differences.

it and by correcting for the premelting and the heat capacity. In one series of measurements, however, the sum of various increments of heat added in the region of the triple-point temperature was used in the determination of heat of fusion. The heat-capacity correction below the triple-point temperature was based on the heat capacity obtained by the combination of extrapolation and of observed heat capacities corrected for premelting. The results of the computation are summarized in table 2. Because of the large uncertainty in the heat-capacity correction below the triple-point temperature and in the premelting correction at high impurity content, the heat of fusion (11,854 abs j $mole^{-1}$) obtained is probably in error by as much as ±100 abs j $mole^{-1}$.

HEAT CAPACITY

The results of the heat-capacity measurements, obtained from about 15° to 300°K, are given in table 3 and figure 1. After conclusion of run 2 a leak developed in the sample container which necessitated the installation of a new sample container and thermometer (#1006754). The results given in table 3 following run 2 were obtained using the new container and thermometer. The results of runs 2 and 4, which were made in the same temperature range with the two sample containers, are in satisfactory agreement.

The observed heat capacities, because of the high impurity content, show premelting as low as 140°K. No obvious bump was observed in the heat capacity below the triple-point temperature to indicate

significant amount of eutectic formation. As the nature of the system or that of the impurities was unknown, the heat capacity between only 140°K and the triple-point temperature was corrected, and this was done quite arbitrarily by the combination of extrapolation and pre-melting correction. Thus, because of the uncertainties in the correction, the heat capacity in this range of temperature could probably be in error by as much as 1 percent.

HEAT OF VAPORIZATION

The heat of vaporization was measured at 300°K. The mass, m, of material vaporized by the energy, Q, is related to the molal heat of vaporization, ΔH_v, by the equation:

$$\Delta H_v = \frac{MQ}{m} - TV\frac{dp}{dT} \qquad (1)$$

where M isthe molecular weight (98.000), V the molal volume of the liquid, and p the vapor pressure. The experimental data and the results of the computation are given in table 4. The value for the heat of vaporization (36,710 abs j $mole^{-1}$) is probably in error by 36 abs j $mole^{-1}$.

VAPOR PRESSURE

Vapor-pressure measurements were made from about 275° to 365°K. From about 335°K, the apparent vapor pressure increased with time. At 335°K the rate was about 0.03 mm Hg per hour. To minimize the extent of decomposition in any vapor-pressure observation, the sample was pumped at 0°C to eliminate volatile impurities and the apparatus was heated rapidly to the desired temperature for measurements. This

procedure was carried out on all vapor-pressure measurements above 350°K. Corrections were applied to the observed vapor pressures to zero time based on the change of apparent vapor pressure with time. The results of the measurements can be represented by the equation,

$$\log_{10} p_{mmHg} = \frac{-2837.8990}{T} - 7.0393186 \log_{10} T + 28.64715, \qquad (2)$$

whose constants have been obtained by the method of least squares. The observed values are compared in table 5 with this equation and with the values obtainable from the equation,

$$\log_{10} p_{mmHg} = \frac{-3502.8}{T} + 1.75 \log_{10} T - 0.01809T + 14.5020, \qquad (3)$$

reported by Stock and Zeidler [8] based on their vapor-pressure measurements.

DERIVED THERMAL PROPERTIES

Empirical equations were fitted to the experimental heat capacities and deviations from the empirical equations were plotted on a large scale. The heat-capacity values given in table 6 at equally spaced integral temperatures were obtained from the empirical equations and the large scale deviation curves. The values below 15°K were obtained by extrapolation using a Debye function fitted to the experimental heat capacities between 15° and 30°K.

The enthalpy, entropy, and Gibbs free energy values given in table 6 were obtained by evaluating the thermodynamic relations:

$$(H_T - H_{0°K}) = \int_0^T C_{satd.}\, dT + \Delta H_f + \int_0^T V\left(\frac{dp}{dT}\right)_{satd.} dT, \qquad (4)$$

$$(S_T - S_{0°K}) = \int_0^T C_{satd.} \frac{dT}{T} + \frac{\Delta H_f}{T_{tp}}, \qquad (5)$$

(In the above equations, the terms in ΔH_f were omitted for $T < T_{tp}$.)

and

$$(F_T - H_{0°K}) = -\int_0^T (S_T - S_{0°K})dT + \int_0^T V\left(\frac{dp}{dT}\right)_{satd.} dT. \qquad (6)$$

All symbols used have the usual significance. The equations were evaluated by numerical integration using 4-point Lagrangian integration coefficients [9].

