eluant than with water. Since the peak concentration of propanol-1 would appear at an effluent volume of 475 ml. with 3.0 *M* ammonium sulfate, the elution of this alcohol was hastened by changing the eluant to water after the elution of ethanol. Some precaution, such as taping the column, should be taken to guard against the hazard due to the

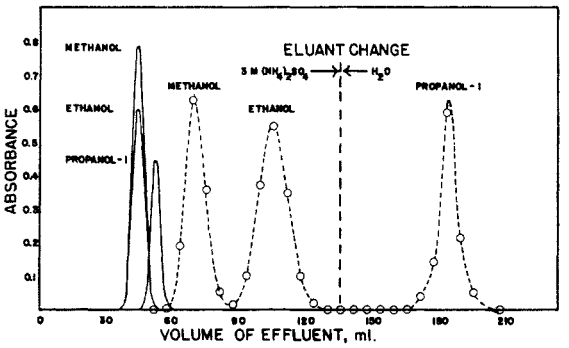

FIG. 1. COMPARISON OF THE SEPARABILITY OF ALCOHOLS WITH WATER AND 3.0 *M* AMMONIUM SULFATE. Eluants: water (————), 3 *M* ammonium sulfate (----------). Column: 25.7 cm. × 2.28 cm.², Dowex 1 × 4, 200–300 mesh, sulfate form. Flow rate: 0.5 cm./min.

swelling of the resin at this point. The quantity of the eluted alcohol in each fraction was determined by oxidation with dichromate in 50% sulfuric acid and the subsequent measurement of the absorbance of Cr(III).[4]

The beneficial effect of an electrolyte in the eluant is probably due to a selective salting-out of the organic compounds from the aqueous to the resinous phase. As the salt concentration in the water phase is increased, the solubility of the organic compound is decreased. Linear increases in the salt concentration produce roughly exponential increases in the abscissas of the peaks of the elution graphs.

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Methoxy- and Hydroxy-styryl Heterocycles

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The marked antiseptic and trypanocidal activity of styrylpyridine and styrylquinoline derivatives has been reported;[1] styrylbenzothiazole compounds have been prepared and tested for similar chemotherapeutic properties.[2]

For use in connection with other studies, several

(1) (a) Ashley, Browning, Cohen, and Gulbranson, *Proc. Roy. Soc. (London)*, **B113**, 293 (1933); (b) Browning, Cohen, Ellingsworth, and Gulbranson, *J. Path. Bact.*, **27**, 121 (1924); (c) Browning, Cohen, Ellingsworth, and Gulbranson, *Brit. Med. J.*, **II**, 326 (1923).

(2) Stephens and Webberly, *J. Chem. Soc.*, 3336 (1950).

TABLE I

METHOXYSTYRYL COMPOUNDS

No.	Compound	M.p., °C.	Yield, %	Recrystallization solvent	Analyses S Calc'd	S Found	N Calc'd	N Found
I	2-(*p*-Methoxystyryl)benzothiazole	142–144	58[a]	Ethanol	11.95	12.00		
II	2-(2′,3′-Dimethoxystyryl)-benzothiazole	90–91	88	Dilute methanol	10.80	10.64		
III	2-(3′,4′-Dimethoxystyryl)-benzothiazole	150–151	67	95% Ethanol	10.75	11.00		
IV	2,6-Di-(2′,3′-dimethoxystyryl)-pyridine	140–141	50	Benzene-pet. ether (b.p. 60–70°)			3.48	3.62
V	2,6-Di-(3′-methoxy-4′-hydroxystyryl)pyridine	173–175	39.5	95% Ethanol			3.73	4.00
VI	2,3-Di-(*p*-methoxystyryl)quinoxaline	163–164	71[b]	Benzene				
VII	2,3-Di-(3′,4′-dimethoxystyryl)-quinoxaline	196–197[c]	64[c]	Benzene-pet. ether (b.p. 60–70°)			6.17	6.39
VIII	2-(*p*-Methoxystyryl)-6-methoxyquinoline	162–163	76	Benzene-pet. ether (b.p. 60–70°)			4.82	5.00
IX	2-(3′,4′-Dimethoxystyryl)-6-methoxyquinoline	137–138	75	95% Ethanol			4.36	4.31
X	2-(2′,3′-Dimethoxystyryl)-4-hydroxy-6-methoxyquinoline	278–280	60	Methyl Cellosolve[d]			4.15	4.27

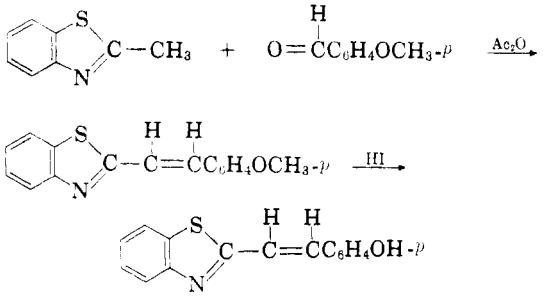
[a] A 3% yield was obtained with piperidine as a catalyst. [b] Previously prepared in 10% yield (see ref. 7). [c] Reported m.p. 208°, in 10% yield (see ref. 7). [d] Trade mark for 2-methoxyethanol.

