

Supporting Information

Stannylplumbylenes: bonding between tetravalent tin and divalent lead

Christian Drost,* Peter Lönnecke and Joachim Sieler

Universität Leipzig, Institut für Anorganische Chemie, Johannisallee 29, 04103 Leipzig, Germany; E-mail: cdrost@uni-leipzig.de

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1. Experimental Details

All manipulations were performed using Schlenk line techniques under an atmosphere of dry nitrogen.

Preparation of $\text{Pb}[\text{C}_6\text{H}_3(\text{O}^i\text{Pr})_2\text{-2,6}]_2$ (**3**)

Lead(II)chloride (1.71 g, 6.2 mmol) was added in small portions to a solution of LiR (2.47 g, 12.3 mmol) in Et_2O (40ml) at 0°C . The suspension was stirred for 3 days at room temperature and subsequently filtered. After the removal of Et_2O in vacuo **3** was crystallised from hexane at -28°C to yield 2.44 g (67%). Characterization of **3**: orange-red crystalline solid; mp (nitrogen, sealed capillary): 94°C ; NMR (C_6D_6 , TMS, 300K): ^1H (400 MHz), $\delta = 1.09$ (d, $J = 6$ Hz, 24H, CHMe_2), 4.24 (sept, $J = 6$ Hz, 4H, CHMe_2), 7.09 (d, $J = 7.6$ Hz, 4H_{meta}), 7.18 (t, $J = 7.6$ Hz, 2H_{para}); $^{13}\text{C}\{^1\text{H}\}$ (100.6 MHz), $\delta = 22.9$ (CHMe_2), 69.8 (CHMe_2), 110.5, 162.3, 221.1 (C_{phenyl}); $^{207}\text{Pb}\{^1\text{H}\}$ (83.7 MHz), $\delta = 5859$; EI-MS: m/z (%): 594 (**3**) $[\text{M}]^+$, 551 (**5**) $[\text{M}-^i\text{Pr}]^+$.

2. Crystallographic Details

The crystallographic data were collected on a Siemens CCD diffractometer (SMART) at 223(2) K with Mo-K α radiation ($\lambda = 0.71073 \text{ \AA}$), solved by direct methods and refined by full-matrix least squares on F^2 .¹

Crystallographic data for **3**: empirical formula $C_{24}H_{34}O_4Pb$, $FW = 593.70$, crystal system monoclinic, space group Cc , $a = 14.645(3)$, $b = 14.514(3)$, $c = 11.599(2) \text{ \AA}$, $\alpha = 90$, $\beta = 93.68(3)^\circ$, $\gamma = 90^\circ$, $V = 2460.4(8) \text{ \AA}^3$, $Z = 4$, $\rho_{\text{calcd}} = 1.603 \text{ Mg m}^{-3}$, 6913 reflections collected, 3395 independent reflections ($R_{\text{int}} = 0.0530$), final R indices ($I > 2\sigma(I)$): $R_1 = 0.0383$, $wR_2 = 0.0945$, R indices (all data): $R_1 = 0.0457$, $wR_2 = 0.0996$.

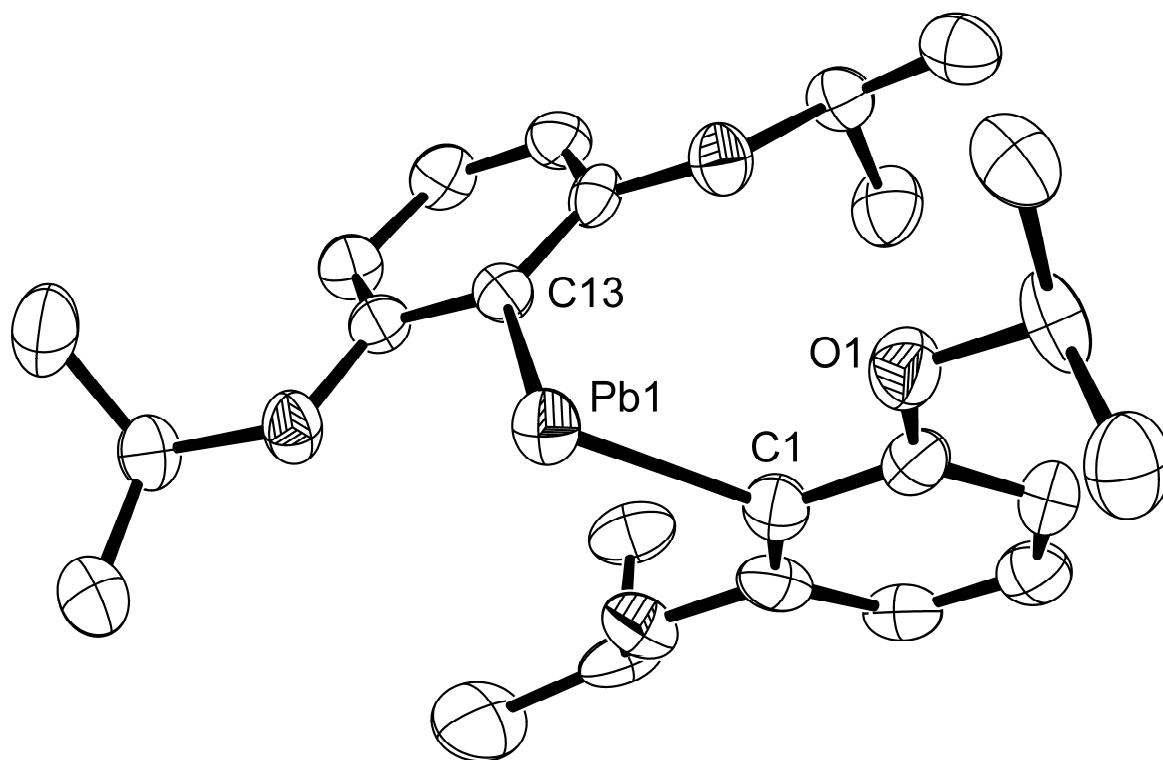


Figure S1: ORTEP representation of the molecular structure of **3** (40% thermal ellipsoids; hydrogen atoms omitted). Selected bond distances [\AA] and angles [$^\circ$]: Pb(1)–C(1) 2.253(10), Pb(1)–C(13) 2.276(10), Pb(1)···O(1) 3.000(8), C(1)–Pb(1)–C(13) 93.0(3).

Reference:

- 1 G.M. Sheldrick, *SHELXS-97* and *SHELXL-97*, Programs for crystal structure analysis, University of Göttingen, Germany, 1997.