The experimental data were used to compute the entropy of the ideal-gas triethylborane at 1 atm and 300°K. The results of the computation are summarized in table 7. The gas imperfection correction, which amounts to an almost negligible value, was obtained on the assumption that the vapor can be represented by the Berthelot equation of state and that the critical constants of triethylborane are the same as that of 3-ethyl pentane [10].

The author wishes to acknowledge his indebtedness to M.L. Reilly, J. M. Averitt, and Rita P. Park for assistance rendered in experimental measurements and computations during the course of this investigation.

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TABLE 1

Equilibrium melting temperatures of triethylborane

Mole fraction impurity = $0.043\Delta T$; °K = 273.16° + °C

Reciprocal of fraction melted	T
1/F	°K
17.63	174.1386
12.06	176.0687
8.14	177.4077
5.70	178.2291
4.24	178.7245
3.32	179.0358
2.71	179.2424
2.28	179.3886
1.84	179.5465
1.54	179.6570
1.32	179.7418
1.16	179.8127
1.03	179.8871
0	180.3[a]

Triple-point temperature, 180.3±0.5°K
Purity, 98±1 mole percent

[a] Extrapolated

TABLE 2

Molal heat of fusion of triethylborane

Molecular weight = 98.000; triple-point temperature = 180.3°K
°K = 273.16° + °C

Temperature Interval °K	Heat Input abs j mole^{-1}	Heat Capacity abs j mole^{-1}	Premelting abs j mole^{-1}	H_f abs j mole^{-1}
171.7337 to 182.6802	13,176[a]	1,893	531	11,814
172.3696 to 181.3860	12,788	1,508	574	11,854
170.4664 to 183.2866	13,657	2,226	463	11,894
			mean	11,854
			Standard deviation[b]	±40

[a] Sum of various increments of heat.

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

TABLE 3

Observed molal heat capacity of triethylborane

Molecular weight = 98.000; °K = 273.16 + °C

T_m °K	$C_{satd.}$[a] abs j deg^{-1} $mole^{-1}$	T °K
	Run 1	
172.9362	235.41	2.4049
175.1037	322.43	1.9301
176.7382	516.32	1.3390
177.8184	918.20	0.8214
178.4768	1600.07	0.4954
178.8802	2615.98	0.3113
179.1391	3999.63	0.2066
179.3155	5716.03	0.1462
179.4676	7984.74	0.1579
179.6018	11454.81	0.1105
179.6994	14957.0	0.0848
179.7773	17915.8	0.0709
179.8499	17054.2	0.0744
181.2837	336.23	2.7931
184.5396	221.67	3.7188
188.2449	223.36	3.6918
191.9236	224.89	3.6656
	Run 3	
15.9176	5.859	0.8105
16.8378	6.916	1.0301
17.9390	8.065	1.1721
19.1062	9.338	1.1624
20.5720	11.044	1.7693
22.3378	13.180	1.7621
24.2216	15.564	2.0056
26.2096	18.111	1.9703
28.3355	20.917	2.2816
30.8420	24.176	2.7314
33.7482	28.029	3.0809
36.9032	32.138	3.2293
40.2636	36.410	3.4914
43.6847	40.665	3.3508
47.5694	45.442	4.4185
51.8028	50.471	4.0483
56.5962	55.933	5.5385
61.7737	61.744	4.8166
66.9096	67.164	5.4551
72.1494	72.354	5.0246
77.2531	77.280	5.1828
82.2748	82.074	4.8606
87.2684	87.016	5.1265
92.2676	91.531	4.8720

T_m °K	$C_{satd.}$ abs j deg^{-1} $mole^{-1}$	T °K
	Run 2	
90.8292	90.275	3.1933
95.4917	93.716	6.1318
101.4000	98.427	5.6374
107.7998	103.40	7.1621
115.6186	109.71	8.4755
124.7692	116.98	9.8258
134.3384	124.53	9.3125
143.4097	133.48	8.8301
151.9079	143.72	8.1664
160.2951	158.04	8.6080
168.4844	185.27	7.7705
176.8778	1418.31	9.0164
186.6984	222.56	10.6248
197.2701	226.77	10.5185
207.7161	229.83	10.3736
218.0346	231.97	10.2635
228.2566	233.32	10.1805
238.3994	234.52	10.1050
248.4687	235.47	10.0336
258.4656	236.37	9.9602
268.8712	237.45	10.8510
	Run 4	
190.2082	224.10	15.1002
205.1327	229.22	14.7487
219.7663	232.36	14.5186
234.8188	234.39	15.5863
249.7351	236.06	14.2463
264.6973	237.55	15.6781
280.1138	239.25	15.1550
290.5776	240.60	5.7726
	Run 5	
165.7345	170.25	9.4639
176.8765	1065.29	12.8202
185.2180	221.85	3.8628
	Run 6	
222.2767	232.76	7.9935
231.0110	233.94	9.4752
240.4524	235.20	9.4076
249.8417	235.88	9.3710
259.1233	236.78	10.2786
269.3680	237.93	10.2108
279.5476	238.89	10.1483
289.6575	240.34	10.0715

