hydrogenation, 1.0 ml. of 10 *M* sodium hydroxide was added and the mixture was heated to boiling for three minutes; after cooling, it was neutralized with glacial acetic acid.

After the deacetylation, ethanol was removed by distillation *in vacuo* and a concentrated aqueous solution of 500 mg. of barium acetate was added. An insoluble precipitate was removed by centrifugation and washed with water. By the addition of 8 volumes of ethanol to the combined water solutions, the barium aldose-1-phosphate was precipitated. It was filtered and washed with ethanol and ethyl ether.

Potassium α-D-Glucose-1-phosphate.—The barium glucose-1-phosphate obtained (350 mg.) was dissolved in water. The solution was filtered and 155 mg. of potassium sulfate was added. The barium sulfate was removed by centrifugation. By the dropwise addition of 1.5 volumes of ethanol at 0°, potassium glucose-1-phosphate precipitated as fine needles (200 mg.). For analysis the product was recrystallized in the same manner, $[\alpha]^{26}\text{D}$ +78 ± 1° (*c* = 0.81, water).[8]

Anal. Calcd. for $C_6H_{11}O_9PK_2 \cdot 2H_2O$: total P, 8.33; labile P, 8.33. Found: total P, 8.16; labile P, 8.12.[10]

The α-glucose-1-phosphate sample obtained by this procedure was converted enzymatically by phosphoglucomutase to the same equilibrium mixture (containing 95% glucose-6-phosphate) as is the naturally occurring 1-ester.[11]

Potassium α-D-Galactose-1-phosphate.—The barium salt obtained (560 mg.) contained: total P, 6.54; labile P, 6.50.

Since the potassium salt of galactose-1-phosphate did not crystallize without seeding, the substance was at first purified as the crystalline brucine salt. To a water solution of 480 mg. of the barium salt 1 g. of brucine sulfate was added. The suspension was heated; barium sulfate was removed by centrifugation from the hot mixture and was washed with hot water. The combined supernatant fluids were concentrated *in vacuo* to 5 ml. Upon the addition of 15 ml. of acetone, the brucine salt crystallized (900 mg.). This salt was suspended in 3 ml. of water and 10% potassium hydroxide was added until a permanent pink color was obtained with phenolphthalein. Brucine was removed by exhaustive extraction with chloroform. During slow addition of ethanol at 0°, potassium α-D-galactose-1-phosphate crystallized in fine needles (240 mg.); $[\alpha]^{32}\text{D}$ +100 ± 1° (*c* = 1.57, water).[12]

Anal. Calcd. for $C_6H_{11}O_9PK_2 \cdot 2H_2O$: total P, 8.33; labile P, 8.33. Found: total P, 8.26; labile P, 8.22.

In another experiment, 490 mg. of the barium salt was treated directly with potassium sulfate in the usual manner for transformation into the potassium salt. The latter salt crystallized upon seeding (295 mg.); it was analytically pure after two recrystallizations (220 mg.).

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[8] The rotation reported by Wolfrom, *et al.*,[9] is $[\alpha]^{20}\text{D}$ +78°.

[9] M. L. Wolfrom and D. E. Pletcher, THIS JOURNAL, **63**, 1050 (1941).

[10] Labile P was determined as the amount of inorganic P formed during ten minutes of hydrolysis in 1 *N* HCl at 100°.

[11] This experiment was carried out by Dr. E. W. Sutherland.

[12] The rotation indicated by Kosterlitz[7] is $[\alpha]^{18}\text{D}$ +98°.

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Vapor Density of Diborane

BY E. B. RIFKIN AND G. W. THOMSON

It is of interest to compare data on unusual compounds with generalized correlations based largely on hydrocarbons and their derivatives. Such a comparison is afforded by recent vapor density data on diborane by Smith and Miller[1] which may be compared with generalized $P-V-T$ relations.[2,3,4] These have been found to be a fair approximation for hydrocarbons and a number of other substances (A, Ne, H_2, N_2, He, Kr, O_2,

TABLE I

VAPOR DENSITY OF DIBORANE (UNSATURATED VAPOR)

