with activated charcoal, and reprecipitated with water. Recrystallization from aqueous ethanol gave 1.3 g. (72%) of 1,1-dichloro-2,2-bis-(*p*-*t*-butylphenyl)-ethylene (II), m. p. 137.6–138.6° (cor.).

Anal. Calcd. for $C_{22}H_{26}Cl_2$: Cl, 19.62. Found: Cl, 19.79.

To a refluxing solution of 199 mg. of the olefin II in 10 ml. of glacial acetic acid, 200 mg. of chromium trioxide was added portionwise. Refluxing was continued for one hour. The reaction mixture was cooled and poured onto ice; the resulting solid was washed by decantation with water, dilute sodium hydroxide solution, and water again. The solid was filtered, dried and recrystallized from methanol. The product, di-*p*-*t*-butylbenzophenone, crystallized as needles melting at 134.1–135.5° (cor.); the yield was 70 mg. (43%).

Anal. Calcd. for $C_{21}H_{26}O$: C, 85.66; H, 8.90. Found: C, 85.78; H, 9.02.

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RECEIVED MARCH 11, 1946

p,p'-Dichlorobenzophenone Hydrazone

A mixture of 5.0 g. (0.02 mole) of *p,p'*-dichlorobenzophenone (m. p. 143–145°, Dow Chemical Company), 1.5 g. (0.025 mole) of 85% hydrazine hydrate (Eastman Kodak Company), and 45 ml. of dry isopropanol was heated in a sealed tube[1] at 150–160° for seven hours. The solvent was evaporated on the steam-bath and the residue crystallized from 1–1 carbon tetrachloride–petroleum ether (60–70°) to give 3 g. of small yellow crystals melting 91–93°; this is 56% of the theoretical yield. The compound did not form at reflux temperature, and the yield was only about 25% when absolute ethanol was used instead of isopropanol at 150–160°.

Anal. Calcd. for $C_{13}H_{10}Cl_2N_2$: Cl, 26.79. Found: Cl, 26.72.

This derivative was characterized by a Wolff-Kishner reduction to di-(*p*-chlorophenyl)-methane. A mixture of 2.23 g. (0.008 mole) of the hydrazone and sodium isopropylate prepared from 0.33 g. (0.014 atom) of sodium and 15 ml. of dry isopropanol was heated in a sealed tube at 160° for three hours. After evaporation of the solvent and crystallization of the residue from absolute ethanol, 0.4 g. (21%) of di-(*p*-chlorophenyl)-methane melting 54–55° was obtained. This product showed no depression in melting point when mixed with an authentic sample prepared according to Montagne.[2]

(1) Grummitt and Hall, THIS JOURNAL, **66**, 1229 (1944).
(2) Montagne, *Rec. trav. chim.*, **25**, 379 (1906).

SHERWIN-WILLIAMS LABORATORY
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RECEIVED MARCH 2, 1946

Tris-(hydroxymethyl)-phosphine Oxide Trilaurate

Tetra-(hydroxymethyl)-phosphonium chloride formed by the action of phosphine on a solution of hydrogen chloride and formaldehyde was converted to tris-(hydroxymethyl)-phosphine oxide by means of barium carbonate. Both reactions followed published procedures of Hoffman.[1] After removal of excess barium as sulfate, a concentrated aqueous solution of the oxide was subjected to a Schotten–Baumann reaction[2] with a slight excess of lauroyl chloride. The crude ester, obtained in 50% yield, was recrystallized from ether–petroleum ether and from methanol–benzene to give feathery crystals with a constant m. p. 65.5–66.5°.

Anal. Calcd. for $(C_{11}H_{23}COOCH_2)_3PO$: P, 4.51. Found (L. M. White): P, 4.36.

The crystallized product is soluble readily in benzene or chloroform, moderately in ether, and slightly in petroleum ether or methanol. It is insoluble in water.

(1) A. Hoffman, THIS JOURNAL, **43**, 1684 (1921); *ibid.*, **52**, 2995 (1930).
(2) Shriner and Fuson, "Identification of Organic Compounds," 2nd ed., John Wiley and Sons, Inc., New York, N. Y., p. 47.

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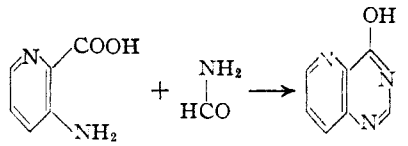
RECEIVED MARCH 5, 1946

4-Hydroxypyrido[3,2-*d*]pyrimidine[1]



β-Amino-α-picolinic acid was prepared from quinolinic acid essentially according to the directions of Sucharda.[2] A 13-g. sample was heated with 8 g. of formamide at 120–130° for two and one-half hours, at 160° for one-half hour and at 170–180° for two hours. The crystalline solid formed (4.3 g., 31%) was collected and washed with water. It separated from aqueous alcohol as white crystals, m. p. 346–347°.

Anal. Calcd. for $C_7H_5N_3O$: C, 57.15; H, 3.43; N, 28.56. Found: C, 57.12; H, 3.54; N, 28.64.

A number of attempts to replace the hydroxyl group with a chlorine atom by the usual procedures failed.

(1) The work reported here was carried out under a contract, recommended by the Committee on Medical Research, between the Office of Scientific Research and Development and the University of Illinois.
(2) Sucharda, *Ber.*, **58**, 1727 (1925).

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RECEIVED FEBRUARY 4, 1946

6-Methoxy-8-(9-diethylamininononylamino)-quinoline Dihydrobromide[1]

Nonamethylene chlorohydrin, b. p. 100–110° (2 mm.), m. p. 26° (lit.,[2] 28°), was prepared in 60% yield essentially according to the directions of Bennett and Mosses.[2] Treatment with diethylamine under pressure at 140° produced the aminoalcohol in 80% yield, b. p. 130–132° (3 mm.), $n^{20}D$ 1.4572 (lit.,[3] b. p. 161.5° (12 mm.), $n^{19}D$ 1.4574). This was converted to the bromide hydrobromide by boiling 42% hydrobromic acid followed by removal of the excess hydrobromic acid by evaporation under diminished pressure at 95°. The salt was then condensed with 6-methoxy-8-aminoquinoline by boiling in a small amount of absolute ethanol. The product, obtained in 53% yield from the aminoalcohol,[4] boiled at 210–214° (0.5 mm.), $n^{20}D$ 1.5562 (lit.,[3] b. p. 218° (0.5 mm.), $n^{17.2}D$

(1) The work reported here was carried out under a contract, recommended by the Committee on Medical Research, between the Office of Scientific Research and Development and the University of Illinois.
(2) Bennett and Mosses, *J. Chem. Soc.*, 1697 (1931).
(3) Altman, *Rec. trav. chim.*, **57**, 941 (1938).
(4) Altman (ref. 3) converted the aminoalcohol to the aminoalkyl chloride with thionyl chloride. The subsequent condensation was then carried out in a sealed tube at 130 to 170°; no yield was reported.