$\alpha\text{-}(\text{formylamino})\text{-pyridine}, \ \text{was} \ 50\%; \ \text{b. p.} \ 135\text{-}137^\circ$ (2 mm.). The hydrochloride melted at 112-114°.

Anal. Calcd. for $C_{11}H_{18}N_2$ ·HC1: Cl, 16.53. Found: Cl, 16.66.

 α -(β '-Cyclohexylethylamino)-pyridine.—Since the crude product was obtained in a solid state it was recrystallized from ether; m. p. 97–98°; yield 38%. The hydrochloride, purified as described for the ethylamino derivatives, melted at 124–126°.

Anal. Calcd. for $C_{13}H_{20}N_2 \cdot HC1$: Cl, 14.74. Found: Cl, 14.74.

 α -(β' -Phenylethylamino)-pyridine.—The base, obtained in 31.5% yield, boiled at $145-148^{\circ}$ (2 mm.). The chloroplatinate melted at $175-176^{\circ}$.

Anal. Caled. for 2 $(C_{13}H_{14}N_2)\cdot H_2PtCl_6$: Pt, 24.2. Found: Pt, 24.6.

1,5-Di-(α -pyridylamino)-pentane.—The sodium derivative, obtained from 27 g. (0.22 mole) of formylaminopyridine, 10 g. (0.25 mole) of sodium amide and 100 cc. of toluene, was heated with 26 g. (0.11 mole) of 1,5-dibromopentane for twenty hours on a steam-bath. The precipitated sodium bromide was removed, the toluene distilled from the filtrate under reduced pressure, and the dark brown, liquid residue, 1,5-di-(α -pyridylformylamino)-pentane, hydrolyzed by heating it with 14 g. of potassium hydroxide, dissolved in 150 cc. of 95% alcohol, for twelve hours. The alcohol was distilled under reduced pressure, water added to the crystalline residue to dissolve inorganic compounds and aminopyridine, the product filtered, washed thoroughly with water, dried and washed with ether. The light yellow pentane weighed 12.5 g. (42%). After recrystallization from methyl alcohol it melted at 147–148°; Sharp¹¹ reported 150°.

1,5-Di-(ethyl- α -pyridylamino)-pentane.—Twenty-one grams (0.166 mole) of α -(ethylamino)-pyridine, dissolved in 150 cc. of dry toluene, was heated with 8 g. (0.205 mole) of powdered sodium amide on a steam-bath for one hour. Ammonia was evolved, and a clear solution was obtained. Twenty grams (0.083 mole) of pentamethylene bromide was added, whereupon a vigorous reaction took place, and sodium bromide began to precipitate. After the mixture had been heated on a steam-bath for twenty-four hours, the precipitate was filtered and the solvent removed; b. p. $185{-}195^{\circ}~(0.02~mm.)\,;\,$ most of the pentaue boiled at 194–195° $(0.02~mm.)\,;\,$ yield 9.5 g. (34.5%).

The base (9.5 g.) was dissolved in dry ether and the solution treated with hydrogen chloride. When the precipitated, gummy hydrochloride was rubbed under ether it became solid. It was dissolved in absolute alcohol, the solution shaken with Norite, filtered, and dry ether added to the filtrate until it became turbid. The mixture was then placed in a refrigerator. The precipitated, crystal-line dihydrochloride weighed 6 g.; m. p. 177-179°.

Anal. Calcd. for $C_{19}H_{28}N_4$ ·2HC1: Cl, 18.42. Found: Cl, 18.45.

1,5-Di-(butyl- α -pyridylamino)-pentane.—This compound and the following one were prepared in the same manner as the corresponding ethyl derivative. From 24 g. of α -(butylamino)-pyridine there was obtained 10.6 g. (36%) of the pentane; b. p. 225-228° (0.02 mm.).

The sulfate separated as a gum when sulfuric acid, dissolved in absolute ether, was added to the base dissolved in the same solvent. The gum became solid when rubbed under ether. The salt was purified by the use of alcohol and ether in the manner described above; m. p. 170-171°.

Anal. Calcd. for $C_{23}H_{36}N_4 \cdot 2H_2SO_4$: S, 11.35. Found: S, 11.22.

1,5-Di-(hexyl- α -pyridylamino)-pentane.—The dihydrochloride melted at 93-94°.

Anal. Calcd. for $C_{27}H_{44}N_{4}$ ·2HC1: Cl, 14.26. Found: Cl, 14.15.

Summary

 α -Ethylamino-, α -butylamino-, α -hexylamino-, α -(β '-cyclohexylethylamino)-, α -(β '-phenylethylamino)-pyridine and 1,5-di-(α -pyridylamino)-pentane have been obtained by interaction of sodium α -formylaminopyridine and the required alkyl halide or alkylene dihalide.

The sodium derivatives of the alkylaminopyridines reacted with 1,5-dibromopentane to yield 1,5-di-(alkyl- α -pyridylamino)-pentanes.

ANN ARBOR, MICHIGAN RECEIVED FEBRUARY 20, 1946

NOTES

The Identification of Trimesic Acid from the Oxidation of s-Trialkyl Aromatic Hydrocarbons

By. J. V. Karabinos, J. I. Wright and H. F. Hipsher

Since permanganate oxidation of s-trialkyl aromatic hydrocarbons gives the water soluble trimesic acid, we have found it convenient to isolate two derivatives with good melting points by the following procedure.

Two grams of hydrocarbon was refluxed with stirring for twelve hours in 500 cc. of water containing 20 g. of potassium permanganate. After filtration and acidification of the filtrate with hydrochloric acid the solution was evaporated to dryness on a steam-bath. After several dryings with absolute ethanol the crystalline residue was divided into two portions and one refluxed with 100 cc. of methanol containing 5% of dry hydrogen chloride for six hours and the other with ethanol-hydrogen chloride. Each was cooled and a small portion of the solvent removed *in vacuo* and the respective esters crystallized on further cooling. In some cases recrystallization of the esters from their respective alcohols was unnecessary. The m. p.'s of the esters from several *s*-trialkyl benzenes as well as literature values are listed below.

Hydrocarbon	M. p. of trimesates, °C. Trimethyl Triethyl
Mesitylene	144-144.5
1,3-Dimethyl-5-ethylbenzene	143-145 134
3,5-Diethyltoluene	143-144.5
1,3,5-Triethylbenzene	144-144.5 133-134
Literature values	143–144 ^a 132–133 ^b
^a Pechmann, Ann, 264, 296	(1891). ^b Shorger, THIS

^a Pechmann, Ann, 264, 296 (1891). ^b Shorger, THIS JOURNAL, 39, 2677 (1917).

Aircraft Engine Research Laboratory National Advisory Committee for Aeronautics Cleveland, Ohio Received January 23, 1946