7.94 (s, 3 H, C_7-CH_3), 7.80 (s, H, C_3-CH_3), 6.91 (s, 3 H, SO_2-CH_3), 4.28 (br s, 1 H, C_6-H), 3.83 (br s, 1 H), C_4-H); mass spectrum *m/e* (rel intensity) 214 (19, M^+), 135 (100, $M^+ - CH_2SO_2\cdot$), 121 (19, $M^+ - CH_3SO_2N$), 106 (25), 39 (12).

Anal. Calcd for $C_9H_{14}N_2O_2S$: C, 50.45; H, 6.59. Found: C, 50.60; H, 6.59.

Elution with methylene chloride gave methanesulfonamide (60 mg, 12.5%). Elution with chloroform–methanol gave starting ylide (172 mg, 16%).

Photolysis of 1-Benzenesulfonylimino-2-methylpyridinium Ylide (16).—This was carried out in benzene–acetonitrile (6:1 v/v) to give the diazepine **17** (54.1%): mp 118.5–119°; nmr $\tau_{TMS}^{CDCl_3}$ 8.00 (s, 3 H, C_8-CH_3), 4.36 (dq, $J_{6,7}$ = 7.0 Hz, $J_{5,6}$ = $J_{6,7}$ = 3.5 Hz, $J_{4,6}$ = 0.5 Hz, 1 H, C_6-H), 4.04 (dd, $J_{6,7}$ = 7.0 Hz, $J_{5,7}$ = 0.5 Hz, 1 H, C_7-H), 3.58 (d, $J_{5,6}$ = $J_{4,5}$ = 3.5 Hz, 2 H, C_4-H and C_5-H), 2.50 (m, $J_{\alpha,\beta}$ = $J_{\alpha',\beta'}$ = 8 Hz, 3 H, C_β-H, $C_{\beta'}$-H, C_α-H), 2.04 (dd, $J_{\alpha,\beta}$ = $J_{\alpha',\beta'}$ = 8.0 Hz, $J_{\alpha,\gamma}$ = $J_{\alpha',\gamma}$ = 2.0 Hz, 2 H, C_α-H, $C_{\alpha'}$-H); mass spectrum *m/e* (rel intensity) 248 (7, M^+), 107 (100, $M^+ - PhSO_2\cdot$), 93 (21, $M^+ - PhSO_2N$), 77 (70, Ph^+), 39 (37).

Anal. Calcd for $C_{12}H_{12}N_2O_2S$: C, 58.05; H, 4.87. Found: C, 58.33; H, 5.08.

Benzenesulfonamide (5.2%), mp 153–155°, and starting ylide (31%) were also isolated.

Photolysis of 1-Benzenesulfonylimino-3,5-dimethylpyridinium Ylide (18).—This photolysis was effected on a benzene–methylene chloride (10:1 v/v) solution using RPR-3000 Å lamps for 80 hr. Chromatography on basic alumina gave a yellow solid (<1%), mp 118–119°, which could be the diazepine but was not available in sufficient quantity for characterization and 2-benzenesulfonylamino-3,5-dimethylpyridine (**19**) (18%), mp 132° (from benzene–cyclohexane), identical with a sample synthesized from 2-amino-3,5-dimethylpyridine and benzenesulfonyl chloride in pyridine: ir (KBr) (main bands only) 3250 (s), 1600 (vs), 1540 (s), 1408 (s), 1372 (s), 1340 (s), 1245 (s), 1130 (s), 1080 (vs), 983 (s), 936 (s), 740 (s), 720 (s), 695 cm^{-1} (s); mass spectrum *m/e* (rel intensity) 262 (4, M^+), 197 (100, $M^+ - H\cdot - SO_2$), 121 (41, $M^+ - PhSO_2\cdot$), 77 (46), 39 (20).

Anal. Calcd for $C_{13}H_{14}N_2O_2S$: C, 59.52; H, 5.38. Found: C, 59.67; H, 5.53.

Benzenesulfonamide (33%) and starting ylide (8.5%), mp 209–211°, were also isolated.

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Registry No.—**7** (R = Ph), 20169-41-9; **17**, 40988-50-9; **19**, 40949-66-4; **22** (R = H), 40949-67-5; **22** (R = Me), 40949-68-6; methanesulfonyl hydrazide, 10393-86-9; 2,4,6-trimethylpyridium perchlorate, 940-93-2.

