

Table III. Literature Values of the Second Virial Coefficients

Temp., ° C.	-B Liters/ Mole	Temp., ° C.	-B Liters/ Mole
<i>n</i> -Pentane (8)		<i>n</i> -Heptane (8)	
25.0	1.194	76.2	1.819
32.9	1.117	85.1	1.691
44.9	1.011	95.3	1.560
55.8	0.923	104.9	1.446
65.8	0.863	115.9	1.325
75.8	0.800	127.6	1.212
84.8	0.758	140.5	1.108
95.4	0.701		
105.7	0.652	Benzene (4)	
115.2	0.612	70.0	1.035
127.8	0.578	80.0	0.971
140.4	0.517		
<i>n</i> -Hexane (8)		100.0	0.852
		100.0	0.839
		125.0	0.733
44.8	1.598	Acetone (7)	
55.8	1.419	46.2	1.644
64.5	1.338	56.3	1.527
74.8	1.235	61.8	1.406
85.3	1.144	67.8	1.362
97.4	1.050	68.2	1.342
104.5	0.992	78.0	1.257
113.5	0.928	88.0	1.134
122.7	0.880	95.2	0.965
136.9	0.800	111.8	0.854
		132.4	0.651

A few checks on the calculated values of B were made by using

$$\lim_{V \rightarrow \infty} (z - A)V$$

The quantity $(z - A)V$ was plotted against $1/V$. The values of B , as given by the intercepts, were in agreement with the values obtained as explained above. The horizontal slope indicated that the third virial coefficient was, within the experimental error, equal to zero.

The values of the second virial coefficient, B , reported in the literature in the temperature range covered in this study are given in Table III and are plotted in Figures 3, 5, and 6. There seems to be close agreement with the authors' results, except for acetone at low temperatures.

The calculation of the intermolecular potential parameters will be discussed in a forthcoming paper.

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Density, Electrical Conductivity, and Vapor Pressure of Concentrated Phosphoric Acid

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Density and specific conductivity for H₃PO₄ in the concentration range 86 to 102 wt. % from room temperature to 170° C. are presented. The vapor pressure portion gives data for 77 to 102 wt. % H₃PO₄ for temperatures between 130° and 170° C. In each case, equations are given which provide a good least squares fit to the experimental data.

THE DENSITIES of phosphoric acid solutions have been determined by others (2, 3) at concentrations up to 92% and at temperatures up to 80° C. This work concerns the experimental determination of densities at higher concentrations and higher temperatures.

EXPERIMENTAL

Pycnometers of 25-ml. capacity were calibrated against water at 25.00° C. The cubical coefficient of expansion of borosilicate glass was used to calculate the volumes at higher temperatures. All weights were corrected to vacuum. Temperature was controlled to 0.01° C. and measured to 0.1° C. in oil thermostats. Solutions of phosphoric acid were prepared by dehydration of the C.P. grade at 150° to 175° C.

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in ovens. To avoid contamination, preparations were made in FEP bottles (fully fluorinated ethylene-propylene copolymer). The bottles were preconditioned at 150° to 175° C. Then, acid of known composition (usually 85.1%, determined by repeated pH titration) was weighed into them. The acid composition was calculated from the weight loss of water after heating. The concentrated phosphoric acid solutions were stored in FEP bottles at room temperature before use. The experimental data is given in Table I.

CORRELATION OF DENSITY DATA

Plots of the data in Table I reveal the density to be a linear function of the temperature, including the points at 25° C. Furthermore, the density is also linear in the concentration over the limited range studied here.

Table I. Experimentally-Determined Densities of C.P. Phosphoric Acid, G./Ml.

Mean Value of Two Determinations Listed Together with Average Deviation from Mean $\times 10^4$

