thallium is 147°, and distillation should be carried out at reduced pressure to avoid explosive decomposition. Some other physical, and some chemical properties are described.

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[14] Original manuscript received November 16, 1942.

NOTES

Iodinated Dialkylaminoalkyl Pyridyl Ethers

BY ALFRED BURGER AND MARIE S. BAILEY

The substitution of the hydroxypyridine for hydroxyphenyl groups in halogenated derivatives of these ring systems has proved a valuable lead in improving bactericidal properties in a number of cases. We hoped, therefore, that replacement of the benzene ring in certain tuberculostatic halogenated dialkylaminoalkyl phenyl ethers[1] by pyridine would produce more highly active compounds. The few examples reported below did not support this expectation; none of the derivatives was tuberculostatic *in vitro*. An analogous basic ether of 5,7-diiodo-8-hydroxyquinoline, as well as 2,5-diiodo-6-hydroxy-nicotinic acid were also inactive.

Experimental

Diiodo-3-hydroxypyridine and triiodo-3-hydroxypyridine were prepared by the method of Binz and Maier-Bode.[2] Both phenols were methylated with diazomethane in ether–methanol solution.

Diiodo-3-methoxypyridine, obtained as colorless shiny crystals in a yield of 90%, melted at 100.5–101°.

Anal. Calcd. for $C_6H_5I_2NO$: N, 3.88. Found: N, 3.88.

Triiodo-3-methoxypyridine crystallized from dilute methanol as colorless, shiny needles, m. p. 113°. The yield was 99%.

Anal. Calcd. for $C_6H_4I_3NO$: N, 2.89. Found: N, 2.73.

2,5-Diiodo-6-hydroxynicotinic Acid.—A solution of 8.15 g. of iodine and 9 g. of potassium iodide in 20 cc. of water was added dropwise to a hot mechanically stirred solution of 10 g. of 6-hydroxynicotinic acid[3] in 125 cc. of 20% ammonium hydroxide. The reaction mixture was filtered from a greenish precipitate, and the filtrate acidified with acetic acid. The resulting brown precipitate was recrystallized three times from a 1:1 mixture of dioxane and ethanol. The shiny yellow needles melted at 242–249° (dec.).

Anal. Calcd. for $C_6H_3I_2NO_3$: N, 3.57. Found: N, 3.82.

Diiodo-3-(2-diethylaminoethoxy)-pyridine.—One and eight-tenths grams of diiodo-3-hydroxypyridine was dissolved in a mechanically stirred solution of 0.07 g. of sodium in 20 cc. of methanol. A solution of diethylaminoethyl chloride, prepared by dissolving 1 g. of diethylaminoethyl chloride hydrochloride in a solution of 0.1 g. of sodium in 10 cc. of methanol, was added, and the mixture refluxed for eighteen hours. The solvent was removed under reduced pressure, the oil dissolved in 75 cc. of ether, and washed with two 35-cc. portions of dilute sodium hydroxide solution and then with water. The ether solution was dried over anhydrous sodium sulfate, the ether distilled, and the residual oil converted to its **dihydrochloride** in acetone solution. Two recrystallizations from methanol furnished a colorless salt of m. p. 204–205°. The yield was 0.7 g. (26%).

Anal. Calcd. for $C_{11}H_{16}I_2N_2O \cdot 2HCl$: N, 5.40. Found: N, 5.50.

Diiodo-3-[(2-methylpiperidino)-3-propoxy]-pyridine Dihydrochloride was prepared in an analogous manner, using a 30% excess of 3-(2-methylpiperidino)-propyl chloride hydrochloride, and the corresponding amounts of the other reagents. The salt, obtained in a yield of 31% after crystallization from methanol, melted at 129–129.5°.

Anal. Calcd. for $C_{14}H_{20}I_2N_2O \cdot 2HCl$: N, 5.08. Found: N, 5.23.

5,7-Diiodo-8-[3-(2-methylpiperidino)-propoxy]-quinoline.—To a mechanically stirred solution of 0.6 g. of sodium in 20 cc. of methanol and 100 cc. of dioxane was added 5 g. of 5,7-diiodo-8-hydroxyquinoline and 3.25 g. of 3-(2-methylpiperidino)-propyl chloride. After boiling under reflux for nine hours, the mixture was worked up in the customary manner. The dihydrochloride crystallized from benzene and melted at 127–128.5°. The yield was 0.9 g. (11.4%).

Anal. Calcd. for $C_{18}H_{22}I_2N_2O \cdot 2HCl$: N, 4.59. Found: N, 4.58.

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[1] Burger, Wilson, Brindley and Bernheim, THIS JOURNAL, **67**, 1416 (1945).

[2] Binz and Maier-Bode, *Z. angew. Chem.*, **49**, 486 (1936).

[3] We are indebted to Dr. W. A. Lott for this material.

The Intermetallic Compound $Mg_4Na_4Pb_3$

BY GEORGE CALINGAERT, HYMIN SHAPIRO AND IVAR T. KROHN

In the course of a thermal investigation of the ternary system Mg–Na–Pb, the alloy $Mg_4Na_4Pb_3$ was found to be a compound with an open maximum. Melts were made in 200-g. batches under an atmosphere of nitrogen in a closed iron bomb fitted with a thermocouple well. The bomb was held at 725° for forty-five minutes with intermittent shaking. The alloy was then allowed to cool at the constant rate of 1° per minute, the rate of cooling being controlled by reduction of the heat input through a calibrated rheostat system. Temperature measurements were made on a Leeds and Northrup K-2 potentiometer, using thermocouples calibrated against the freezing point of lead. The cooling curves were interpreted after the manner of Tammann.[1]

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[1] Tammann, "A Textbook of Metallography," The Chemical Catalog Co., Inc., New York, 1925, p. 181.