Temp.,[a] °K.	Pressure, p. s. i. a.	Density, g./cc.	T_R	P_R	$z = PV/RT$ Obsd.[1]	Gen.[b]	Berth.[c]
243.6	85	0.00873	0.841	0.145	0.92	0.89	..
243.6	131	.0148	.841	.224	.83	.83	0.86
243.6	172	.0332	.841	.294	.49[d]	.77	.82
243.6	173	.0392	.841	.295	.42[d]	.77	..
250.0	57	.00522	.864	.097	1.00	.94	..
250.0	71	.00669	.864	.121	0.97	.92	..
250.0	125	.0133	.864	.213	.86	.86	..
250.0	178	.0220	.864	.304	.74	.79	..
250.0	183	.0214	.864	.312	.78	.78	..
250.0	208	.0397	.864	.355	.48[d]	.75	..
256.1	64	.00583	.885	.109	0.98	.93	..
256.1	68	.00610	.885	.116	1.00	.93	..
256.1	131	.0136	.885	.224	0.86	.86	.88
256.1	198	.0232	.885	.338	.77	.77	..
256.1	243	.0359	.885	.415	.61[d]	.71	.78
268.1	109	.00943	.926	.186	.99	.90	..
268.1	181	.0187	.926	.309	.83	.83	..
268.1	276	.0326	.926	.471	.72	.72	..
268.1	324	.0427	.926	.553	.65	.65	..
277.8	118	.0108	.960	.201	.90	.91	..
277.8	153	.0138	.960	.261	.92	.88	..
277.8	298	.0345	.960	.509	.71	.74	..
277.8	420	.0604	.960	.717	.58	.58	..

[a] Data at 284.1, 287.1 and 292.5°K. are not shown since generalized correlations are not considered to be reliable at such high values of T_R (0.981, 0.992, 1.010). [b] Generalized $P-V-T$ correlation.[2,3] [c] Berthelot equation. [d] These points show greatest divergence from generalized correlation but agree with data on the saturated gas.

TABLE II

VAPOR DENSITY OF DIBORANE (SATURATED VAPOR)

Temp., °K.	Pressure,[a] mm.	Density, g./cc.	T_R	P_R	$z = PV/RT$ Obsd.[1]	Gen.[b]
207.0	2531	0.0151	0.715	0.0836	0.36	0.91
215.5	3569	.0145	.744	.118	.51	.88
227.9	5626	.0673	.787	.186	.16	.83
235.4	7238	.0348	.813	.239	.39	.79
240.3	8461	.0407	.830	.279	.38	.77
246.2	10130	.0382	.850	.335	.48	.74
251.3	11750	.0393	.868	.388	.53	.71
255.3	13150	.0470	.882	.434	.49	.69
260.8	15260	.0492	.901	.504	.53	.66
265.8	17370	.0541	.918	.574	.54	.62
269.9	19260	.0567	.932	.636	.56	.59
270.7	19640	.0588	.935	.649	.55	.59
274.0	21290	.0625	.946	.703	.55	.56
278.9	23890	.0581	.963	.789	.66	.52
283.0	26240	.0759	.978	.867	.54	.47
285.2	27560	.0927	.985	.910	.46	.45
286.2	28180	.0950	.989	.931	.46	.40
288.2	29430	.1100	.996	.972	.41	.40

[a] Calculated from vapor pressure equation. [b] Generalized $P-V-T$ correlation.[2,3]

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[1] S. H. Smith, Jr., and R. R. Miller, THIS JOURNAL, **72**, 1452 (1950).

[2] J. Q. Cope, W. K. Lewis and H. C. Weber, *Ind. Eng. Chem.*, **23**, 887 (1931).

[3] G. W. Thomson, *ibid.*, **35**, 895 (1943).

[4] R. H. Newton, *ibid.*, **27**, 302 (1935).

CO_2, NH_3, H_2O, SO_2, $(C_2H_5)_2O$, CH_3Cl, CF_2Cl_2, NO, CH_3OH, C_2H_5OH). Two sets of data have been presented on diborane, saturated gas densities in their Table III[1] said to be accurate to ±3%, and unsaturated gas densities (obtained in a different apparatus) in their Table V, said to be accurate to ±1%. For convenience in comparison both these sets of data are presented in the form of compressibility factors ($z = PV/RT$ per mole) in Tables I and II. For the saturated gas densities the pressure was computed from their vapor pressure equation

$$\log P\ (\text{mm.}) = 7.185 - 782.8/(t + 273.2)$$

where t is °C. Figure 1 is a plot of z *versus* reduced pressure $P_R = P\ (\text{mm.})/30300$.