Wt. % H ₃ PO ₄	Solution Temp., ° C.					
	25	130	140	150	160	170
86.68	...	1.6141 ± 3	1.6028 ± 23	1.5940 ± 20
88.28	1.7261 ± 0	1.6324 ± 4	1.6237 ± 0	1.6146 ± 8
89.58	...	1.6479 ± 9	1.6387 ± 5	1.6301 ± 0	1.6215 ± 0	...
90.63	...	1.6594 ± 2	1.6508 ± 6	1.6420 ± 2	1.6332 ± 4	1.6249 ± 4
92.97	...	1.6894 ± 4	1.6806 ± 2	1.6724 ± 7	1.6630 ± 1	1.6544 ± 3
93.82	...	1.6992 ± 0	1.6918 ± 0	1.6821 ± 0	...	1.6653 ± 0
94.38	1.8010 ± 3	1.7080 ± 5	1.7000 ± 6	1.6911 ± 6	1.6826 ± 5	1.6737 ± 1
97.48	1.8390 ± 1	1.7478 ± 1	1.7395 ± 1	1.7308 ± 1	1.7224 ± 1	1.7145 ± 3
98.66	1.8522 ± 1	1.7607 ± 1	1.7528 ± 1	1.7446 ± 2	1.7353 ± 2	1.7270 ± 0
100.93	1.8818 ± 2	1.7908 ± 4	1.7830 ± 2	1.7752 ± 2	1.7662 ± 0	1.7571 ± 2
101.60	1.8875 ± 18	1.7973 ± 10	1.7890 ± 10	1.7807 ± 13	1.7723 ± 16	1.7643 ± 13

Accordingly, the following equation was fitted to the data by the method of least squares.

$$d = A_0 + A_1W - (A_2 - A_3W)t \quad (1)$$

For density, d , expressed in grams per cm.³, the concentration, W , expressed in weight per cent, and the temperature, t , as °C., the constants in Equation 1 have the following values: $A_0 = 0.68235$; $A_1 = 1.20811 \times 10^{-2}$; $A_2 = 1.2379 \times 10^{-3}$; $A_3 = 3.7938 \times 10^{-6}$. Equation 1 reproduces the data in Table I with an average deviation of 0.0009 gram per cm.³ (0.05%).

For comparison with the work of Egan and Luff (3), their data extrapolated to 90, 95, and 100 wt. % are listed in Table II, together with the differences between these literature-derived values and those calculated from Equation 1 in a temperature range where Egan and Luff's data were taken.

The agreement is good below 100% acid, with a tendency for the equation to predict very slightly lower densities than the listed extrapolated values from the literature.

Obviously, the equations derived here do not yield accurate acid densities over the entire range of concentration 0 to 100%. The density is not a linear function of concentration over a large range of concentration. Luff and Egan have fitted cubic equations to their data, and the work here shows that the density vs. concentration plots at a constant temperature begin to show very slight curvature below about 88% acid.

CONDUCTIVITY OF PHOSPHORIC ACID

References (4), (7), and (9) give previous H₃PO₄ conductivity data. The results of the Monsanto Co. agree with those of Fedorchenko *et al.* at low temperatures, but the difference between them increases consistently for higher temperatures and becomes about 25% at 140°C., with the Monsanto measurements giving the larger conductivities. In the range where they overlap, our results are in general agreement with those of the Monsanto Co., but are 5 to 7% higher. These discrepancies might be due to differences in the composition arising from the "freezing

Table II. Extrapolated Data of Egan and Luff (3) With Deviation Calculated from Equation 1

Wt. % H ₃ PO ₄	Solution Temp., ° C.			
	25	40	60	80
90	1.7452	1.7331	1.7168	1.6996
	+0.0020	+0.0007	-0.0010	-0.0017
95	1.8082	1.7959	1.7792	1.7612
	-0.0001	-0.0010	-0.0018	-0.0014
100	1.8741	1.8616	1.8446	1.8257
	-0.0051	-0.0054	-0.0056	-0.0039

in" of higher-temperature equilibria on cooling after preparation of the concentrated acids (6) or from their attack on glass containers. These factors should be minimal in the work reported here, since preparation temperatures were in the same range as those of the measurements, and the acids were always handled in chemically resistant plastic containers except during the time required for a measurement. The conductivity data which follows cover concentration from 85 to 100 wt. %, between 130° and 170° C.

EXPERIMENTAL

Measurements were made in a Jones type conductivity cell made of borosilicate glass and designed to allow deep immersion in a silicone oil bath. The cell constant was 296.16, determined by standardization with 1D KCl at 25°C. Temperature was controlled and measured to 0.1°C. Resistance measurements were made at a frequency of 1000 cycles per second with a Model 291A Universal Impedance Bridge and were reproducible after temperature cycling. Solutions were prepared in the same manner as were those for the density determinations described in Section I. Table III and Figure 1 show the data obtained.