TABLE II
HYDROXYSTYRYL COMPOUNDS

No.	Compound	M.p., °C.	Yield, %	Analyses S Calc'd	S Found	N Calc'd	N Found
XI	2-(*p*-Hydroxystyryl)benzothiazole	212–213[a]	84.5	12.65	12.68		
XII	2-(2′,3′-Dihydroxystyryl)benzothiazole	dec. 185–190[b]	52	11.89	11.65		
XIII	2-(3′,4′-Dihydroxystyryl)benzothiazole	dec. 220–230[b]	62	11.89	11.69		
XIV	2-(*p*-Hydroxystyryl)-6-hydroxyquinoline	dec. 270–275[c]	58			5.35	5.15

[a] Recrystallized from ethanol. [b] Purified by dissolving in hot glacial acetic acid and adding to an excess of water. [c] Purified by dissolving in sodium hydroxide and precipitating with dilute acetic acid.

heretofore unreported styryl derivatives of nitrogen-containing heterocycles have been prepared. A specific example of the preparation of 2-(*p*-hydroxystyryl)benzothiazole is given and illustrates the general method of synthesis employed for the reported compounds.



Although it has been indicated[3] that acetic anhydride may be inferior as a condensing agent, our results, in general, substantiate the contradictory findings of other workers.[4,5]

EXPERIMENTAL[6]

The procedures described below are typical for the preparation of the methoxystyryl compounds (Table I) and the hydroxystyryl compounds (Table II).

2-(p-Methoxystyryl)benzothiazole. A mixture of 14.9 g. (0.1 mole) of 2-methylbenzothiazole, 13.6 g. (0.1 mole) of *p*-anisaldehyde, and 10.2 g. (0.1 mole) of acetic anhydride was refluxed for 24 hours under a nitrogen atmosphere. On cooling, the mixture formed a crystalline mass; this was triturated with 50% ethanol, filtered, and washed with 80% ethanol. The product was recrystallized from ethanol to give a solid melting at 142–144°. The yield was 15.0 g. (58%).

2,3-Di-(*p*-methoxystyryl)quinoxaline (VI, Table I) was prepared in 71% yield as compared to the 10% yield obtained by Bennett and Willis.[7] In the latter preparation, perhaps an excessive amount of acetic anhydride (which lowers the reflux temperature of the reaction mixture) was used and the mixture was not heated long enough.

2-(p-Hydroxystyryl)benzothiazole. In 200 ml. of a 1:1 mixture of 47% hydriodic acid and glacial acetic acid was dissolved 7.5 g. of 2-(*p*-methoxystyryl)benzothiazole; the resulting solution was refluxed for 24 hours. On cooling, the mixture was diluted with water and made basic with ammonium hydroxide. Crystals separated (m.p. 211–212°) which were filtered off and washed with water. This product was dissolved in 5% sodium hydroxide, treated with Norit A, and filtered. Acidification of the filtrate with acetic acid caused precipitation. Following filtration and recrystallization from ethanol, 6.0 g. (84.5%) of 2-(*p*-hydroxystyryl)-benzothiazole, m.p. 212–213°, was obtained.

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(3) Tipson, *J. Am. Chem. Soc.*, **67**, 507 (1945).
(4) Gilman and Karmas, *J. Am. Chem. Soc.*, **67**, 342 (1945).
(5) Chiang and Hartung, *J. Org. Chem.*, **10**, 21 (1945).
(6) All melting points are uncorrected.
(7) Bennett and Willis, *J. Chem. Soc.*, 1960 (1928).

A Synthesis of Optically Active Styrene Oxide and Other Epoxides[1]

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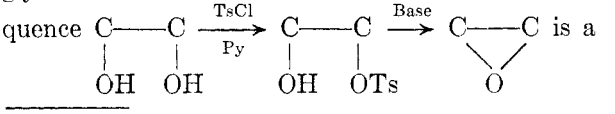
The synthesis of simple alicylic epoxides,[3] sugar epoxides[4] and epoxides in the cyclitol series[5] by ring closure of glycol monotosylates is well known. However, the only application of this reaction to simple aliphatic epoxides seems to be the synthesis of *l*-glycidol from *l*-glycerol-1-tosylate[6] obtained from a sugar derivative *via* 2,3-isopropylidene-*l*-glycerol. We have now found that the reaction sequence  is a

* Original manuscript received January 11, 1956.
(1) Contribution from the Radiation Project of the University of Notre Dame supported in part under Atomic Energy Commission contract AT(11-1)-38 and Navy equipment loan contract Nonr-06900.
(2) Shell Research Fellow, 1955–1956.
(3) L. N. Owen, *et al.*, *J. Chem. Soc.*, 315 (1949); 4026 (1952); 2582 (1953).
(4) *Cf.* S. Peat, *Advances in Carbohydrate Chemistry*, **2**, 41 (1946); R. S. Tipson, *Advances in Carbohydrate Chemistry*, **8**, 166 (1953).
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(6) J. C. Sowden and H. O. L. Fischer, *J. Am. Chem. Soc.*, **64**, 1291 (1942).