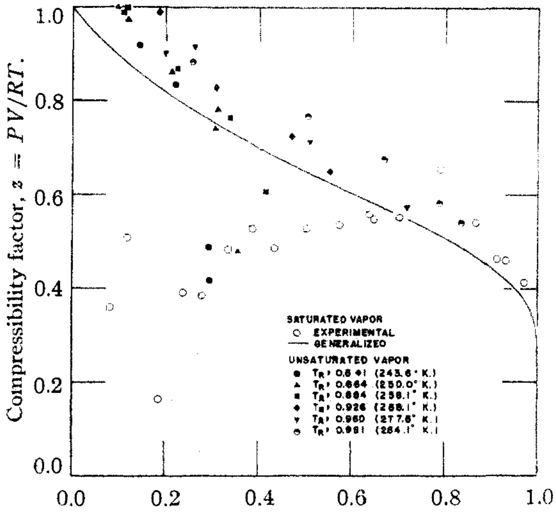

Fig. 1.—Compressibility of gaseous diborane.

It is at once evident from Fig. 1 that the data are considerably less precise than was reported; the scatter of the points for the saturated gas is quite wide, especially at the lower pressures. Another feature is the marked difference between these values for the saturated gas and those from the usual generalization below the critical point[2,3] shown as a solid line in the figure. This correlation indicates that z should decrease continuously from 1 at $P_R = 0$ to 0.70 at $P_R = 0.4$ to about 0.27 at $P_R = 1$. In contrast with this the observed results approach very low values of z at $P_R = 0$ and rise to a maximum around $P_R = 0.6$. At pressures above $P_R = 0.6$ the data and the generalized curve are in much better agreement than at the lower pressures.

The unsaturated vapor densities (except for a few values near the saturation line) are slightly lower (by 0 to 10%, average 2%) than the predicted values from the generalized correlation (Table I). The use of the Berthelot equation gives about the same results as the generalized correlation. A few examples are given in Table I.

In their pioneer studies on diborane, Stock and Kuss[5] made one measurement of vapor density: 0.0808 g. occupied 128.8 cc. at 17.3° and 405.7 mm. This corresponds to $z = 0.989$ at $T_R = 1.003$ and $P_R = 0.0134$ which is in excellent agreement with the generalized correlation (0.995) and indicates the absence of association in the vapor phase at this pressure.

It would be of interest to determine whether diborane is an exception to the generalizations as a result of some unexplained phenomenon or whether the saturated gas densities of Smith and Miller are in error.

(5) A. Stock and E. Kuss, *Ber.*, **56B**, 789 (1923).

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Experiments with Xanthotoxin and Imperatorin Obtained from the Fruits of *Ammi majus* (L.)

BY ALEXANDER SCHÖNBERG AND ALY SINA

The fruits of *Ammi majus* (L.) were described as a remedy for leukodermia in old Arabic literature.[1,2] The drug was also mentioned by Dragendorff.[3] In Egypt, the public takes the powdered fruits by mouth and then the leukodermic patches are exposed to sunlight.

Fahmy and Abu-Shady isolated[4,5] two crystalline principles from the fruits of *Ammi majus* which they named "Ammoidin" m. p. 148° and "Ammidin" m. p. 102°. We wish to thank them for allowing us to elucidate the nature of these compounds. As already stated in a previous note,[6] it was found that ammoidin and ammidin are identical with xanthotoxin[7] (Ib) and imperatorin[8] (II), respectively. In this paper, the experimental details leading to their identification are given.

Experiments with Xanthotoxin (Ib).—The demethylation of xanthotoxin was effected by heating with magnesium iodide in the absence of a solvent (compare the demethylation of khellin),[9] leading to the production of 8-hydroxy-4′,5′-6,7-furo-coumarin (Ia), also known as xanthotoxol.[10] (Ia) was converted back into xanthotoxin by the potassium carbonate–methyl iodide method; by a similar procedure the ethyl, *n*-pro-

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(1) Ibn El-Bitar, "Mofradat El-Adwia," Vol. I, p. 4.

(2) Dawood El-Antaki, "Tazkaret Oli El-Albab," 3rd edition, Vol. I, p. 32 (1923).

(3) "Die Heilpflanzen der verschieden Völker und Zeiten," Stuttgart, 1898, p. 488.

(4) Fahmy, Abu-Shady, Schönberg and Sina, *Nature*, **160**, 468 (1947).

(5) Fahmy and Abu-Shady, *Quarterly J. Pharmacy and Pharmacology*, **20**, 281 (1947); *ibid.*, **21**, 499 (1948).

(6) Schönberg and Sina, *Nature*, **161**, 481 (1948).

(7) Thoms, *Ber.*, **44**, 3325 (1911); Späth and Pailer, *ibid.*, **69**, 767 (1936).

(8) Späth and Holzen, *ibid.*, **68**, 1123 (1935).

(9) Schönberg and Sina, THIS JOURNAL, **72**, 3396 (1950).

(10) Späth and Vierhapper, *Ber.*, **70**, 248 (1937).