with water, and dried over anhydrous sodium sulfate. Removal of the solvent gave 20.1 g. of crude product. This was distilled through a three-inch Vigreux column and the fraction boiling from 74–92° (17 mm.) (mostly 75° (17 mm.)) collected. The yield was 11.6 g. (70%). The residue weighed 3.6 g. On redistillation the b. p. was 76–79° (17 mm.). The boiling point and density (d^{27}_{25} 0.939) agree with literature values[4] obtained for the product prepared by different methods. Refractive index n^{21}_D 1.4929.

Attempted Asymmetric Syntheses.—Attempts were made to impart an asymmetric bias to the above reaction by conducting it, in the same manner, in the presence of various optically active agents. The quantities of reagents and results are given in Table I. The amount of phenylmagnesium bromide used in each case was sufficient to react completely with the optically active agent and the ethyl α-chloroethyl ether, plus a 100% excess of the quantity required for the latter reaction. All rotations were taken in 95% ethanol.

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[4] Beilstein, "Handbuch der organische Chemie," 4th ed., Vol. VI, p. 475, (236).

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7-Chloro-4-(1-diethylamino-4-pentylamino)-quinazoline

BY M. K. MCKEE, R. L. MCKEE[1] AND R. W. BOST

Our attention has been called to the preparation of 7-chloro-4-(1-diethylamino-4-pentylamino)-quinazoline by Price and co-workers.[2] We wish to record an alternative route for the preparation of this compound which had been worked out in our Laboratory when this article[2] appeared.

7-Chloro-4-hydroxyquinazoline.—A mixture of 19.5 g. of 4-chloro-anthranilamide,[3] 75 cc. of ethyl orthoformate, and 45 cc. of diethylene glycol was heated to 105–110° for three hours and to 120° for twenty-four hours. The resulting gel was warmed with dilute hydrochloric acid, made alkaline (sodium hydroxide), treated with Norite and filtered. The filtrate was acidified (acetic acid) and finally made faintly alkaline with ammonium hydroxide. The product was recrystallized from alcohol, giving 18.9 g. (87% yield) of white material melting at 250–253°. A small sample was sublimed at atmospheric pressure, giving a product melting at 251.5–253° for analysis.

Anal. Calcd. for $C_8H_5ClN_2O$: N, 15.51. Found: N, 15.52.

7-Chloro-4-(1-diethylamino-4-pentylamino)-quinazoline.—Five grams (0.028 mole) of the above quinazolone was refluxed with 6.3 g. (0.030 mole) of phosphorus pentachloride in 25 cc. of freshly distilled phosphorus oxychloride for twenty-four hours. After removal of excess solvent by distillation (water pump), 11 g. (0.07 mole) of 1-diethylamino-4-aminopentane was added and refluxed for two hours. Aqueous alkali was added, and the product was extracted with ether, dried over sodium hydroxide and distilled. The material boiling from 210–215° at 4.5 mm. was recrystallized from benzene–petroleum ether to yield 7.1 g. (80% yield based on the quinazolone) of white crystals melting at 117–118°.

Anal. Calcd. for $C_{17}H_{25}ClN_4$: N, 17.46. Found: N, 17.23.

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[1] The Wm. S. Merrell Co., Postdoctoral Fellow.

[2] Price, Leonard and Curtin, THIS JOURNAL, **68**, 1305 (1946).

[3] Hunn, *ibid.*, **45**, 1024 (1923).

Electron Deficient Molecules. III. The Entropy of Diborane

BY KENNETH S. PITZER

In order to see whether measurement of the entropy of diborane would contribute further definite evidence concerning its structure, the writer calculated the translational-rotational entropy for both ethanelike and protonated double bond[1] models using distances and angles given by Bauer.[2] Much to his surprise the difference in moments of inertia almost compensated the difference in symmetry number so that the total entropies differ hardly more than the expected experimental error.

This makes it unlikely that an entropy measurement would contribute significantly to the structural problem. For the same reason, the calculated entropy is reasonably certain even if one may doubt which structure is correct. Since this entropy value may be of interest in thermodynamic calculations, it seems worthwhile to present it at this time.

Table I itemizes the calculations for the two separate models. It should be noted that the experimental heat capacity data of Stitt[3] were used to compute the vibrational contribution. Thus no assumption was needed with respect to the actual vibrational assignment. Likewise, if there is an electronic contribution, it is included within that labelled vibrational. The estimates of error are based, respectively, on the uncertainties in bond distances and in the experimental heat capacities together with their extrapolation to a point where the vibrational contribution is negligible.

TABLE I

ENTROPY OF DIBORANE AT 298.16°K

Model	Ethane-like	Protonated double bond
Moments of inertia	1.44	1.08
× 10^{39} g. sq. cm.	5.67	4.73
	5.67	5.05
Symmetry no.	6	4
$S_{\text{trans.} + \text{rot.}}$	53.09 ± 0.1	53.31 ± 0.1
$S_{\text{vibr.}}$	2.15 ± .1	2.15 ± .1
S_{total} (cal. per degree mole)	55.24 ± .2	55.46 ± .2

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[1] K. S. Pitzer, THIS JOURNAL, **67**, 1126 (1945) (paper I of this series).

[2] S. H. Bauer, *Chem. Rev.*, **31**, 43 (1942); private communication reported by Longuet-Higgins and Bell, *J. Chem. Soc.* (London), 250 (1943).

[3] F. Stitt, *J. Chem. Phys.*, **8**, 981 (1940).