

The iminochloride was also obtained from 9-fluorylamine hydrochloride by the method of Peterson.³

Photochemical Decomposition of 9-Fluoryldichloramine.—Within a few hours of exposure to sunlight of a solution of 11.8 g. of 9-fluoryldichloramine in 100 cc. of dry benzene contained in a stoppered Erlenmeyer flask, a white crystalline deposit settled on the wall of the flask. This behavior necessitated daily filtration and reexposure of the filtrate. In the course of a week 4.52 g. of 9-fluorylamine hydrochloride was deposited, which when recrystallized melted at 255° and the free amine melted at 61–62°. The melting points of mixtures with authentic specimens were unaltered. Subsequent exposures of the benzene solution yielded 0.95 g. of fluorylidene-imine hydrochloride, m. p. 300–305°. The hydrochloride was converted to the free base by treatment with aqua ammonia and crystallized from hexane, yellow needles, m. p. and mixed m. p. with fluorylidene-imine 124°. Upon concentration of the benzene filtrate to a brownish red oil and dilution with alcohol it yielded 0.55 g. of fluorenone ketazine, m. p. and mixed m. p. with an authentic specimen 269°. Attempts to identify other products in the oily residue were not successful.

Photochemical Decomposition of Fluorylidene-iminochloride.—This decomposition study was carried out in a manner similar to the one described above. From 7.8 g. of fluorylidene-iminochloride were obtained the following products in designated yields: 3.68 g. of fluorylidene-imine hydrochloride, 0.62 g. of fluorenone ketazine, 0.60 g. of fluorenone and 0.05 g. of dichlorodibiphenylene-ethane,⁴ m. p. 236°.

Anal. Calcd. for C₂₈H₁₆Cl₂: C, 78.18; H, 4.04. Found: C, 78.21; H, 4.20.

Fractionation of the residual oil did not yield any other identifiable products.

(3) Peterson, *Am. Chem. J.*, **46**, 325 (1911).

(4) Schmidt and Wagner, *Ber.*, **43**, 1796 (1910).

BUREAU OF PLANT INDUSTRY
SOILS AND AGRICULTURAL ENGINEERING
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NEW COMPOUNDS

2-(*p*-Chlorophenyl)-1-(1-diethylamino-4-pentyl)-5-methoxybenzimidazole

N-(1-Diethylamino-4-pentyl)-2-nitro-4-methoxyaniline¹ (7.3 g., 0.024 mole) was dissolved in 20 cc. of dry ether and reduced at room temperature in the presence of Raney nickel under an initial pressure of 2 atmospheres of hydrogen. Complete reduction was indicated by the complete loss of the original deep red color coincident with the theoretical consumption of hydrogen. The resulting solution² was dried over potassium carbonate and filtered free of drying agent and catalyst. To this solution was added 25 cc. of dry pyridine and 4.6 g. (0.026 mole) of *p*-chlorobenzoyl chloride, and after allowing to stand for one hour, the mixture was heated overnight on a steam-bath under an air-cooled condenser. Dilute alkali was added, the product was extracted with ether, and the ethereal solution was dried over potassium carbonate and distilled through a small Claisen flask having a heated Vigreux column. The product was obtained in a yield of 5 g. (52% yield) of a red-brown oil boiling at 240° (3 mm.).

(1) Clemo and Swan, *J. Chem. Soc.*, 274 (1944).

(2) Since previous experience had indicated that the hydrogenation proceeded quantitatively and that the resulting amine was unstable, the ethereal solution was used directly for preparation of the benzimidazole.

Anal. Calcd. for C₂₃H₃₀ClN₃O: N, 10.51. Found: N, 10.34.

THE VENABLE CHEMICAL LABORATORY
UNIVERSITY OF NORTH CAROLINA
CHAPEL HILL, NORTH CAROLINA

R. L. MCKEE³

R. W. BOST

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(3) Wm. S. Merrell Co., Postdoctoral Fellow.

1-(3-Diethylamino-1-propyl)-6-methoxy-1,2,3,4-tetrahydroquinoline

To a boiling solution of 21.5 g. (0.132 mole) of 6-methoxy-1,2,3,4-tetrahydroquinoline¹ (thalline) in 200 cc. of 95% alcohol was added dropwise over a period of three hours 15.0 g. (0.101 mole) of 3-diethylaminopropyl chloride.² After refluxing for twelve hours, the solvent was distilled and the residue was heated to 100° for six hours (oil-bath). The dark oil was treated with dilute sodium hydroxide, extracted with ether, and the ethereal solution was dried over sodium sulfate. After removal of the solvent, the oil was distilled under diminished pressure.

A small fore-run was collected, the main portion of the product distilling from 157 to 165° (3 mm.). Redistillation yielded a product boiling at 158° (3 mm.), weighing 22 g. (79% of the theoretical). The product was water-clear but began to darken quickly on exposure to air. Consequently, dry hydrogen chloride was passed through a dry ethereal solution, the dihydrochloride separating as a gum which solidified on chilling. The ether was decanted and the solid was recrystallized from dry methyl alcohol by the addition of dry acetone.

The product appeared as a white, crystalline powder, melting at 185–186° with evolution of gas. Its analysis was rendered difficult by its extremely hygroscopic nature.

Anal. Calcd. for C₁₇H₂₈N₂O·2HCl: N, 8.02. Found: N, 7.60.

THE VENABLE CHEMICAL LABORATORY
UNIVERSITY OF NORTH CAROLINA
CHAPEL HILL, NORTH CAROLINA

R. L. MCKEE³

M. K. MCKEE

R. W. BOST

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(1) Cromwell, Caughlan and Gilbert, *THIS JOURNAL*, **66**, 401 (1944).

(2) Goldfarb, *ibid.*, **63**, 2280 (1941); Marxer, *Helv. Chim. Acta*, **24**, 209 E (1941).

(3) Wm. S. Merrell Co., Postdoctoral Fellow.

Phenoxy and Hydroxylated Aliphatic Acids¹

ω -Phenoxyundecylic Acid.—A neutral solution of 106 g. (0.40 mole) of ω -bromoundecylic acid (m. p. 49–50°) in 400 cc. of 75% aqueous alcohol was added dropwise to a boiling solution of 40 g. (0.42 mole) of phenol and 26 g. of potassium hydroxide in 200 cc. of 75% alcohol. After refluxing overnight most of the alcohol was removed under reduced pressure. The residue was diluted with water and acidified to yield the desired product. After recrystallization from acetic acid there was obtained 83.5 g. (75%) of crystals melting at 76–77°.

Anal. Calcd. for C₁₇H₂₈O₃: C, 73.4; H, 9.5; neut. equiv., 278. Found: C, 73.4; H, 9.4; neut. equiv., 276.

ω -(*p*-Chlorophenoxy)-undecylic Acid.—In a like manner from 68 g. (0.25 mole) of ω -bromoundecylic acid and 33 g. (0.26 mole) of *p*-chlorophenol there was obtained 54.5 g. (68%) of product, m. p. 72–73°.

Anal. Calcd. for C₁₇H₂₅O₃Cl: C, 65.2; H, 8.1; Cl, 11.3; neut. equiv., 312.5. Found: C, 65.5; H, 8.1; Cl, 11.7; neut. equiv., 310.

ω -(*m*-Trifluoromethylphenoxy)-undecylic Acid.—By the same procedure as above this compound was obtained in

(1) These compounds were prepared under a contract between the B. F. Goodrich Company and The Ohio State Research Foundation.