Reactivity of Thiazole in Electrophilic Reactions as Determined from Solvolysis Rates[1]

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Solvolysis rates for the three isomeric 1-thiazolylethyl chlorides have been determined in 80% ethanol. The general reactivity of thiazole in electrophilic substitution reactions has been discussed and the decreasing order of reactivity of 5-thiazolyl > 4-thiazolyl > phenyl > 2-thiazolyl has been established.

The solvolysis of α-arylethanol derivatives is a useful probe of aromatic reactivity. Streitwieser, *et al.*,[2–4] have recently compared the reactivities of a large number of aromatic hydrocarbons, and the solvolysis rates of the corresponding arylmethyl tosylates. There is good correspondence in the two series, covering a wide variety of types and conditions,[2] and to various semiempirical MO methods;[4] the results lead to a useful set of σ constants for the aromatic moiety, designated σ^+ by Streitwieser.[3]

This concept has been extended to heterocyclic systems by Hill,[5] by the senior author,[6] by Taylor,[7] and by Marino.[8] Particularly pertinent are the observed relationships of reactivity of thiophene and its derivatives[6,9] and of furan and its derivatives.[10]

The solvolysis reaction has several distinct advantages for the investigation of basic heterocyclic systems, as it avoids the uncertainties of whether reaction is occurring *via* the protonated form or the free base. Studies from these laboratories have established σ^+ values for pyridine moieties in this fashion.[11]

In the present study we examine the thiazole system. Each of the isomeric 1-(thiazolyl)ethyl chlorides was prepared and solvolyzed. The data are given in Table I, and the rates are compared to the solvolysis rate for 1-phenylethyl chloride, which is of similar reactivity. (See Table I.)

From the rate data in Table I, we calculate σ^+ values appropriate for the various thiazole moieties,[12] using ρ for the solvolysis −5.12.[13] Thus the replacement $\sigma_{Ar}{}^+$ are as follows: 5-thiazolyl −0.18; 4-thiazolyl −0.01; 2-thiazolyl +0.26.

There have been a number of MO calculations carried out on thiazole. Metzger and his coworkers[14] have recently summarized calculations carried out to various levels of sophistication. All of these methods agree that the reactivity order is 5 > 4 > 2.

The greater reactivity of 1-(5-thiazolyl)ethyl chlo-

(1) Supported in part by a grant from the National Science Foundation, GP-6133X.

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TABLE I

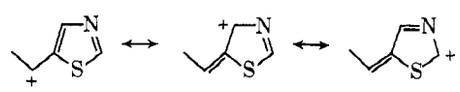
SOLVOLYSIS RATE CONSTANTS FOR 1-ARYLETHYL CHLORIDES IN 80% ETHANOL

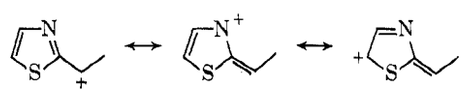
Compd solvolyzed	Temp, °C	$10^5 k$, sec^{-1}	Method[a]	Relative rate
1-(5-Thiazolyl)ethyl chloride (3)	25.00	8.55 ± 0.2	A	
	45.00	80.9 ± 0.7	A	174
1-(4-Thiazolyl)ethyl chloride (8)	25.00	0.922 ± 0.02	A	
	45.00	11.5 ± 0.6	A	25
	60.00	46.9 ± 1.0	A	
1-(2-Thiazolyl)ethyl chloride (5)	45.00	0.464[b]		1.00
	60.00	2.29 ± 0.06	B	
	75.00	10.7 ± 0.4	B	
	100.00	88.0 ± 3	C	
1-Phenylethyl chloride	45.00	9.76[c]	A	21

[a] A, constant pH: B, aliquot; C, sealed ampoules. [b] Extrapolated from data at higher temperatures. [c] Determined by B. Bartman; agrees well with values reported by V. J. Shiner, W. E. Buddenbaum, B. L. Murr, and G. Lamaty, *J. Amer. Chem. Soc.*, 90, 418 (1968).

ride (3) compared to 1-(4-thiazolyl)ethyl chloride (8) is in accord with the generally more facile electrophilic reactions of the 2 position in thiophene than of the 3 position in thiophene.[6] The reactivity ratio is sharply reduced in the case of the thiazole compounds. The comparison of 1-(5-thiazolyl)ethyl chloride (3) with 1-(2-thiazolyl)ethyl chloride (5) reflects the deleterious influence of replacing one of the carbons by nitrogen, as shown by considering the contributing canonical forms for the two cations A and B, where in B one of the forms places electron deficiency directly on the nitrogen. This is reminiscent of the comparison of 3-pyridyl systems with 2-pyridyl systems.

