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Some Physical Properties of Borazole

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The density, viscosity and surface tension of borazole have been measured over a temperature range from approximately 225 to 313 °K. The refractive index has been measured at 20°. A melting point differing from that in the literature has been obtained.

A method for the synthesis of borazole, $B_3N_3H_6$ has been described in a recently published paper.[1]

The density, viscosity and surface tension have been measured over a range of temperatures between the boiling and freezing points and the refractive index has been measured at 20°. A melting point differing from that in the literature has been obtained.

Density.—The density of borazole was determined in a Pyrex dilatometer charged with 3.6900 g. of borazole. Corrections were made for the change in dilatometer volume with temperature, and the data in Table I were taken. (Dilatometer volume was calibrated with mercury weighed to 0.1 mg.)

TABLE I

DENSITY

Accuracy ±0.1%.

Temp. (°K.)	238.2	264.1	273.2	280.8
Density (g./cc.)	0.8988	0.8720	0.8618	0.8540
Temp. (°K.)	288.4	305.5	312.4	313.9
Density (g./cc.)	0.8454	0.8265	0.8204	0.8172

The equation of the line described by these data was determined by the method of least squares to be

$$d = 1.1551 - 0.001074T \quad (d = \text{g./cc.}; T = \text{°K.})$$

Viscosity.—The absolute viscosity of borazole was determined in the manner described by Smith and Miller[2] with the same viscometer and the same resultant accuracy. It was necessary to handle borazole in a vacuum system to prevent the hydrolysis which occurred readily. The data taken are presented in Table II.

TABLE II

VISCOSITY

Accuracy ±2.5%.

Temp. (°K.)	T (sec.)	d (g./cc.)	h (cm.)	n(millipoises)
224.8	57.9	0.9138	8.250	6.85
228.3	54.6	.9100	8.475	6.57
230.2	53.4	.9080	8.465	6.39
243.3	45.0	.8939	8.450	5.16
251.4	41.6	.8852	8.435	4.65
273.2	35.6	.8618	8.240	3.65
281.5	33.8	.8529	8.215	3.36
292.3	31.8	.8413	8.190	3.05
299.6	30.5	.8334	8.185	2.85

The equation of the curve representing these data is

$$n = 47.3(10^{-5})d^{1/2}e^{665.5d/T} \quad (n = \text{poises}; d = \text{g./cc.}; T = \text{°K.})$$

The curve of Fig. 1 is drawn according to this equation and shows the agreement with actual data.

(1) H. I. Schlesinger, *et al.*, THIS JOURNAL, **76**, 3303 (1954).
(2) S. H. Smith, Jr., and R. R. Miller, *ibid.*, **72**, 1452 (1950).

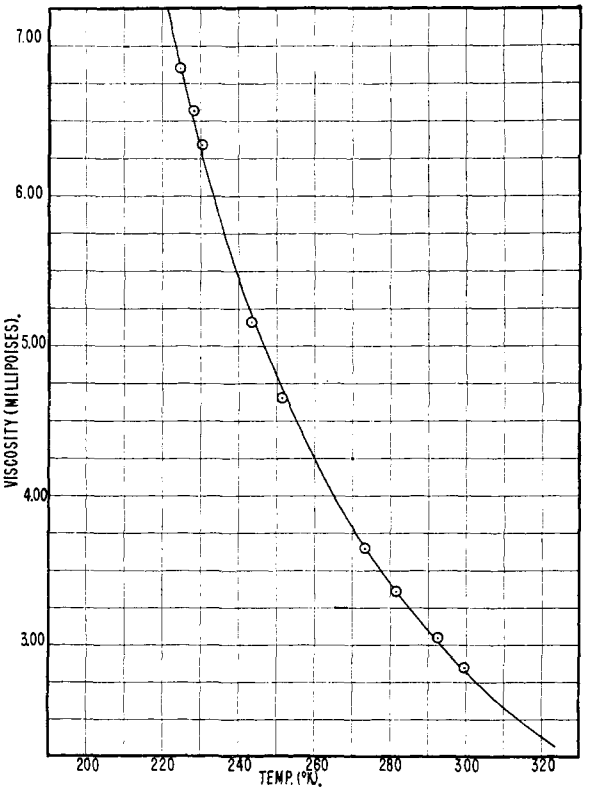

Fig. 1.—Borazole viscosity.

Surface Tension.—Since the dimensions of the capillary on the viscometer were known accurately, the surface tension could be measured readily by the capillary rise method at the same time viscosity data were being taken. Surface tension data are shown in Table III.

TABLE III

SURFACE TENSION

Accuracy ±3%.