[a] $C_{satd.}$ is the heat capacity along the saturation line.

TABLE 4

Molal heat of vaporization of triethylborane
at 300°K

Molecular weight = 98.000; °K = 273.16° + °C

Pressure = 56.27 mm Hg

MQ/m abs j mole^{-1}	TV dp/dT abs j mole^{-1}	ΔH_v abs j mole^{-1}
36696	16	36680
36738	16	36722
36743	16	36727
36732	16	36716
36723	16	36707
	mean	36710
	Standard deviation[a]	±19

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

TABLE 5

Vapor pressure of triethylborane

°K = 273.16° + °C

T °K	p, obs mm Hg	p, calc mm Hg	p, obs-calc mm Hg	p, S.+Z.[a] mm Hg
		Series I		
273.16	12.94	12.80	.14	10.02
273.16	12.92	12.80	.12	10.02
304.94	71.88	71.37	.51	70.17
304.94	71.99	71.37	.62	70.17
295.22	44.20	44.27	- .07	41.61
295.24	44.19	44.33	- .14	41.65
299.95	56.34	56.13	.21	54.05
299.96	56.30	56.17	.13	54.08
315.00	113.07	112.60	.47	113.68
315.02	113.19	112.70	.49	113.79
		Series II		
315.02	111.34	112.70	-1.36	113.79
344.89	357.15	359.08	-1.93	352.86
344.91	357.41	359.36	-1.95	353.08
354.88	500.78	500.67	.11	472.59
364.86	686.04	681.58	4.46	609.54
364.85	685.27	681.37	3.90	609.39
		Series III		
273.16	12.60	12.80	- .20	10.02
273.16	12.68	12.80	- .12	10.02
289.48	32.80	32.78	.02	29.70
289.48	32.92	32.78	.14	29.70
306.39	76.39	76.39	.00	75.49
306.42	76.43	76.50	- .07	75.60
315.13	113.05	113.24	- .19	114.35
315.14	113.00	113.30	- .30	114.40
325.84	176.31	176.95	- .64	179.99
337.48	275.69	276.00	- .31	276.79
337.48	275.94	276.00	- .06	276.79
		Series IV		
351.24	444.00	444.77	- .77	426.80
351.24	444.04	444.77	- .73	426.80

[a] Stock and Zeidler [8]

TABLE 6

Molal heat capacity, enthalpy, entropy, and
Gibbs free energy of triethylborane

Molecular weight = 98.000; °K = 273.16° + °C

T °K	$C_{satd.}$[a] abs j deg^{-1} $mole^{-1}$	$(H_T-H_{0°K})$ abs j $mole^{-1}$	$(S_T-S_{0°K})$ abs j deg^{-1} $mole^{-1}$	$-(F_T-H_{0°K})$ abs j $mole^{-1}$
0	0	0	0	0
5	0.7010	1.67	0.624	1.45
10	2.095	8.20	1.476	6.57
15	4.943	25.07	2.807	17.03
20	10.37	62.94	4.947	36.00
25	16.55	130.0	7.915	67.87
30	23.08	229.0	11.51	116.2
35	29.69	360.9	15.56	183.7
40	36.07	525.4	19.95	272.4
45	42.29	721.4	24.56	383.6
50	48.35	948.1	29.33	518.2
55	54.13	1204.	34.21	677.0
60	59.78	1489.	39.16	860.4
65	65.18	1802.	44.16	1069.
70	70.24	2140.	49.18	1302.
75	75.11	2504.	54.19	1561.
80	79.91	2891.	59.19	1844.
85	84.76	3303.	64.18	2152.
90	89.55	3739.	69.17	2486.
95	93.33	4196.	74.11	2844.
100	97.31	4673.	79.00	3227.
105	101.2	5169.	83.84	3634.
110	105.2	5686.	88.64	4065.
115	109.2	6222.	93.41	4520.
120	113.1	6777.	98.14	4999.
125	117.1	7353.	102.8	5502.
130	120.9	7948.	107.5	6027.
135	125.1	8563.	112.1	6577.
140	129.0	9198.	116.8	7149.
145	133.2	9854.	121.4	7744.
150	137.5	10531.	126.0	8363.
155	141.8	11229.	130.5	9004.
160	146.0	11948.	135.1	9668.
165	150.3	12689.	139.7	10355.
170	154.6	13451.	144.2	11065.
175	158.9	14235.	148.8	11797.
180	163.2	15040.	153.3	12552.
180.3	163.7	15089.	153.6	12598.
180.3	219.6	26943.	219.3	12598.
185	221.8	27981.	225.3	13692.

