TABLE I

COMPARISON OF THE SPECTRA OF 4-(4-DIETHYLAMINO-1-METHYLBUTYLAMINO)-QUINOLINE WITH 7-SUBSTITUTED CONGENERS, IN 0.1 *N* HYDROCHLORIC ACID

Compound	Maxima, mμ and ($\epsilon \times 10^3$)			
4-(4-Diethylamino-1-methylbutylamino)-quinoline	232 (18.9)		330 (18.8)	340 (18.5)
4-(4-Diethylamino-1-methylbutylamino)-7-phenoxyquinoline	228 (33.6)	250[a] (19.0)	330 (18.8)	336 (18.6)
4-(4-Diethylamino-1-methylbutylamino)-7-ethoxy-3-methylquinoline	233 (28.7)	259[b] (17.8)	330 (14.5)	

[a] Inflection point. [b] Small secondary maximum.

quinoline[2,3] (SN 10,663)[4] and the parent compound, 4-(4-diethylamino-1-methylbutylamino)-quinoline (SN 6732)[4,5] both in 0.01 *N* hydrochloric acid, shows that there is a slight but definite hypsochromic shift of 4 mμ throughout the whole spectrum. This is doubtless due to the combined influences of an ether link and the unconjugated aryl group attached in position 7. As we have earlier noted,[6] the views of Irvin and Irvin[7] concerning the effect of a 7-substituent upon the tautomerism of the 4-aminoquinoline type are well substantiated. In this instance, the weighting due to the phenoxy group is greater than the halogens and is also more pronounced. The unconjugated phenyl radical behaves very much as the simpler alkoxy substituents in the case of the 4-(4-diethylamino-1-methylbutylamino)-7-ethoxy-3-methylquinoline,[8] inasmuch as benzene shows no pronounced absorption in this general region.[9] Since the ethoxy compound also bears a methyl substitution, it also exhibits the slight batho- and hypochromic shift inherent to 3-methyl substitution, as we have shown (*cf.* Fig. 2, ref. 6).

[2] Drake, *et al.*, THIS JOURNAL, **68**, 1208 (1946); Riegel, *et al.*, *ibid.*, **68**, 1264 (1946); Clinton and Suter, *ibid.*, **69**, 704 (1947).

[3] The authors are indebted to Dr. R. O. Clinton for the sample employed.

[4] All drugs identified by Survey Numbers (SN) in the files of the Antimalarial Survey office have been tabulated, with antimalarial activities, in the work by Wiselogle, editor, "Antimalarial Drugs, 1941-1945," Edwards Bros., Ann Arbor, Mich., 1946.

[5] Steck, Hallock and Suter, THIS JOURNAL, **70**, unpublished (1948).

[6] Steck, Ewing and Nachod, *ibid.*, **70**, 3410 (1948).

[7] Irvin and Irvin, *ibid.*, **69**, 1091 (1947).

[8] Steck, Ewing and Nachod, *ibid.*, forthcoming paper.

[9] Dimroth, *Angew. Chem.*, **52**, 548 (1939).

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Preparation of Methanesulfonyl Chloride

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Three methods commonly have been used for the preparation of methanesulfonyl chloride, a valuable reagent, namely, by the action of phosphorus pentachloride on sodium methanesulfonate,[1] and by the action of chlorine and water on *s*-methylisothiourea[2] or on methyl thiocyanate.[3]

Yields are not very satisfactory by the first method, and it is difficult to remove phosphorus compounds from the product.[4] The second method is potentially dangerous since violent explosions of products apparently formed by excessive chlorination have been reported.[5] The third method works well but is disagreeable because cyanogen chloride is one of the products of the reaction.

So far as the writers are aware, there is no reference in chemical literature to the preparation of sulfonyl chlorides directly from sulfonic acids by the action of thionyl chloride, although this reagent commonly is used to prepare carboxylic acid chlorides from carboxylic acids. With the availability of methanesulfonic acid commercially, the preparation of methanesulfonyl chloride by this reaction was tried. It was obtained in good yield and the procedure developed far excels those previously reported.

ADDED TO PROOF:—A recent publication[6] indicates that alkanesulfonyl chlorides may be prepared by the action of either thionyl chloride or phosphorus trichloride on the sulfonic acids. Directions are given for the preparation of methanesulfonyl chloride in 57% yield from methanesulfonic acid and phosphorus trichloride.

Experimental

In a 200-cc., three-necked flask, fitted with a mechanical stirrer, a reflux condenser, a thermometer and a separatory funnel, and set up in a hood, is placed 152 g. (105 cc., 1.5 moles) of 95% methanesulfonic acid (Standard Oil Co. of Indiana). The acid is heated to 95° on a steam-bath, and 146 cc. (238 g., 2.0 moles) of thionyl chloride (Eastman Kodak Co. grade) is added over a period of four hours. The temperature is kept at 95° throughout the addition, and heating is continued for three and one-half hours after the addition has been completed.

The product is transferred to a modified Claisen flask and distilled at reduced pressure, using an oil-bath to supply heat. Most of the thionyl chloride distills at room temperature. A free flame should be avoided, since local superheating causes charring and decomposition. The fumes from the decomposition cause the normally clear product to darken. The bath temperature should not exceed 115° at the end of the distillation. The yield of product distilling at 67–73° (20 mm.) is 122 g. (71% of the theoretical amount).

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[1] Marvel, Helfrick and Beasley, THIS JOURNAL, **51**, 1272 (1929).

[2] Johnson and Sprague, *ibid.*, **58**, 1348 (1936).

[3] Johnson and Douglass, *ibid.*, **61**, 2548 (1939).

[4] Helferich and Gnüchtel, *Ber.*, **71**, 712 (1938).

[5] Folkers, Russell and Bost, THIS JOURNAL, **63**, 3530 (1941).

[6] Proell, Adams and Shoemaker, *Ind. Eng. Chem.*, **40**, 1129 (1948).

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Reduction of Sugar Epoxides to Desoxysugars

BY D. A. PRINS

Derivatives of 2-desoxy-D-allose have been prepared from the convenient and readily available