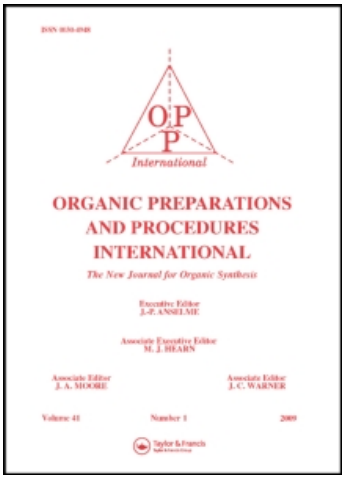

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Diphenyllead Dicarboxylates

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filtrate is reduced to one-half its volume and made strongly basic with sodium hydroxide pellets. The solution is extracted with two 100 ml. portions of methylene chloride, the extracts are dried over potassium carbonate, filtered, and evaporated to dryness to give 32-35g. of trans-I (98.4% pure by gc analysis[1]). The product readily forms a carbonate in air and is more conveniently stored as the dihydrochloride salt, mp. 336-338° (lit.[3] 338-339°).

Similarly, cis-II (92% pure) is converted to cis-I (90.5% pure). Dihydrochloride salt, mp. 308-311° (lit.[3] 312-314°).

References

1. Aldrich Chemical Co., 66% trans-, 29% cis- and 5% unidentified matter (by gc). Column: 1/8 in x 15 ft. packed with 10% DC-710 on Chromosorb W pre-treated with KOH.

2. A.I. Smith, U.S. Patent 3,163,675 (1964); Chem. Abstr., 62, 7656f (1965).

3. G. Swift and D. Swern, J. Org. Chem., 32, 511 (1967).

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The preparation of several additional diphenyllead dicarboxylates according to a previously described procedure is reported. The following table lists the yields obtained and some physical properties.

Table I. PHYSICAL PROPERTIES AND ANALYTICAL DATA OF DIPHENYLLEAD DICARBOXYLATES

Compound	Yield (%)	M.p. (°C)	Analysis (%)	Found	Calcd.
$(C_6H_5)_2Pb[OOC(C_6H_3F)]_2$ [a]	98	236-237	C:	48.39	48.83
			H:	3.01	2.81
$(C_6H_5)_2Pb[OOC(C_6H_3F)]_2 \cdot C_5H_5N$	98	246-247	C:	52.00	51.81
			H:	3.28	3.22
$(C_6H_5)_2Pb[OOC(C_6H_3F)]_2$ [a]	99	250-251	C:	49.13	48.83
			H:	2.80	2.81
$(C_6H_5)_2Pb[OOC(C_6H_4F)]_2$ [a]	98	238-240	C:	48.83	48.83
			H:	2.88	2.81
$(C_6H_5)_2Pb[OOC(C_6H_4F)]_2 \cdot (CH_3)_2SO$	99	238-239	C:	46.37	46.86
			H:	3.68	3.37
$(C_6H_5)_2Pb[OOC(CH_2)_2(C_6H_2F_3)]_2$ [b]	95	285-287	C:	42.49	42.91
			H:	2.10	2.16
$(C_6H_5)_2Pb[OOCCH_2(C_6H_3CF_3)]_2$ [b]	90	166-167	C:	46.65	46.93
			H:	2.95	2.88
$(C_6H_5)_2Pb[OOCCH_2(C_6H_4CF_3)]_2$ [b]	93	219-220	C:	46.92	46.93
			H:	2.74	2.88
$(C_6H_5)_2Pb[OOC(C_6H_3(CF_3)_2)]_2$ [a]	94	240-241	C:	40.79	41.15
			H:	2.01	1.84
$(C_6H_5)_2Pb[OOC(C_6H_3(CF_3)_2)]_2 \cdot 2(CH_3)_2SO$	94	238-240	C:	39.61	39.58
			H:	2.42	2.73

Table I. PHYSICAL PROPERTIES AND ANALYTICAL DATA OF DIPHENYLLEAD DICARBOXYLATES (continued)

Compound	Yield (%)	M.p. (°C)	Analysis (%) Found	Calcd.
$(C_6H_5)_2Pb[OOCCF_2CF_3]_2$[b]	94	247-24[illegible]	C: 31.29 H: 1.52	31.33 1.48
$(C_6H_5)_2Pb[OOCCH_2 \cdot \underset{\displaystyle CF_3}{\underset{\vert}{C}}H \cdot CH_3]_2$[b]	85	189-191	C: 39.21 H: 2.97	39.35 3.30
$(C_6H_5)_2Pb[OOCCH=\underset{\displaystyle CF_3}{\underset{\vert}{C}}CH_3]_2$[c]	90	176-178	C: 39.21 H: 2.69	39.58 2.72
$(C_6H_5)_2Pb[OOCCF_2Cl]_2$[b]	95	242	C: 30.69 H: 1.57	30.97 1.62

a) Purified via crystalline adduct;

b) Recrystallized from acetone;

c) Recrystallized from carbon tetrachloride.

The typical infrared absorption for lead carboxylate was found in the region 1340-1700 cm^{-1} for all the compounds.

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