TABLE 6 (Cont.)

T °K	$C_{satd.}$[a] abs j deg^{-1} $mole^{-1}$	$(H_T-H_{0°K})$ abs j $mole^{-1}$	$(S_T-S_{0°K})$ abs j deg^{-1} $mole^{-1}$	$-(F_T-H_{0°K})$ abs j $mole^{-1}$
190	224.1	29096.	231.2	14833.
195	226.0	30221.	237.1	16004.
200	227.6	31355.	242.8	17204.
205	229.2	32497.	248.4	18432.
210	230.5	33646.	254.0	19688.
215	231.5	34801.	259.4	20971.
220	232.3	35961.	264.7	22282.
225	233.1	37125.	270.0	23619.
230	233.7	38292.	275.1	24981.
235	234.3	39462.	280.1	26369.
240	234.8	40634.	285.1	27782.
245	235.3	41810.	289.9	29220.
250	235.9	42988.	294.7	30681.
255	236.4	44169.	299.4	32167.
260	237.0	45352.	304.0	33675.
265	237.5	46539.	308.5	35206.
270	238.1	47728.	312.9	36760.
275	238.6	48920.	317.3	38335.
280	239.2	50114.	321.6	39932.
285	239.7	51312.	325.8	41551.
290	240.3	52512.	330.0	43190.
300	241.4	54921.	338.2	46532.

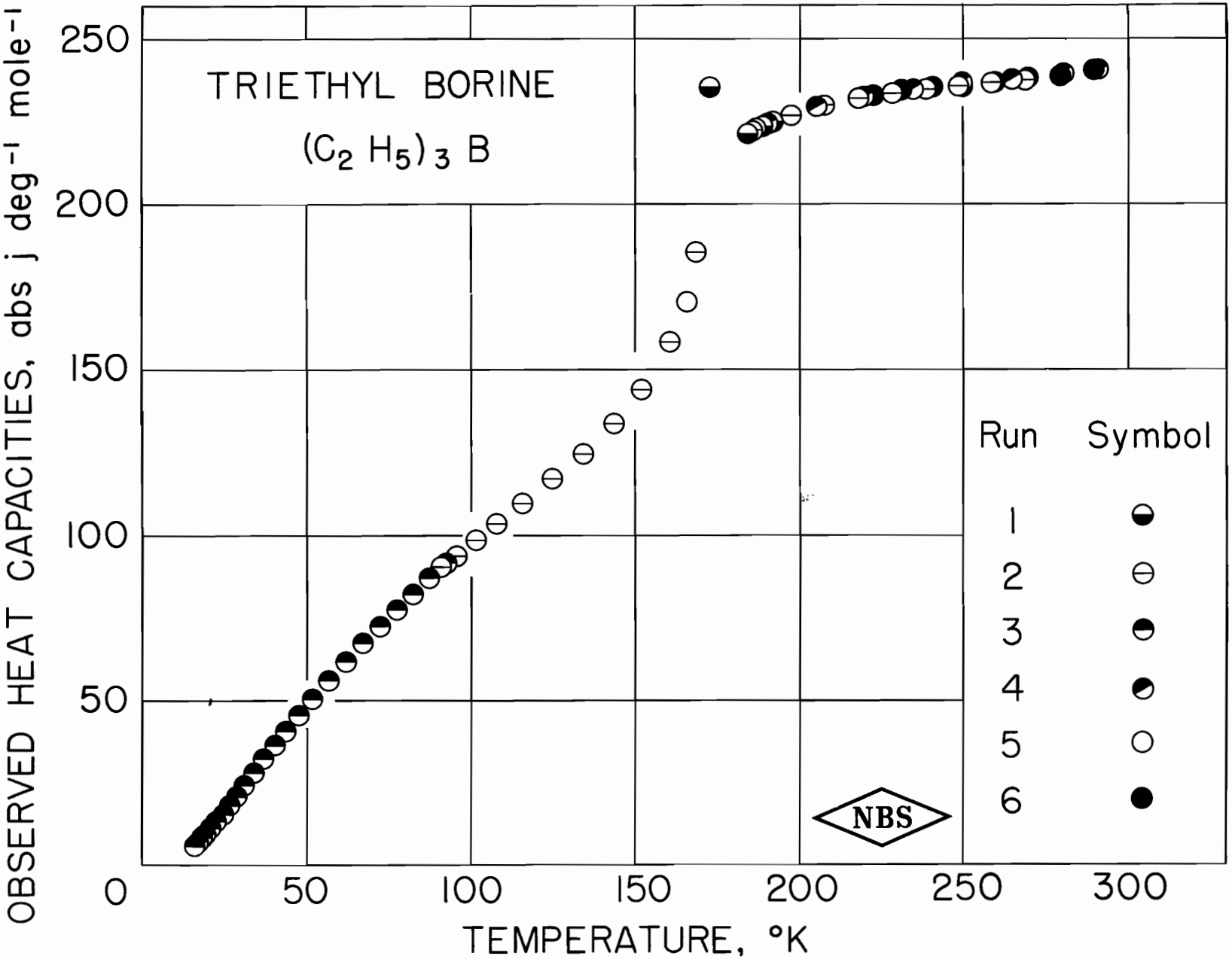
[a] $C_{satd.}$ is the heat capacity along the saturation line.

