

**1-Butyl-3-pyrrolidone.**—Dieckmann ring closure using sodium ethoxide in xylene at 120°<sup>1</sup> followed by hydrolysis and decarboxylation gave the aminoketone in 73% yield; b.p. 38° (0.5 mm.);  $n_D^{20}$  1.4510;  $d_4^{20}$  0.918.

*Anal.* Calcd. for  $C_8H_{15}NO$ : C, 68.04; H, 10.71;  $MR_D$ , 41.06. Found: C, 67.81; H, 10.70;  $MR_D$ , 41.38.

The picrate of 1-butyl-3-pyrrolidone was obtained as fine yellow needles, m.p. 157.5–158°, with decomposition, after formation in ethanol and recrystallization from methanol.

*Anal.* Calcd. for  $C_{14}H_{18}N_4O_8$ : C, 45.41; H, 4.90. Found: C, 45.33; H, 5.17.

**Ethyl  $\beta$ -N-(2-Carboxypyrrolidyl)-butyrate.**—A solution of 25 g. (0.175 mole) of ethylproline and 100 g. (0.877 mole) of ethyl crotonate was heated at 100° for 48 hours.<sup>9</sup> The excess ethyl crotonate was removed by distillation at atmospheric pressure and the diester was obtained as a colorless liquid, b.p. 99–100° (0.3 mm.);  $n_D^{20}$  1.4566;  $d_4^{20}$  1.064; yield 13.8 g. (31%).

*Anal.* Calcd. for  $C_{18}H_{28}NO_4$ : C, 60.69; H, 8.98; N, 5.44. Found: C, 60.52; H, 8.96; N, 5.62.

**1-Keto-3-methylpyrrolizidine.**—The procedure of McElvain and Rorig<sup>11</sup> employing sodium hydride was used for the Dieckmann ring closure of ethyl  $\beta$ -N-(2-carboxypyrrolidyl)-butyrate. The aminoketone obtained in 50% yield after hydrolysis and decarboxylation was converted directly and no attempt was made to isolate both of the possible racemates. The hydrochloride was hygroscopic. The picrate, prepared in ether and recrystallized from methanol, separated as long yellow needles, m.p. 189–190°, with decomposition.

*Anal.* Calcd. for  $C_{14}H_{16}N_4O_8$ : C, 45.65; H, 4.37; N, 15.21. Found: C, 45.79; H, 4.43; N, 15.24.

The picrolonate, prepared in ether and recrystallized from ethanol, formed orange leaflets, m.p. 206–207°, with decomposition.

*Anal.* Calcd. for  $C_{18}H_{22}N_6O_8$ : C, 53.59; H, 5.25; N, 17.36. Found: C, 53.86; H, 5.32; N, 17.07.

**Wolff-Kishner Reduction of 1-Keto-3-methylpyrrolizidine. 3-Methylpyrrolizidine.**—A solution of 0.20 g. of 1-keto-3-methylpyrrolizidine, 1.0 g. of hydrazine hydrate (85%), and 1.0 g. of potassium hydroxide in 10 ml. of triethylene glycol was boiled under reflux for one hour. The solution was then distilled until the distillate was no longer basic. The distillate was saturated with potassium carbonate and extracted with ether. The ethereal solution was dried and treated with picric acid in ether. The picrate thus obtained crystallized from methanol as yellow elongated plates, m.p. 251–252°, with decomposition.

*Anal.* Calcd. for  $C_{14}H_{18}N_4O_7$ : C, 47.46; H, 5.12; N, 15.81. Found: C, 47.62; H, 5.12; N, 15.83.

The Clemmensen reduction of 1-keto-3-methylpyrrolizidine gave results which were inconclusive, however; the crude product yielded a small amount of  $C_8H_{15}N$  picrate, m.p. 251–252° (dec.), which was found to be identical with the 3-methylpyrrolizidine picrate described above by mixed melting point and infrared absorption spectra determinations.

(11) S. M. McElvain and K. Rorig, *THIS JOURNAL*, **70**, 1820 (1948).

THE NOYES CHEMICAL LABORATORY  
UNIVERSITY OF ILLINOIS  
URBANA, ILL.

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### The Optical Rotation of 2-Chlorobutane

BY ROBERT L. LETSINGER, LUCIEN G. MAURY AND ROBERT L. BURWELL, JR.

We have determined limiting values for the rotation of optically pure 2-chlorobutane by means of a displacement reaction with benzylsodium. The use of this reagent was suggested by the characteristics of its reaction with (+)-2-bromobutane.<sup>1</sup>

Benzylsodium and 2-chlorobutane ( $\alpha_D^{25}$   $-10.93^\circ$ ) combined to give 1-phenyl-2-methylbutane with  $\alpha_D^{25}$   $+3.26^\circ$ . On the basis of  $\alpha_D^{25}$

(1) R. L. Letsinger, *THIS JOURNAL*, **70**, 406 (1948).