The various calculations summarized by Metzger, *et al.*,[14] do not agree about the reactivity of the thiazoles relative to benzene. Our data provide a direct


(A)


(B)

experimental evaluation of this relationship. There are relatively limited data on aromatic substitution reactions with which to make comparison. The acetoxymercuration of various thiazoles has suggested the reactivity order 5 > 4 > 2;[15] direct comparison with benzene is not available. Some systems have been studied which allow indirect comparison in competitive situations. Though the nitration of the various phenylthiazoles[16] gives products resulting from nitration in the benzene ring, bromination of 4-chloro-2-phenylthiazole occurs in the thiazole ring.[17] These results are in accord with our reactivity sequence.

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Experimental Section[18]

1-(2-Chloro-5-thiazolyl)ethanol (1).—To a stirred solution of dry ether (100 ml) at −80° under a nitrogen atmosphere was added 2-chlorothiazole (9.81 g, 0.082 mol) in 75 ml of ether. Simultaneously from another dropping funnel, precooled *n*-butyllithium (0.09 mol, 55.8 ml) in hexane was added. After the 1-hr addition period, the light tan solution was stirred for 2.5 hr with gradual warming to −20°. Acetaldehyde (17.0 ml, 0.30 mol) was rapidly added. The solution was stirred for 1 hr and then quenched with 100 ml of cold water. The layers were separated and the aqueous layer was exrracted with 3 × 30 ml of ether. The combined ether layers were dried ($MgSO_4$) and filtered. Distillation afforded 11.76 g (88%) of alcohol 1: bp 173° (51 mm); nmr ($CDCl_3$) δ 1.55 (d, 3, J = 6.5 Hz, $CHCH_3$), 4.73 (s, 1, CHOH), 5.03 (q, 1, J = 6.5 Hz, $CHCH_3$), 7.22 (s, 1, 4-H).

1-(5-Thiazolyl)ethanol (2),—A solution of 1-(2-chloro-5-thiazolyl)ethanol (10.0 g) in 50 ml of acetic acid was stirred with heating to near boiling before adding zinc dust (8.0 g). The reaction mixture was refluxed for 4 hr. The resulting solution was diluted with 100 ml of ether and adjusted to pH 8 by the slow addition of ammonium hydroxide. The layers were then separated, and the aqueous layer was extracted with 3 × 40 ml of ether. The combined ether extracts were dried over anhydrous magnesium sulfate and filtered. Removal of the ether on a rotary evaporator gave a yellow liquid which had spectral properties indicating a 50:50 mixture of the desired alcohol 2 and its acetate. The acetate was hydrolyzed by heating this mixture for 2 hr under reflux with 150 ml of 1 *N* HCl. After cooling, the solution was diluted with 200 ml of ether and neutralized. The layers were separated and the aqueous layer was extracted with 3 × 30 ml of ether. The combined extracts were dried ($MgSO_4$), filtered, and distilled to give 3.97 g (50%) of alcohol 2: bp 126–128° (25 mm); nmr ($CDCl_3$) δ 1.55 (d, 3, J = 6.5 Hz, $CHCH_3$), 4.70 (s, 1, OH), 5.12 (q, 1, J = 6.5 Hz, $CHCH_3$), 7.53 (s, 1, 4-H), 8.62 (s, 1, 2-H).

Anal. Calcd for C_5H_7NOS: C, 46.49; H, 5.46; N, 10.84; S, 24.82. Found: C, 46.54; H, 5.36; N, 11.02; S, 24.64.

1-(5-Thiazolyl)ethyl Chloride (3).—A stirred solution of 1-(5-thiazolyl)ethanol (3.5 g) in 50 ml of cargon tetrachloride was treated under nitrogen with phosphorus pentachloride (5.64 g) for 2 hr. After the volume was reduced to approximately 10 ml on a rotary evaporator, the solution was diluted with 100 ml of ether and stirred for 20 min with a 5-ml aqueous slurry of sodium bicarbonate. The resulting layers were separated, and the organic layer was dried ($MgSO_4$), filtered, and concentrated. The resulting light yellow oil was further purified by chromatography on silica gel. Elution with 95% hexene–5% ether resulted in 2.35 g (67%) of the pure chloride 3: nmr ($CDCl_3$) δ 1.90 (d, 3, J_{CH,CH_3} = 6.7 Hz, $CHCH_3$), 5.33 (q, 1, J_{CH,CH_3} = 6.7 Hz, CHCl), 7.75 (s, 1, 4-H), 8.73 (s, 1, 2-H).

