### Experimental[9]

**1-(2-Deoxy-5-*O*-trityl-β-D-lyxosyl)uracil (II).**—To a cooled solution of 1.73 g. (3.68 mmoles) of 5'-*O*-trityl-2'-deoxyuridine[10] (I) in 10 ml. of dry pyridine was added 0.57 ml. (7.36 mmoles) of methanesulfonyl chloride and the mixture held at 5° for 16 hr. Water (2.0 ml) was added and, after 0.5 hr. at room temperature, the reaction mixture was poured into 500 ml. of stirred ice–water. The off-white solid was collected and washed with generous quantities of water. The air-dried product was dissolved in 80 ml. of 50% ethanol that contained 14.5 ml. of *N* sodium hydroxide and the solution was refluxed for 4 hr. The volume was then reduced *in vacuo* to *ca.* 40 ml.; the reaction mixture was then chilled and carefully acidified (pH 2) with dilute hydrochloric acid. The gelatinous product was collected, washed with water, and sucked dry. The dry product was readily transformed to a crystalline solid on stirring with 50 ml. of ethanol at room temperature for 0.5 hr., wt. 1.64 g. (95% yield), m.p. 225–228° Two recrystallizations from ethanol provided an analytical sample, m.p. 239–240°, $[\alpha]^{25}D$ −14.9° (*c* 0.93, DMF); $\lambda_{max}^{EtOH}$ 262 mμ (ε 10,630), and $\lambda_{min}$ 243 mμ (ε 6370).

*Anal.* Calcd. for $C_{28}H_{26}N_2O_5$: C, 71.47; H, 5.57; N, 5.96. Found: C, 71.30; H, 5.73; N, 5.69.

**1-(2-Deoxy-β-D-lyxofuranosyl)uracil (III).**—A solution of 1.45 g. (3.08 mmoles) of II in 10 ml. of 80% acetic acid was refluxed for 10 min. The reaction mixture was evaporated to dryness *in vacuo* and the residue was evaporated from three 10-ml. portions of ethanol. The product crystallized from methanol–ethyl acetate, 0.58 g. (two crops, 83% yield), m.p. 163–165°. A second recrystallization from methanol failed to alter the melting point; $[\alpha]^{25}D$ +58.2 (*c* 0.55, ethanol); in $H_2O$, $\lambda_{max}$ 263 mμ (ε 10,620), and $\lambda_{min}$ 232 mμ (ε 2760); 0.1 *N* HCl, $\lambda_{max}$ 262 mμ (ε 9610), and $\lambda_{min}$ 231 mμ (ε 2220); 0.1 *N* NaOH, $\lambda_{max}$ 262 mμ (ε 7070), add $\lambda_{min}$ 241 mμ (ε 4520).

*Anal.* Calcd. for $C_9H_{12}N_2O_5$: C, 47.36; H, 5.30; N, 12.28. Found: C, 47.22; H, 5.40; N, 12.09.

**1-(2-Deoxy-β-D-lyxofuranosyl)-5-iodouracil (IV).**[11]—A mixture of 0.25 g. (1.1 mmoles) of III, 0.25 g. (1 mmole) of iodine, 2.5 ml. of *N* nitric acid, and 1.3 ml. of chloroform was refluxed for 2 hr. On cooling, a colorless crystalline solid was deposited. The product was collected, washed free of iodine with ether, and recrystallized from water, 0.23 g. (57% yield), m.p. 180–181° dec., $[\alpha]^{25}D$ −4.9° (*c* 0.81, ethanol); in $H_2O$, $\lambda_{max}$ 289 mμ (ε 6530) and $\lambda_{min}$ 247 mμ (ε 970); 0.1 *N* HCl, $\lambda_{max}$ 288 (ε 4330), and 255 mμ (ε 2010); 0.1 *N* NaOH, $\lambda_{max}$ 278 mμ (ε 5310) and $\lambda_{min}$ 254 mμ (ε 2830).

*Anal.* Calcd. for $C_9H_{11}IN_2O_5$: C, 30.52; H, 3.13; I, 35.84; N, 7.91. Found: C, 30.28; H, 2.88; I, 35.70; N, 8.09.

(9) Melting points are corrected. Ultraviolet spectra were recorded by a Cary Model 11 spectrophotometer. Analyses were performed by Micro-Tech Laboratories, Skokie, Ill.

(10) J. Smrt and F. Sorm, *Collection Czech. Chem. Commun.*, **25**, 553 (1960); *Chem. Abstr.*, **54**, 12145 (1960).

(11) This procedure is identical with that described by W. H. Prusoff, *Biochim. Biophys. Acta*, **32**, 295 (1959), for the preparation of 5-iodo-2'-deoxyuridine.

