

**Procedure.**—Furoyl chloride was prepared by heating one mole (112 g.) of the acid<sup>1</sup> at 110–120° with a 15% excess of thionyl chloride until the evolution of hydrogen chloride ceased. The excess thionyl chloride was removed at the water pump and the residue distilled *in vacuo* to yield 90% (118 g.) of furoyl chloride boiling at 90° (20 mm.).

The benzyl and cyclohexyl esters were prepared by fluxing 0.5 mole (65 g.) of the acid chloride with 0.5 mole of the alcohol until evolution of hydrogen chloride ceased. The ester was purified by distillation *in vacuo*. The *t*-butyl ester was prepared by the general method in Organic Syntheses.<sup>2</sup>

TABLE I

Esters	B. p.,		Yield, %	$n_D^{20}$	For- mula	Sapon. equiv.	
	°C.	Mm.				Calcd.	Found
<i>t</i> -Butyl	90	24	20	1.4639	C <sub>9</sub> H <sub>12</sub> O <sub>2</sub>	168	167
Benzyl	141–142	2	41	1.5505	C <sub>12</sub> H <sub>16</sub> O <sub>2</sub>	202	203
Cyclohexyl <sup>a</sup>	122–124	2	49	1.4499	C <sub>11</sub> H <sub>14</sub> O <sub>2</sub>	194	194

<sup>a</sup> M. p. 32–33°.

Consistent analytical results on the benzyl ester could be obtained only by the method of Maglio.<sup>3</sup> The other esters gave no trouble on saponification.

Incidental to the preparation of the acid chloride, it was observed that the reaction mixture hydrolyzed very rapidly if moisture was not excluded. This phenomenon is in contrast to the pure halide which is hydrolyzed only very slowly by boiling water. Hartman and Dickey<sup>4</sup> call attention to the varying yields reported in the literature, and state that this may be due to impurities in the furoic acid and/or reagents used. Our observation would indicate that these impurities catalyze the hydrolysis and that the previously reported low yields may have been due to failure to exclude moisture.

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(1) The commercial 2-furoic acid generously supplied by the Quaker Oats Co. was used without further purification.

(2) "Organic Syntheses," **24**, 19 (1944).

(3) Maglio, *Chem. Analyst*, **25**, 39 (1946).

(4) Hartman and Dickey, *Ind. Eng. Chem.*, **24**, 151 (1932).

### *p*-Ethoxyphenylglyoxal

Eighty-two grams (0.5 mole) of *p*-ethoxyacetophenone in a mixture of 300 ml. of dioxane and 11 ml. of water was oxidized with 56 g. (0.5 mole) of selenium dioxide according to the method of Riley and Gray.<sup>1</sup> After working up in the usual manner, the product was fractionated through a 20-cm. Vigreux column at 103–105° (4 mm.) to give 38 g. (40.5%) of a yellow oil. The monosemicarbazone was prepared according to the directions of Shriner and Fuson.<sup>2</sup> The derivative could be crystallized from aqueous ethanol, m. p. 206–207° (dec.).<sup>3</sup>

*Anal.* (by Arlington Laboratories, Fairfax, Virginia). Calcd. for C<sub>11</sub>H<sub>13</sub>N<sub>3</sub>O<sub>2</sub>: C, 56.16; H, 5.57. Found: C, 56.56; H, 5.78.

(1) Riley and Gray, "Organic Syntheses," Coll. Vol. II, John Wiley and Sons, Inc., New York, N. Y., 1943, p. 509.

(2) Shriner and Fuson, "The Systematic Identification of Organic Compounds," John Wiley and Sons, Inc., New York, N. Y., 1946, p. 142.

(3) Melting points were taken with a Fisher–Johns apparatus.

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### 2-(*p*-Ethoxyphenyl)-quinoxaline

Two and three-tenths grams (0.01 mole) of *p*-ethoxyphenylglyoxal<sup>1</sup> in 25 ml. of glacial acetic acid was mixed with 2.2 g. of *o*-phenylenediamine, and treated according to the method of Fuson, *et al.*<sup>2</sup> The product was recrystallized twice from aqueous ethanol to give crystals melting at 128°.

*Anal.* (by Arlington Laboratories, Fairfax, Virginia). Calcd. for C<sub>16</sub>H<sub>14</sub>N<sub>2</sub>O: C, 76.78; H, 5.64; N, 11.19. Found: C, 76.39; H, 5.56; N, 11.57.

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(1) Kipnis, Soloway and Ornfelt, *THIS JOURNAL*, **69**, 1231 (1947).

(2) Fuson, Emerson and Gray, *ibid.*, **61**, 482 (1939).

## COMMUNICATIONS TO THE EDITOR

### THE CRYSTAL STRUCTURE OF LITHIUM BOROHYDRIDE LiBH<sub>4</sub>

Sir:

As a consequence of the remarks of Dr. H. C. Brown to one of us concerning the interest in the structure of lithium borohydride<sup>1</sup> and its commercial availability, we undertook an X-ray diffraction investigation of its crystal structure and are now making the following preliminary report of the results obtained.

Experimentally, the density was found to be 0.66 g./cc. indicating the presence of four molecules per unit cell. Data obtained from rotation

(1) For a description of the properties of LiBH<sub>4</sub>, see Schlesinger and Brown, *THIS JOURNAL*, **62**, 3429 (1940). The material used was purchased from the Lithalloys Corporation, 444 Madison Ave., New York, N. Y.

and oscillation photographs using CuK $\alpha$  radiation show that the unit cell of lithium borohydride is orthorhombic with the dimensions  $a_0 = 6.81$ ,  $b_0 = 4.43$  and  $c_0 = 7.17$  kX. The density calculated from these cell dimensions is 0.666 g./cc.

An examination of the extinctions indicates that the arrangement of lithium and borohydride ions satisfies the symmetry of the space group Pcmn. Intensity calculations show that the hydrogens can make an appreciable contribution to the intensities and that a tetrahedral borohydride ion appears to be compatible with the data.

The spatial arrangement is such that each lithium ion is associated with four borohydride ions. Two of the lithium ions are separated from the boron by 2.56 kX and the other two by 2.47 kX.