Temp. (°K.)	Capillary rise (cm.)	γ(dynes/cm.)	Temp. (°K.)	Capillary rise (cm.)	γ(dynes/cm.)
239.6	1.86	27.6	273.2	1.62	23.1
246.1	1.82	26.8	280.4	1.57	22.2
253.0	1.77	25.8	286.0	1.54	21.6
261.5	1.72	24.9	291.0	1.50	20.9
268.0	1.67	23.9	295.8	1.49	20.6

The equation fitting these data is

$$\gamma = (58.3 - 0.120T)d^{2/3}$$

(γ = dynes/cm.; T = °K.; d = g./cc.)

The curve in Fig. 2 is drawn according to this equation.

Refractive Index.—The refractive index of borazole was measured with a Bausch and Lomb dipping

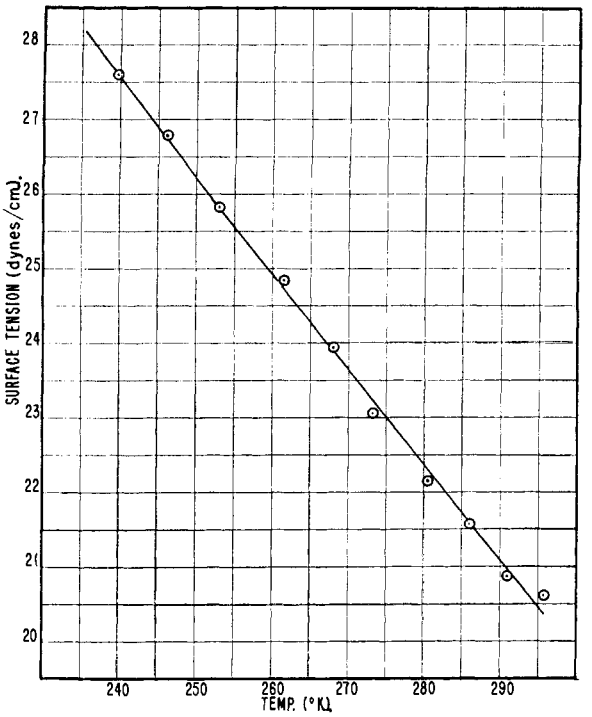

Fig. 2.—Borazole surface tension.

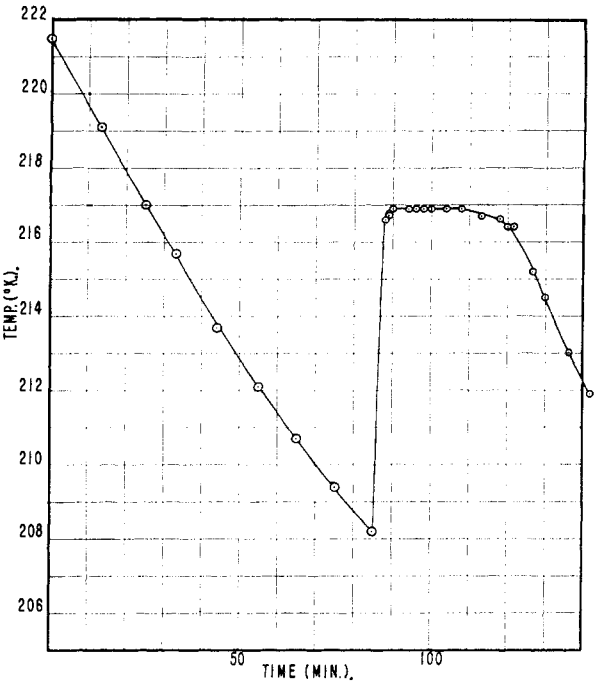

Fig. 3.—Borazole freezing point curve.

refractometer, making use of the accessory cup which could be mounted against a tapered joint on the instrument with a threaded collar. The cup was placed in a vacuum system, and the requisite volume of borazole distilled into it; then the cup was transferred to the instrument under a nitrogen blanket in a dry-box.

A dipping refractometer is capable of giving data accurate to ±0.00002 unit under carefully controlled conditions. Unfortunately, under the conditions of our measurement, the line of demarcation between the bright and dark fields could not be resolved sharply, and the accuracy of the reading was only ±0.0002 unit. The $n^{20}D$ value obtained for borazole was 1.3821.

Melting Point.—Since the preparation of a pure sample of borazole was rather involved and the compound was readily subject to deterioration by moisture, it seemed advisable to check the purity of the sample after the foregoing measurements had been completed. This was done by a determination of the freezing point in an apparatus similar to that described by Skau.[3] The temperature was read to ±0.1° by iron–constantan thermocouple. The temperature–time curve of Fig. 3 was obtained. Considerable supercooling occurred and the crystallization was seeded by touching the tip of the freezing point tube with liquid nitrogen. The temperature leveled off at 216.9°K. This differs slightly from the literature value of 215.2°K., but on the basis of the slope and relative length of the flat portion of our freezing curve coupled with the fact that our melting point is high rather than low, we believe our sample was of high purity.

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(3) E. L. Skau, *Proc. Am. Acad. Arts, Sci.*, **67**, 551 (1932).