TABLE 7

Summary of experimental molal entropy of triethylborane.

molecular weight = 98.000; °K = 273.16° + °C

	abs j deg^{-1} $mole^{-1}$
$S_{15°}$, Debye extrapolation	2.8 ± .3
$\Delta S_{15°}$ to 180.3°, solid	150.8 ± .3
ΔS, fusion 11,854/180.3	65.7 ± .6
$\Delta S_{180.3°}$ to 300°, liquid	118.9 ± .2
ΔS, vaporization, 36710/300	122.4 ± .1
ΔS, gas imperfection	0.1
ΔS, compression to 1 atm., R ln(56.27/760)	-21.6
S, ideal gas at 1 atm and 300°K	439.1 ± 0.8


TRIETHYL BORINE
$(C_2H_5)_3B$
OBSERVED HEAT CAPACITIES, abs j deg⁻¹ mole⁻¹
TEMPERATURE, °K
0
50
100
150
200
250
300
Run
Symbol
1
2
3
4
5
6
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THE NATIONAL BUREAU OF STANDARDS

Functions and Activities

The functions of the National Bureau of Standards are set forth in the Act of Congress, March 3, 1901, as amended by Congress in Public Law 619, 1950. These include the development and maintenance of the national standards of measurement and the provision of means and methods for making measurements consistent with these standards; the determination of physical constants and properties of materials; the development of methods and instruments for testing materials, devices, and structures; advisory services to Government Agencies on scientific and technical problems; invention and development of devices to serve special needs of the Government; and the development of standard practices, codes, and specifications. The work includes basic and applied research, development, engineering, instrumentation, testing, evaluation, calibration services, and various consultation and information services. A major portion of the Bureau's work is performed for other Government Agencies, particularly the Department of Defense and the Atomic Energy Commission. The scope of activities is suggested by the listing of divisions and sections on the inside of the front cover.

Reports and Publications

The results of the Bureau's work take the form of either actual equipment and devices or published papers and reports. Reports are issued to the sponsoring agency of a particular project or program. Published papers appear either in the Bureau's own series of publications or in the journals of professional and scientific societies. The Bureau itself publishes three monthly periodicals, available from the Government Printing Office: The Journal of Research, which presents complete papers reporting technical investigations; the Technical News Bulletin, which presents summary and preliminary reports on work in progress; and Basic Radio Propagation Predictions, which provides data for determining the best frequencies to use for radio communications throughout the world. There are also five series of nonperiodical publications: The Applied Mathematics Series, Circulars, Handbooks, Building Materials and Structures Reports, and Miscellaneous Publications.

Information on the Bureau's publications can be found in NBS Circular 460, Publications of the National Bureau of Standards ($1.25) and its Supplement ($0.75), available from the Superintendent of Documents, Government Printing Office. Inquiries regarding the Bureau's reports and publications should be addressed to the Office of Scientific Publications, National Bureau of Standards, Washington 25, D. C.