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 reëxposure 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 $C_{26}H_{16}Cl_2$: 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.

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

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

BUREAU OF PLANT INDUSTRY
SOILS AND AGRICULTURAL ENGINEERING
U. S. DEPARTMENT OF AGRICULTURE
BELTSVILLE, MARYLAND

RECEIVED AUGUST 16, 1946

NEW COMPOUNDS

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

N-(1-Diethylamino-4-pentyl)-2-nitro-4-methoxyaniline[1] (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 solution[2] 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.).

Anal. Calcd. for $C_{23}H_{30}ClN_3O$: N, 10.51. Found: N, 10.34.

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RECEIVED SEPTEMBER 23, 1946

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[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.

[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-tetrahydroquinoline[1] (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 $C_{17}H_{28}N_2O\cdot 2HCl$: N, 8.02. Found: N, 7.60.

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RECEIVED NOVEMBER 25, 1946

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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 Acids[1]

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 $C_{17}H_{26}O_3$: 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 $C_{17}H_{25}O_3Cl$: 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

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

43% yield from *m*-trifluoromethylphenol and ω-bromoundecylic acid. The compound boiled at 195–199° at 1 mm. and when recrystallized from Skellysolve B it melted at 42–43°.

Anal. Calcd. for neutral equivalent: 346. Found: 351.

Benzilidene-9,10-dihydroxystearic Acid.—Dry hydrogen chloride was passed into a solution of 24 g. (0.076 mole) of 9,10-dihydroxystearic acid and 9 g. (0.084 mole) of benzaldehyde in 300 cc. of dry benzene. The solution was refluxed and the water removed as formed. On distillation there was obtained 21 g. (68%) of product boiling at 240° at 0.1 mm.

Anal. Calcd. for $C_{25}H_{40}O_4$: C, 74.2; H, 9.9; neut. equiv., 405. Found: C, 74.0; H, 10.0; neut. equiv., 401.

***o*-Chlorobenzilidene-9,10-dihydroxystearic Acid.**—This compound was prepared by the same procedure as above. From 75 g. (0.24 mole) of 9,10-dihydroxystearic acid and 35 g. (0.25 mole) of *o*-chlorobenzaldehyde there was obtained 63.7 g. (62%) of product boiling at 245–250 at 0.1 mm.

Anal. Calcd. for $C_{25}H_{39}ClO_4$: C, 68.4; H, 8.9; Cl, 8.1. Found: C, 68.8; H, 9.1; Cl, 7.7.

Dilauryl Mercaptal of 4-Aldehydophenoxyacetic Acid.—Dry hydrogen chloride was passed into a solution of 22 g. (0.12 mole) of 4-aldehydophenoxacetic acid and 50 g. (0.25 mole) of lauryl mercaptan in 250 cc. of dioxane. The solution was refluxed for one hour and then poured into ice water. The crystals were collected and after recrystallization from alcohol melted at 70–74°; yield, 41 g. (54%). An analytical sample recrystallized from Skellysolve B melted at 74–75°.

Anal. Calcd. for $C_{33}H_{58}O_3S_2$: C, 69.8; H, 10.3; neut. equiv., 566. Found: C, 69.7; H, 10.6; neut. equiv., 568.

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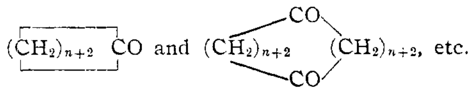
RECEIVED OCTOBER 7, 1946

COMMUNICATIONS TO THE EDITOR

A NEW SYNTHETIC METHOD FOR MANY-MEMBERED CARBON RINGS

Sir:

The recently described method for obtaining ketene dimers[1] made it seem likely that under conditions of high dilution an appropriate bifunctional ketene should undergo self-condensation to form macrocyclic diketenes readily convertible to many-membered carbocyclic ketones. Thus, a bifunctional ketene such as O=C=CH—$(CH_2)_n$—CH=C=O should afford, after dimerization followed by hydration and decarboxylation, cyclic ketones of the type



$(CH_2)_{n+2}CO$ and $(CH_2)_{n+2}\overset{CO}{\underset{CO}{}}(CH_2)_{n+2}$, etc.

The usefulness of this method has been tested employing sebacyl chloride as the starting material for the preparation of a bifunctional ketene of the type described above. From a consideration of the relative ease of forming many-membered carbon rings,[2] this ketene ($n = 6$) should cyclize to yield ultimately cycloöctadecane-1,10-dione as the principal product.

To a stirred, refluxing mixture of 7.2 g. (0.071 mole) of triethylamine, 0.4 g. of triethylamine hydrochloride and 500 cc. of absolute ether was added during thirty-eight hours 4.403 g. (0.0184 mole) of sebacyl chloride in 200 cc. of absolute ether. Stirring and refluxing were continued for four and one-half hours more. Then, after standing at room temperature overnight, all but 250 cc. of the ether was distilled. The remaining ether was separated from the precipitated amine salt by washing with water. A small amount of solid material (probably polymeric compounds) proved to be insoluble in both ether and water. The ether solution was washed once with 3 *N* hydrochloric acid and twice with water. After drying over magnesium sulfate, the ether was distilled, leaving a light yellow, mobile oil. To a solution of the oil in 10 cc. of absolute ethanol was added 3 g. (0.053 mole) of potassium hydroxide dissolved in 50 cc. of 95% ethanol. After refluxing on the steam-bath for three hours, the clear solution was acidified to litmus with dilute hydrochloric acid. To this was added 150 cc. of water and 100 cc. was then distilled in order to remove alcohol and any cyclononanone present. The distillate had a very pleasant odor, but attempts to isolate cyclononanone as the semicarbazone were unsuccessful. The residue from the steam distillation was made basic to phenolphthalein with 10% sodium carbonate and extracted with ether. Distillation of the dried ether layer left a small amount of pale yellow liquid, which crystallized into beautiful, large crystals on standing. Vacuum distillation gave 430 mg. (16%) of crude crystalline cycloöctadecane-1,10-dione, collected from 135–214° (0.4 mm.). The light yellow crystalline product was used directly in the preparation of the disemicarbazone (m.p. 225–227° dec. (cor.)) and the dioxime (m.p. 163–165° (cor.)). A sample of diketone after sublimation melted at 95–96° (cor.).[3]

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[1] Hanford and Sauer, "Organic Reactions," Vol. III, pp. 108–140 (1946). This excellent chapter presents a complete literature survey of the preparation of ketenes and ketene dimers.

[2] K. Ziegler and R. Aurnhammer, *Ann.*, **513**, 43–64 (1934).

[3] L. Ruzicka, *et al.*, *Helv. Chim. Acta*, **11**, 506 (1928). These workers report cycloöctadecane-1,10-dione (m. p. 96–97°); disemicarbazone (m. p. above 230° dec.); and dioxime (m. p. 166–168°).