## 5-Aryl-1,3-dihydro-2*H*-1,3,4-benzotriazepin-2-ones

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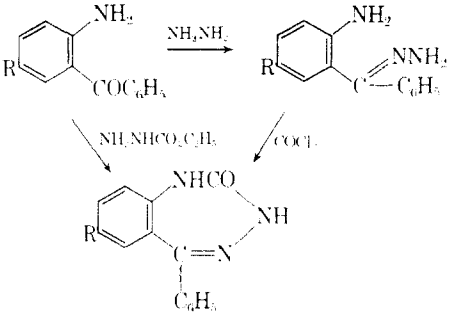
Because of our interest in 1,4-benzodiazepin-2-ones[1] and 3,1,4-benzoxadiazepin-2(1*H*)ones,[2] it seemed desirable to prepare some aza analogs of these ring systems. Accordingly, several 5-aryl-1,3-dihydro-2*H*-1,3,4-benzotriazepin-2-ones have been prepared by two related methods, as shown in the reaction scheme.




### Experimental

**5-Phenyl-1,3-dihydro-2*H*-1,3,4-benzotriazepin-2-one.**—A solution of 12 g. of 12.5% phosgene in benzene was added dropwise to a cooled solution of 3.2 g. of 2-aminobenzophenone hydrazone and 5 ml. of triethylamine in 50 ml. of benzene. After addition was completed, the mixture was stirred at room temperature for 1 hr. After separating the triethylamine hydrochloride by filtration, the solution was evaporated to dryness *in vacuo*. Recrystallization of the residue from ethanol afforded 1.4 g. of product, m.p. 238°.

*Anal.* Calcd. for $C_{14}H_{11}N_3O$: C, 70.87; H, 4.68; N, 17.70. Found: C, 70.69; H, 4.53; N, 18.04.

**7-Methyl-5-phenyl-1,3-dihydro-2*H*-1,3,4-benzotriazepin-2-one,** m.p. 253–255°, was prepared similarly from 2-amino-5-methylbenzophenone hydrazone in a yield of 50%.

*Anal.* Calcd. for $C_{15}H_{13}N_3O$: C, 71.70; H, 5.21; N, 16.72. Found: C, 71.61; H, 5.43; N, 16.64.

**7-Chloro-5-phenyl-1,3-dihydro-2*H*-1,3,4-benzotriazepin-2-one.**—A mixture of 5 g. of 2-amino-5-chlorobenzophenone and 5 ml. of ethyl hydrazinecarboxylate was heated at 190° for 1 hr. The mixture was cooled and dissolved in 75 ml. of ethanol. On standing there was obtained 1.6 g. of product, m.p. 246–248°.

*Anal.* Calcd. for $C_{14}H_{10}ClN_3O$: C, 61.89; H, 3.72; Cl, 13.05; N, 15.47. Found: C, 61.65; H, 3.72; Cl, 13.27; N, 15.18.

Concentration of the mother liquor afforded 0.5 g. of 2-amino-5-chlorobenzophenone hydrazone ethyl carboxylate, m.p. 209°.

*Anal.* Calcd. for $C_{16}H_{16}ClN_3O_2$: C, 60.47; H, 5.07; Cl, 11.16; N, 13.23. Found: C, 60.16; H, 5.07; Cl, 11.25; N, 13.04.

(1) S. C. Bell, T. S. Sulkowski, C. Gochman, and S. J. Childress, *J. Org. Chem.*, **27**, 562 (1962).

(2) T. S. Sulkowski and S. J. Childress, *ibid.*, **27**, 4424 (1962).

## Quinazolines and 1,4-Benzodiazepines. XIX.[1] N-Alkyl Derivatives of Substituted 1,3,4,5-Tetrahydro-5-phenyl-2*H*-1,4-benzodiazepin-2-ones

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As a continuation of our investigation on psychotherapeutic agents of the 1,4-benzodiazepine class of compounds, we have prepared a series of 1,3,4,5-tetrahydro-5-phenyl-2*H*-1,4-benzodiazepin-2-ones and from these compounds a number of N-alkyl derivatives. For the sake of simplicity, the Experimental section of this paper will concern itself with the chemistry of only one of these compounds, namely, 7-chloro-5-(2-fluorophenyl)-1,3,4,5-tetrahydro-2*H*-1,4-benzodiazepin-2-one and its N-methyl derivatives. As shown in the Experimental, because of the difference in basicity between the two nitrogen atoms, we found it possible to alkylate the 1-nitrogen independently of the 4-nitrogen and *vice versa*. All other compounds and derivatives prepared by the same procedures will be found in Table III.

(1) Paper XVIII: L. H. Sternbach, E. Reeder, A. Stempel, and A. I. Rachlin, *J. Org. Chem.*, **29**, 332 (1964).