CORRELATION OF CONDUCTIVITY DATA

At any given concentration, the conductivity, K , is a linear function of the temperature. The conductivity can, therefore, be expressed as an equation of the form

$$K = f(W) + g(W)t \quad (2)$$

where $f(W)$ and $g(W)$ are functions of the concentration.

It was impossible to obtain a good fit to the data over the whole range of concentration, using polynomial functions for $f(W)$ and $g(W)$. However, by dividing the data into two groups, all the data below 94 wt. % H₃PO₄ could be fit satisfactorily using linear concentration functions, while that above 94 wt. % could be fit by quadratic func-

Table III. Specific Conductivity (1/Ohm-Cm.) Data for Phosphoric Acid

Composition, Wt. % H ₃ PO ₄	Solution Temp., ° C.				
	130.00	140.12	150.25	160.92	170.42
85.10	0.4904	0.5299	0.5685
89.10	0.4747	0.5169	0.5589	0.6033	...
90.45	0.4702	0.5133	0.5568	0.6020	0.6411
91.42	0.4665	0.5098	0.5536	0.5996	0.6397
92.75	0.4606	0.5055	0.5495	0.5960	0.6384
96.97	0.4369	0.4816	0.5280	0.5763	0.6184
97.96	0.4274	0.4717	0.5178	0.5664	0.6082
98.93	0.4137	0.4578	0.5029	0.5516	0.5936
99.57	0.4019	0.4461	0.4904	0.5386	0.5819
99.72	0.3986	0.4421	0.4872	0.5347	0.5774

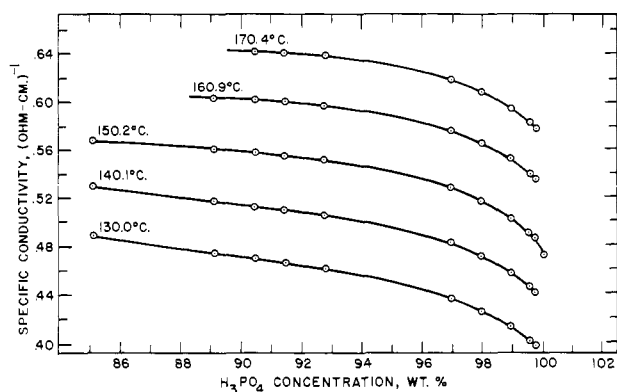


Figure 1. Electrical conductivity of H₃PO₄ solutions

tions. The resulting equations, obtained by least squares, are given below:

For $W = 84$ to 94 wt. % H₃PO₄

$$K \text{ (ohm-cm.)}^{-1} = A_0 - A_1 W - (A_2 - A_3 W)t \quad (3A)$$

where

$$A_0 = 1.01365$$

$$A_1 = 1.21548 \times 10^{-2}$$

$$A_2 = 1.5447 \times 10^{-3}$$

$$A_3 = 6.42463 \times 10^{-5}$$

$$\text{Av. Dev., Calcd. - Obsd.} = 0.08\%$$

For $W = 95$ to 99 wt. % H₃PO₄

$$K \text{ (ohm-cm.)}^{-1} = -A_0 + A_1 W - A_2 W^2 - (A_3 - A_4 W + A_5 W^2)t \quad (3B)$$

where

$$A_0 = 3.45285$$

$$A_1 = 7.77924 \times 10^{-2}$$

$$A_2 = 4.50762 \times 10^{-4}$$

$$A_3 = 6.24637 \times 10^{-2}$$

$$A_4 = 1.387186 \times 10^{-3}$$

$$A_5 = 7.18336 \times 10^{-6}$$

$$\text{Av. Dev., Calcd. - Obsd.} = 0.17\%$$

WATER VAPOR PRESSURE OVER PHOSPHORIC ACID

This study presents detailed vapor pressure data obtained for H₃PO₄ solutions in the concentration range from 75 to 100 wt. % and between the temperatures 130° and 170° C. Earlier work (1, 5) has been concerned mostly with much higher temperatures and compositions than those of interest here.

EXPERIMENTAL

A modified version of Stokes' method of bithermal equilibration through the vapor phase (10, 11) was used. In this method, pure water held at a constant temperature is equilibrated with a solution held at a higher temperature. The composition of the solution changes until its vapor pressure is the same as that of the water. Since the vapor pressure over pure water is known accurately as a function of temperature (8), it remains only to determine the equilibrium composition of the solution, which is calculated from the change in weight after equilibration.