$+9.9^\circ$  ( $[\alpha]_D^{25} +11.6^\circ$ ) as the maximum rotation for this hydrocarbon,<sup>1</sup> therefore,  $\alpha_D^{25}$   $33.2^\circ$  is an upper limit for the rotation of optically pure 2-chlorobutane. The actual value may be somewhat lower since  $9.9^\circ$  is probably a little high for the hydrocarbon and some loss of asymmetry may have occurred in the benzylsodium condensation.

Of reported cases, the preparation of 2-chlorobutane which involved the least racemization was the conversion of 2-butanol ( $[\alpha]_D^{25} -5.54^\circ$ ) to 2-chlorobutane ( $[\alpha]_D^{25} +13.70^\circ$ ).<sup>2</sup> Calculation of these data to the basis of optically pure alcohol ( $[\alpha]_D^{25}$   $13.64^\circ$ )<sup>3</sup> gives  $\alpha_D^{25}$   $29.2^\circ$  (or  $[\alpha]_D^{25}$   $33.8^\circ$ ) as a rotation of 2-chlorobutane experimentally obtainable. The rotation for the pure enantiomorph must therefore lie between  $\alpha_D^{25}$   $29.2^\circ$  and  $33.2^\circ$ .

### Experimental

The symbol,  $\alpha_D^{25}$ , designates the homogeneous rotation in a 1 dm. tube.

**2-Chlorobutane.**—2-Butanol (5.66 g.,  $\alpha_D^{25}$   $+7.48^\circ$ ,  $n_D^{25}$  1.3944) was saturated with hydrogen chloride gas at zero degrees. The solution was then sealed in a tube and heated at 75° for 72 hours, during which time it separated into two layers. After cooling, the tube was opened and the contents washed successively two times with water, three times with concentrated hydrochloric acid, three times with concentrated sulfuric acid, and finally three times again with water. The 2-chlorobutane thus obtained was dried over calcium chloride; 6.8 g. product (96%);  $\alpha_D^{25}$   $-12.24^\circ$ ,  $n_D^{25}$  1.3942. Two samples prepared in this manner were combined to give a product with  $\alpha_D^{25}$   $10.93^\circ$ ;  $n_D^{25}$  1.3942.

**1-Phenyl-2-methylbutane.**—Butylsodium was prepared as previously described<sup>1</sup> from *n*-butyl chloride (23.1 g., 0.25 mole) and sodium (11.5 g., 0.5 g. atom) with hexane as a solvent. To ensure complete reaction of the halide, the butylsodium was stirred two hours at 25° and then permitted to stand at room temperature for 20 hours. At the end of this time, toluene (160 cc.) was added and the mixture stirred for an additional six hours at 25°. Metalation was comparatively rapid as evidenced by the change from blue to green in less than ten minutes, and then to yellow in about half an hour.

A solution of 2-chlorobutane (8.67 g., 0.0937 mole,  $\alpha_D^{25}$   $-10.93^\circ$ ) in 10 cc. of hexane was added over a period of five minutes to the rapidly stirred suspension of benzylsodium, which was maintained at 15°. After an additional ninety minutes of stirring, the mixture was carbonated on powdered Dry Ice. The hydrocarbon portion was separated by conventional procedures and the 1-phenyl-2-methylbutane isolated by distillation; 7.63 g. (55%); b.p. 90–91° (27 mm.);  $\alpha_D^{25}$   $+3.26^\circ$ ,  $d_4^{25}$  0.8544,  $n_D^{25}$  1.4848.

(2) J. F. Lane and S. E. Ulrich, *ibid.*, **72**, 5132 (1950).

(3) R. H. Pickard and J. Kenyon, *J. Chem. Soc.*, **99**, 45 (1911). This value was interpolated from rotations measured at 20° and 50°.

DEPARTMENT OF CHEMISTRY  
NORTHWESTERN UNIVERSITY  
EVANSTON, ILLINOIS

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### An Analysis of the Electron Diffraction Data for Decaborane

BY CHARLYS M. LUCHT

Silbiger and Bauer<sup>1</sup> have presented their electron diffraction data for the molecular structure of decaborane,  $B_{10}H_{14}$ . They had tried all the molecular models which had been proposed in the literature up to that time and also calculated many others which might have been possible. The arrangement of planar hexagons suggested by Pitzer<sup>2</sup> and several

(1) C. Silbiger and S. H. Bauer, *THIS JOURNAL*, **70**, 115 (1948).

(2) K. S. Pitzer, *ibid.*, **67**, 1136 (1945).