Anal. Calcd for C_5H_6ClNS: C, 40.68; H, 4.09; Cl, 24.02; N, 9.49. Found: 40.78; H, 3.94; Cl, 24.28; N, 9.62.

1-(2-Thiazolyl)ethanol (4).—Halogen–metal interchange between 2-bromothiazole and butyllithium at −80° was followed by addition of a threefold excess of acezaldehyde. Isolation in the usual manner and distillaton afforded 4 in 51% yield: bp 115–116° (26 mm) [lit.[19] bp 112–115° (13–15 mm)]; nmr (CCl_4) δ 1.48 (d, 3, J = 7.0 Hz, $CHCH_3$), 5.05 (q, 1, J = 7.0 Hz, $CHCH_3$), 5.70 (s, 1, OH), 7.13 (d, 1, $J_{4,5}$ = 3.5 Hz, 5-H), 7.50 (d, 1, $J_{4,5}$ = 3.5 Hz, 4-H).

1-(2-Thiazolyl)ethyl Chloride (5).—Conversion of alcohol 4 to the chloride 5 was accomplished using thionyl chloride. Work-up and distillation afforded 42% of the chloride 5: bp 84–85° (36 mm); nmr ($CDCl_3$) δ 1.90 (d, 3, J = 7.0 Hz, $CHCH_3$), 5.35 (q, 1, J = 7.0 Hz, CHCl), 7.25 (d, 1, J = 3.5 Hz, 5-H), 7.67 (d, 1, J = 3.5 Hz, 4-H).

Anal. Calcd for C_5H_6ClNS: C, 40.68; H, 4.09; Cl, 24.02; N, 9.49; S, 21.72. Found: C, 40.50; H, 4.07; Cl, 23.98; N, 9.60; S, 21.53.

4-Formylthiazole (6).—The procedure used was a modifica-

(18) Melting points and boiling points are uncorrected. Routine infrared spectra were obtained using a Perkin-Elmer Infracord Model 237. Nmr spectra were obtained using either a Varian A-60 or a Varian T-60 spectrometer. Analyses are by the Chemical Analytical Services Laboratory, College of Chemistry, University of California, Berkeley, Calif. 94720.

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tion of that of Baganz and Rüger.[20] Thioformamide[21] and tribromoacetone[22] were combined in ether and kept cold for 2 days, and the resulting solid was thoroughly washed to give 4-dibromomethyl-4-hydroxy-Δ^2-thiazoline hydrobromide, mp 120–123°. Following Baganz and Rüger,[20] the hydrobromide was treated with sulfuric acid to give 4-dibromomethylthiazole, mp 89–90° (hexane), which was hydrolyzed to **6**: mp 59–61° (lit.[23] 65–66°); nmr ($CDCl_3$) δ 8.31 (d, 1, $J_{2,5}$ = 1.9 Hz, 5-H), 8.98 (d, 1, $J_{2,5}$ = 1.9 Hz, 2-H), 10.13 (s, 1, CHO).

1-(4-Thiazolyl)ethanol (7).—Treatment of **6** with methylmagnesium bromide gave 90% of the alcohol **7**: bp 164–165° (55 mm); nmr ($CDCl_3$) δ 1.57 (d, 3, J = 6.3 Hz, $CHCH_3$), 4.08 (s, 1, CHOH), 5.05 (q, 1, J = 6.3 Hz, $CHCH_3$), 7.20 (d, 1, $J_{2,5}$ = 1.9 Hz, 5-H), 8.53 (d, 1, $J_{2,5}$ = 1.9 Hz, 2-H).

1-(4-Thiazolyl)ethyl Chloride (8).—Alcohol **7** was converted to chloride **8** using phosphorus pentachloride in 74% yield: nmr ($CDCl_3$) δ 1.90 (d, 3, J = 6.8 Hz, $CHCH_3$), 5.28 (q, 1, J = 6.8 Hz, $CHCH_3$), 7.32 (d, 1, $J_{2,5}$ = 2.0 Hz, 5-H), 8.75 (d, 1, $J_{2,5}$ = 2.0 Hz, 2-H). Decomposition occurs on attempted distillation.

