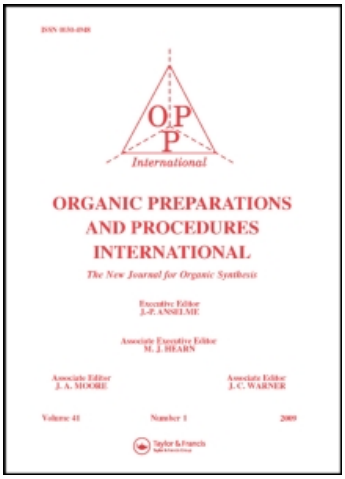

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REACTIONS OF DIPHENYLLEAD OXIDE, SYNTHESIS OF DIPHENYLLEAD DIACYLATES

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REACTIONS OF DIPHENYLLEAD OXIDE. SYNTHESIS OF DIPHENYLLEAD DIACYLATES

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$$Ph_2PbO + 2RCOOH \longrightarrow Ph_2Pb(OOCR)_2 + H_2O$$

Three synthetic routes are available for the synthesis of diphenyllead diacylates, $Ph_2Pb(OOCR)_2$. Tetraphenyllead reacts with organic acids[1], diphenyllead oxide (Ph_2PbO) with organic acids[2], and diphenyllead dichloride with alkali metal salts of organic acids[3] to yield diphenyllead diacylates under specific conditions. We were interested in determining the most generally applicable method to obtain diphenyllead diacylates.

The reaction of tetraphenyllead is slow with weak acids, and under some conditions the reaction temperatures required to obtain reasonable yields is high enough to cause thermal decomposition of the desired product.

In the reaction of diphenyllead dichloride with alkali salts of organic acids it is frequently difficult to separate the product from the alkali halide concomitantly formed.

The reaction of diphenyllead oxide with an organic acid proceeds smoothly, quantitatively and with no contaminating by-products.

$$Ph_2PbO + 2RCOOH \longrightarrow Ph_2Pb(OOCR)_2 + H_2O$$

Yields from reactions we have studied have thus far been quantitative.

The syntheses of some diphenyllead diacylates by the above reaction are reported in Table I. Future considerations will involve a variety of acids of varying strength such as octynoic, nonynoic, propiolic, acrylic, benzoylbenzoic, p-chloro-, p-bromo-, p-iodobenzoic and acetylene dicarboxylic acid and a large number of fluoro and perfluoro acids.

Experimental

Diphenyllead oxide was prepared by the method of Willemsens[4].

Preparation of Diphenyllead Diperfluorobenzoate: A well stirred mixture of diphenyllead oxide (3.8g) and pentafluorobenzoic acid (4.1g) in acetone (150 ml) was refluxed for 6-1/2 hours. The solution so obtained was filtered and the solvent was removed under reduced pressure. The white crystalline product obtained (7.3g) was recrystallized from acetone.

Preparation of Diphenyllead Dinaphthoxyacetate: A well stirred mixture of naphthoxyacetic acid (4.1g) and diphenyllead oxide (3.7g) in acetone (250 ml) was refluxed for 8 hours. The white precipitate obtained was filtered and dried to a white powder (7.2g) insoluble in common organic solvents.

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TABLE I - DIPHENYLLEAD DIACYLATES

Compound	Yield (%)	M.p. (°C)	Analysis (%) Found	Calcd
$(C_6H_5)_2Pb\left[OOCC{\equiv}C(CH_2)_2CH_3\right]_2$ [a]	92	132 dec.	C: 49.36 H: 4.11	49.39 4.14
$(C_6H_5)_2Pb\left[OOCC{\equiv}C(CH_2)_3CH_3\right]_2$ [a]	90	112 dec.	C: 51.0 H: 4.57	51.04 4.61
$(C_6H_5)_2Pb(OOCC_6F_5)_2$ [b]	94	231 dec.	C: 39.81 H: 1.31	39.85 1.29
$(C_6H_5)_2Pb\left[OOCC{\equiv}C\text{-}C_6H_5\right]_2$ [c]	95	160-164 dec.	C: 55.19 H: 3.11	55.29 3.09
$(C_6H_5)_2Pb(OOCCH_2OC_{10}H_7)_2$ [c]	96	226 dec.	C: 56.51 H: 3.60	56.60 3.69

a) Recrystallized from carbon tetrachloride; b) Recrystallized from acetone; c) Products insoluble in common organic solvents.

The typical infrared absorption for lead carboxylate was found in the region 1340-1670 cm^{-1} for all the compounds. For compounds 1, 2, and 4 the C≡C stretching band was found in the region 2150 - 2270 cm^{-1}.

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