The H₃PO₄ solution and water samples were contained in thin-walled borosilicate glass flasks of about 80-ml.

volume, with long narrow necks to prevent convection currents and to allow deep immersion in the oil baths. To eliminate slow corrosion of the glass by the acid, noticed while taking the data at 150° C., the flasks were lined with gold for the other determinations. The flasks were connected to a portable vacuum manifold constructed of borosilicate glass and high-vacuum stopcocks. Apiezon H, a vacuum grease having a high melting point and low vapor pressure up to 225° C., was used. To prevent condensation of water in the connecting manifold, it was heated by an electric heating tape wrapped around it and covered with alternating layers of aluminum foil and thermal insulation.

To make a determination, 4 or 5 grams of Fisher Certified H₃PO₄ solution of known composition (usually 85.2 wt. %) were weighed into one flask. Distilled water was placed in the other and the flasks were connected to the vacuum manifold. The liquids were degassed by alternate freezing and warming under a vacuum of 10⁻⁴ torr. After degassing, the manifold was disconnected from the vacuum system and placed such that each flask was immersed in a separate oil bath with temperatures controlled to ±0.01° C. Sixteen hours proved to be ample equilibration time. After equilibration, the flask stopcocks were closed, and the flasks were disconnected and weighed. The final composition was calculated from the weight change, corrected for the weight of water vapor in the flask above the solution. The original solution composition was determined by careful pH titration. Table IV and Figure 2 show the data obtained.

Table IV. Water Vapor Pressure Over Concentrated H₃PO₄, Mm. of Hg

129.65° C.		139.72° C.		149.88° C.		169.75° C.	
Wt. % H ₃ PO ₄	Vapor Pressure	Wt. % H ₃ PO ₄	Vapor Pressure	Wt. % H ₃ PO ₄	Vapor Pressure	Wt. % H ₃ PO ₄	Vapor Pressure
74.44	753.24	79.52	753.24	83.68	753.10	89.30	751.89
75.78	699.63	80.98	677.12	84.63	700.13	89.53	725.37
77.03	652.82	82.33	608.64	86.18	610.18	90.61	629.24
79.45	558.53	84.13	517.90	86.66	582.05	91.90	519.84
81.42	483.40	86.86	394.36	87.72	519.80	93.25	428.56
83.51	411.94	89.21	298.97	88.69	446.60	93.30	428.56
85.10	350.90	90.76	242.88	88.98	446.60	94.07	362.40
86.61	298.97	92.40	184.26	89.12	428.60	94.11	365.32
88.17	251.45	93.37	154.14	89.19	430.20	95.02	297.74
89.70	211.40	94.58	122.07	89.90	380.30	95.11	289.96
91.08	169.10	95.63	95.81	89.99	397.80	95.88	241.86
92.45	134.10	96.60	74.50	91.18	337.00	96.91	185.07
93.76	105.64	97.82	48.89	92.06	273.90	97.41	157.90
94.82	82.46	99.02	31.28	93.30	231.70	98.24	122.08
95.93	63.79	100.07	19.47	94.13	193.54	98.33	122.04
96.81	48.89	102.89	4.58	94.17	193.78	98.93	95.80
97.69	35.06			94.25	193.10	99.61	74.50
98.46	26.43			94.29	193.09	100.57	48.89
99.14	18.88			94.66	173.14	101.50	31.28
101.94	4.58			94.72	172.82	102.17	21.98
				95.06	158.01	102.36	20.69
				95.13	158.01	105.66	4.58
				95.72	133.86		
				95.84	134.95		
				95.95	133.35		
				96.50	111.19		
				96.60	111.02		
				96.61	110.82		
				96.62	110.39		
				96.94	98.72		
				97.14	97.14		
				97.77	74.50		
				98.82	48.89		
				99.80	31.64		
				99.87	33.12		
				100.01	30.39		
				100.67	20.57		
				103.79	4.58		
				103.97	4.58		