Anal. Calcd for C_5H_6ClNS: C, 40.68; H, 4.09; Cl, 24.02; N, 9.49. Found: C, 40.82; H, 4.33; Cl, 24.22; N, 9.28.

Kinetic Procedures.—Absolute ethanol was prepared by the method of Lund and Bjerrum.[24] Four volumes of absolute ethanol were diluted with one volume of water. Rate constants were determined in three different fashions. Method A was by maintenance of a static pH. This method is particularly convenient when measurements are made near room temperature and half-lives are reasonably short. A Radiometer automatic titration apparatus was used, consisting of a no. TTT 1c automatic titrator, a no. ABU 1c autoburet (with a 2.5-ml buret), a TTA 3c titrator assembly, and a no. SBR 2c recorder. A 49-ml sample of the reaction medium was brought to temperature in the reaction cell in a constant-temperature bath. Reaction was initiated by injecting, *via* syringe, *ca.* 0.0005 mol of substrate dissolved in 1 ml of 80% ethanol–20% water into the reaction cell. The reaction solution was maintained at a constant apparent pH of 7.5 by the automatic addition of 0.30 *M* potassium hydroxide in 80% ethanol. The recorder plotted a continuous curve of the addition of base *vs.* time.

Alternatively, standard aliquot techniques (method B) or sealed ampoules (method C) were used. First-order rate constants were computed using the nonlinear least squares program, LSKIN 1.[25]

Registry No.—**1**, 40982-18-1; **2**, 41040-84-0; **3**, 41040-85-1; **4**, 40982-30-7; **5**, 40982-31-8; **6**, 3364-80-5; **7**, 41040-89-5; **8**, 3364-77-0; 2-chlorothiazole, 3034-52-4; acetaldehyde, 75-07-0; 2-bromothiazole, 3034-53-5; thioformamide, 115-08-2; tribromoacetone, 3770-98-7; 4-dibromomethyl-4-hydroxy-Δ^2-thiazoline hydrobromide, 41040-92-0; 4-dibromomethylthiazole, 41040-93-1; methyl bromide, 74-83-9.

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Transmission of Substituent Effects in Heterocyclic Systems. Rates of Solvolysis of Substituted Thiazolylethanol Derivatives[1,2]

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The rates of solvolysis for several substituted 1-(5-thiazolyl)ethanol derivatives have been measured in 80% ethanol and are compared with similar studies on other heterocycles. For the group 1-(2-X-5-thiazolyl)ethyl chlorides, reaction rates are well correlated by σ_p^+ for the substituents X. Similar correlation is observed for the solvolysis rates of 1-(5-X-2-thiazolyl)ethyl chlorides. These results are discussed in terms of the application of molecular orbital calculations relevant to the thiazole system.

In continuing studies from these laboratories[2–6] on the modes of transmission of substituent effects in heteocyclic systems, we have examined the relative reactivities of a number of substituted thiazoles.

There have been a few previous investigations of the application of the Hammett equation to thiazole derivatives. Imoto, Otsuji, and coworkers[7,8] have measured saponification rates for substituted ethyl thiazolecarboxylates and the dissociation constants of the corresponding acids. Moderately satisfactory correlation with Hammett σ values was observed; however, the value of ρ was occasionally surprising.

Our previous studies have shown that the solvolysis reaction is a useful probe for examination of heterocyclic systems[4,9] and that reactivities can often be related to parameters obtained from molecular orbital calculations. Metzger, *et al.*,[10] have recently summarized the results of a number of different molecular orbital calculations on thiazole. All of the various levels of approximation agree that the susceptibility to electrophilic aromatic substitution is in the order position 5 > position 4 > position 2; however, there is not agreement as to reactivity relative to benzene. Our studies[2] have shown that the order relative to benzene is 5-thiazolyl > 4-thiazolyl $\simeq$ phenyl > 2-thiazolyl.

Effect of Substituents.—We have prepared a number of 2-substituted 1-(5-thiazolyl)ethanols (A), and have measured the rates of solvolysis of the respective chlorides or *p*-nitrobenzoates. The large differences in reactivity dictated the use of different leaving groups. A pair of 5-substituted 1-(2-thiazolyl)ethanols (B) has likewise been examined.

In the case of series A, the measured rates are col-

(1) Supported in part by a grant from the National Science Foundation, GP-6133X.
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