

Diacyl Derivatives of 4,4''-Diamino-*o*-Terphenyl

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RECENTLY during the preparation of 4,4''-diamino-*o*-terphenyl we experienced the same lack of stability as reported by Allen and Pingert (1). The diamine could be stored only as the hydrochloride salt. Attempts to purify the amine for a melting point determination were not successful and a literature search indicated that no derivatives had been listed. The preparation of the diacetyl and dibenzoyl derivatives are reported now as an aid to future identification.

EXPERIMENTAL

4,4''-Diamino-*o*-terphenyl dihydrochloride (I). The diamine was prepared by the catalytic reduction of 19.5 grams of 4,4''-dinitro-*o*-terphenyl (1) in 275 ml. of dioxane. The reduction was run at room temperature with hydrogen at a cold pressure of 50 p.s.i. and 0.2 gram of platinum oxide catalyst. When reduction was complete the solution was saturated with dry HCl gas. The precipitated salt was washed with ether and stored under vacuum; 20.0 grams (98.5%).

4,4''-Diacetamido-*o*-terphenyl (II). To a solution of 3 ml. of acetic anhydride in 50 ml. of benzene was added 2.0 grams of (I) and 4.0 grams of sodium acetate. After

refluxing the suspension for one hour the solid was removed by filtration. Extraction with hot absolute alcohol yielded the diamide as a granular solid, 0.45 gram. The analytical sample, m.p. 323.5–324°, was recrystallized from absolute ethanol. All melting points in this report are uncorrected.

Anal. Calcd. for $C_{22}H_{20}N_2O_2$: C, 76.73; H, 5.85. Found: C, 76.80; H, 5.67.

4,4''-Dibenzamido-*o*-terphenyl (III). A suspension of 1.0 grams of (I) 30 ml. of 10% NaOH and 5 ml. of benzoyl chloride was shaken at room temperature for 10 minutes. After washing the amorphous mass with ether, 1.2 grams of (III) was obtained. Solution of the amorphous solid was effected by warming with 75 ml. of methyl cellosolve and adding 20 ml. pyridine until the solid disappeared. The solution on cooling yielded pale yellow needles, m.p. 321–321.5°.

Anal. Calcd. for $C_{32}H_{24}N_2O_2$: C, 82.03; H, 5.16. Found: C, 81.89; H, 5.39.

LITERATURE CITED

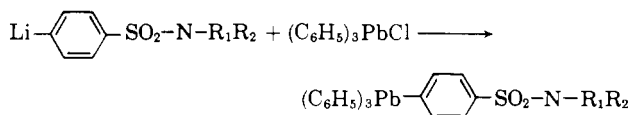
- (1) Allen, C.F.H., Pingert, F.P., *J. Am. Chem. Soc.* **64**, 2643 (1942).

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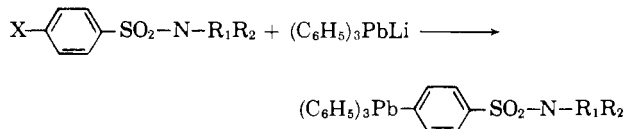
Organolead Compounds Containing the Sulfonamido Group

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THIS COMMUNICATION describes the preparation of triphenyllead(*N,N*-diethylbenzene) sulfonamide (I) and triphenylleadbenzenesulfonamide (II), Table I. Synthesis of I was accomplished by two methods. The first method,



was the reaction of *p*-lithio-*N,N*-diethylbenzenesulfonamide, prepared by the method of Gilman and Arntzen (1) with triphenyllead chloride, which gave I in 45% yield. The second method,



was the reaction of triphenylleadlithium, prepared by the

method of Gilman, Summers, and Leeper (2), with a halogen-containing compound. With *p*-iodo-*N,N*-diethylbenzenesulfonamide, the second method gave I in 30% yield (but, for unknown reasons, the desired reaction did not always occur). The second method never yielded compound II when attempted with *p*-bromobenzenesulfonamide. The failure may be presumed to result from the reaction of the active hydrogen of *p*-bromobenzenesulfonamide with the triphenylleadlithium. Compound II was prepared in a yield of 43% by the first method, i.e., by coupling *p*-lithio-*N*-lithio-*N*-phenylbenzenesulfonamide with triphenyllead chloride. Repeated trials of these two methods of coupling suggest that, for the synthesis of organolead compounds containing an active hydrogen, the first method is more dependable.

Because the preparation of *p*-lithio-*N*-lithio-*N*-phenylbenzenesulfonamide has not been reported previously, the halogen-metal interconversions between *p*-bromobenzenesulfonamide and *n*-butyllithium and between *p*-iodobenzenesulfonamide and *n*-butyllithium were studied. The yields of *p*-carboxybenzenesulfonamide (III) obtained under several conditions were determined by carbonation and are reported in Table II. [III melted at 277–279° with slight decomposition. Remsen, Hartman, and Mucken-

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