Both the 1,1- and 1,2-dichlorohexafluoropropanes have been prepared by Henne and Waalkes.[4,5]

In our work the 1,3-dichloride was made by first adding hydrogen fluoride to 1,2-dichloro-2-propene to form the difluoromonochloride, then chlorinating this to the hexachlorodifluoride and finally fluorinating the latter to the hexafluorodichloride.

Experimental

Preparation of $CH_3–CF_2–CH_2Cl$.—The procedure used was similar to that of Henne and Haeckl[6] with the important exceptions that the temperature was kept at 100°, by means of a steam-bath, instead of at 50–60° and that hydrogen chloride was bled off at a pressure of 270 lb. per sq. in. gage. This process resulted in a 60% yield of difluorochloride.

Preparation of $CCl_3–CF_2–CCl_3$.—Ten moles (1145 g.) of $CH_3–CF_2–CH_2Cl$ was chlorinated in sunlight at 75–85° until the theoretical weight of chlorine had been absorbed. The crude material was fractionated to give a practically quantitative yield of $CCl_3–CF_2–CCl_3$, boiling range 193–195°.[7]

Preparation of $CF_2Cl–CF_2–CF_2Cl$.—In a steel pressure cylinder eight moles (1430 g.) of antimony trifluoride was treated with four moles (284 g.) of chlorine. Four moles (1147 g.) of $CCl_3–CF_2–CCl_3$ was added and the mixture was heated in an oil-bath at 210° for one hour, 230° for four hours and 250° during two hours. The pressure eventually rose to 300 lb. per sq. in. gage. The organic material was distilled from the cylinder, washed with concd. hydrochloric acid, neutralized and fractionated. There were obtained 370 g. of $CF_2Cl–CF_2–CF_2Cl$, b.p. 35.8°, $n^{20}{}_D$ 1.3030, $d^{20}{}_4$ 1.5730[8] and 300 g. of $CFCl_2–CF_2–CF_2Cl$ intermediate, which can be converted to the above hexafluorodichloride.

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(5) A. L. Henne and T. P. Waalkes, *ibid.*, **68**, 496 (1946).

(6) A. L. Henne and F. W. Haeckl, *ibid.*, **63**, 2692 (1941).

(7) A. L. Henne and M. W. Renoll, *ibid.*, **59**, 2434 (1937).

(8) Young and Murray[9] found the freezing range of this compound to be −126.3 to −125.4°.

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Some Quaternary Salts of 8-Hydroxyquinoline[1]

BY CARL T. BAHNER, LLOYD A. WALKER, FRANCES PIERCE AND EMMA KITE

The preparation of quaternary salts of substituted quinolines[2] has been extended to include salts of 8-hydroxyquinoline.

The salts were prepared by heating a mixture of the base and the appropriate organic halide at 100°, dissolving the resulting dark amorphous mass in hot alcohol, and adding ethyl acetate, ethyl ether or petroleum ether to throw out crystals of the product which was then recrystallized to a constant melting point before analysis. A 30-minute reaction period sufficed for the phenacyl bromides, but β-phenylethyl iodide required 48 hours and β-cyclohexylethyl bromide did not react satisfactorily even when heated 96 hours. The products were yellow, crystalline solids which were not readily soluble in water. The melting points and analytical data are shown in Table I.

Acknowledgments.—We wish to express our appreciation to Dr. M. J. Shear and Dr. J. L. Hartwell of the National Cancer Institute for arranging to screen these compounds against tumors in mice, and to Mr. George Biggerstaff and Mr. Lilburn Norton for assisting in the purification and analysis of one of these products.

TABLE I

8-HYDROXYQUINOLINIUM SALTS

Salt of 8-hydroxyquinoline with:	Empirical formula	M.p., °C.	Yield, %[a]	Analyses, % ionic halogen Calcd.	Found[b]
β-Phenylethyl iodide	$C_{17}H_{16}INO$	168.5–169[c]	85	33.64	33.46
Phenacyl bromide	$C_{17}H_{14}BrNO_2$	234–235	58	23.21	22.98
p-Methylphenacyl bromide	$C_{18}H_{16}BrNO_2$	223.5–224	46	22.26	22.09
p-Bromophenacyl bromide	$C_{17}H_{13}Br_2NO_2$	244.5–245	50	18.89	18.78
p-Iodophenacyl bromide	$C_{17}H_{13}BrINO_2$	257–258	58	16.99	16.68
p-Methoxyphenacyl bromide	$C_{17}H_{16}BrNO_3$	230	10	21.35	21.19
β-Naphthacyl bromide	$C_{21}H_{16}BrNO_2$	234–235	42[d]	20.02	19.93

[a] Crude yield of crystalline material unless otherwise indicated. [b] Average of two Volhard analyses for ionic halogen. After final recrystallization from acetic acid. [d] Yield of pure material.

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(1) This investigation was supported in part by a research grant from the National Cancer Institute, of the National Institutes of Health, Public Health Service, for which we are grateful.

(2) C. T. Bahner, W. K. Easley, M. D. Pickens, H. D. Lyons, L. L. Norton, B. G. Walden and G. Biggerstaff, This Journal, **73**, 3499 (1951).

Morpholinium Salts[1]

BY CARL T. BAHNER, EMMA KITE, FRANCES PIERCE, LYDIA MOORE RIVES, MADGE DEEL PICKENS AND CLIFFORD MYERS

In extending the study of analogs of quaternary pyridinium salts as possible anti-cancer agents[2] we have synthesized tertiary amine hydrohalides and quaternary ammonium salts containing a morpholine ring. Lutz and his associates[3] have prepared a number of N-substituted morpholines for use as intermediates or for testing as anti-malarials and Hazard, Corteggiani and Renard[4] have reported that 4-methyl-4-(2-phenylethyl)-morpholinium iodide produced marked hypertension in dogs at a dose of 5 mg./kg.

The tertiary amine hydrohalides were obtained by reaction of morpholine with the appropriate organic halide in equimolecular proportions and the quaternary salts were prepared from N-substituted morpholines in the same manner. The time required for reaction varied from a few minutes to a

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(2) *Cf.*, C. T. Bahner, M. Fielden, L. M. Rives and M. D. Pickens, This Journal, **73**, in press (1951).

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