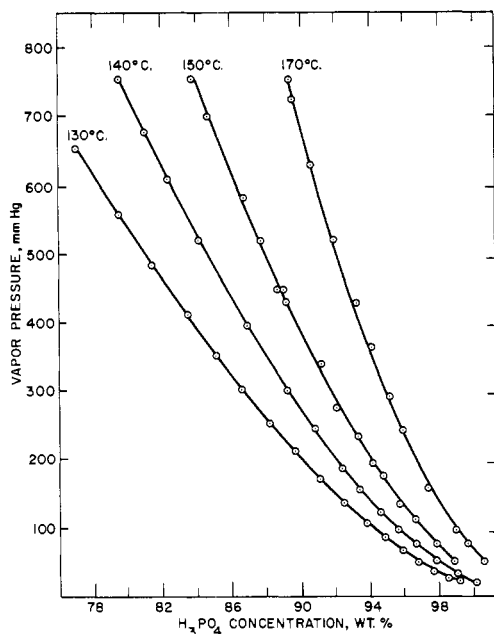


Figure 2. Vapor pressure above H_3PO_4 solutions

CORRELATION OF VAPOR PRESSURE DATA

An equation of very simple form can be fitted to the data given in Table IV for all the temperatures and for all concentrations up to those at which pyrophosphoric acid begins to form. The vapor pressure, P_0 , above pure water is given by statistical mechanics as

$$P_0 = C_0 RT \frac{f_g}{f_l} \exp(-\Delta H_0/RT) \quad (4)$$

where f_g and f_l are the partition functions for the gas and the liquid, respectively, C_0 is the molar concentration of liquid water at absolute temperature T , R is the gas constant, and ΔH_0 is the heat of vaporization. If we assume that the heat of vaporization from H_3PO_4 solution is the weighted average of the heat of vaporization, ΔH_A , of a water molecule completely surrounded by H_3PO_4 molecules and ΔH_0 , a similar equation may be written for the H_3PO_4 solution. Dividing this by Equation 4 yields

$$\frac{P}{P_0} = \frac{C}{C_0} \kappa \exp[-x(\Delta H_A - \Delta H_0)/RT] \quad (5)$$

where P and C are the vapor pressure and water concentration, respectively, of the solution. κ is the ratio of the partition functions for pure liquid water and for the solution. It is a slowly varying function of temperature and concentration, but will be treated here as a constant. In Equation 5, x is the mole fraction of H_3PO_4 in the solution. The mole fraction can be calculated from the weight per cent, W , by

$$x = \frac{0.01 W}{0.01 W + 0.0544 (100 - W)}$$

and C is given using data for density, ρ , by

$$C = (1 - x) \rho / [18.016 + 79.99 (1 - x)]$$

In Figure 3, the vapor pressure data is plotted in the form suggested by Equation 5. All the data points cluster about the same straight line except at concentrations where

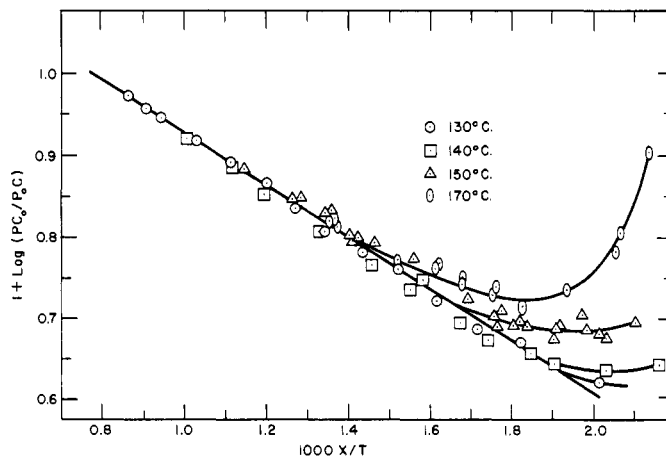


Figure 3. Graphical fit of Equation 5 to H_3PO_4 vapor pressure data

Table V. Coefficients for Equation 6 and Thermal Data

H_3PO_4 Concn.		ΔH Kcal. Vapor	B.P., °C.
Wt. %, W	Mole %, X		
84	49.11	10.744	150.92
85	51.02	10.749	153.97
86	53.04	10.763	157.24
87	55.16	10.789	160.78
88	57.41	10.828	164.60
89	59.80	10.883	168.74
90	62.33	10.956	173.25
91	65.02	11.053	178.15
92	67.89	11.176	183.50
93	70.95	11.332	189.35
94	74.23	11.528	195.77
95	77.74	11.771	202.80
96	81.52	12.072	210.54
97	85.60	12.445	219.04
98	90.01	12.906	228.40
99	94.79	13.476	238.70
100	100.00	14.181	250.01

Correlating equation for vapor pressure of H_3PO_4 (80 to 101 wt. % acid in range 130° to 170° C.)

$$\text{Log } P = (A_0 - A_1 X + A_2 X^2 + A_3 X^3)^{-1}$$

$$(A_4 - A_5 X + A_6 X^2 + A_7 X^3)^{T^{-1}}$$

P = Water vapor pressure, mm. of Hg; T = Temperature, °K.; X = Mole % $H_3PO_4 = W/0.01W + 0.0543955 (100 - W)$; $A_0 = 10.73502$; $A_1 = 0.0740558$; $A_2 = 5.46493 \times 10^{-4}$; $A_3 = 1.03388 \times 10^{-7}$; $A_4 = 2838.25$; $A_5 = 18.1247$; $A_6 = 0.125648$; $A_7 = 8.16949 \times 10$.

pyrophosphoric acid is formed. This begins at $1000 x/T = 1.6, 1.7, 1.8,$ and 1.9 for $170^\circ, 150^\circ, 140^\circ, 130^\circ$ C., respectively. All these points lie in the range 92 to 95 wt. % H_3PO_4 , where other work (9, 15) also indicates formation of $H_4P_2O_7$.

Evaluating the constants in Equation 5 by means of the plot in Figure 3 gives the following equation, which gives vapor pressure values to within an average error of <3% over the range of its applicability. Equation 6 would probably give satisfactory results also at somewhat lower concentrations than those studied in this report.

$$\frac{P}{P_0} = 1.77 \frac{C}{C_0} \exp(-735 x/T) \quad (6)$$

To find empirical equations which would reproduce the data over the entire concentration range, least squares fits

to polynomials of various orders were tried. To obtain a good fit without using polynomials of very high order, it was necessary to express concentrations as mole per cent, X , rather than weight per cent. Doing this, the data for $\log P$ could be fit by a cubic polynomial for each of the four temperatures. At any given concentration, $\log P$ was a linear function of $1/T$. Therefore, a least squares fit to all the data was obtained for the following expression

$$\log P = A_0 + A_1X + A_2X^2 + A_3X^3 + (A_4 + A_5X + A_6X^2 + A_7X^3)T^{-1} \quad (7)$$

The values for the coefficients in Equation 7 are listed in Table V, along with heats of vaporization and boiling points, which may be calculated from it. Equation 7 reproduces the experimental data to within $\pm 0.4\%$ in $\log P$ (or about 2% in P).

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Heat Capacities and Thermodynamic Properties of α -Beryllium Nitride from 25° to 310° K.

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The low-temperature heat capacity of α -Be₃N₂ was measured from 25° to 310° K. by adiabatic calorimetry. No thermal anomalies were observed. Corrections were applied for the impurities, 2.7% BeO, 0.2% Be, 0.1% C, and 0.3% Fe. Smoothed thermodynamic functions at even temperatures are tabulated. The values of C_p° , S_p° , $(H_p^\circ - H_f^\circ)/T$, and $-(G_p^\circ - H_f^\circ)/T$ at 298.15° K. are 15.39, 8.170, 5.682, and 2.488 cal. per mole ° K.

THE LOW-TEMPERATURE heat capacity of α -Be₃N₂ has been measured in connection with a program to measure and tabulate the thermodynamic properties of rocket exhaust products. Heretofore, the entropy of α -Be₃N₂ has been determined from high-temperature equilibrium studies (7, 15) with relatively low precision. Recently, Furukawa and Reilly (5) reported the heat capacity of α -Be₃N₂. The high-temperature enthalpy has been reported (1, 13) in two investigations.

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EXPERIMENTAL

Sample. The sample was supplied by Brush Beryllium Co. Before measurements were made, the sample was checked for purity and crystal structure. The x-ray powder pattern revealed only cubic or α -Be₃N₂ (2). The various analyses showed the following composition: α -Be₃N₂, 96.7% (hydrolysis of nitride to ammonia and titration); BeO, 2.7% (neutron activation analysis for oxygen); Be, 0.2% (quantitative hydrogen gas evolution in acid); C, 0.1% (combustion to CO₂); Fe, 0.3% (neutron activation and spectrographic analyses).