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Stannylplumbylenes: bonding between tetravalent tin and divalent lead †‡

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Depending on stoichiometry, reactions of the mixed valence Sn(0)/Sn(III) compound $Sn(SnAr_3)_2$ (1) (Ar = $C_6H_4(O^iPr)_2$ -2,6) with the likewise substituted plumbylene PbAr₂ (3) afforded either the homoleptic distannylplumbylene Pb(SnAr_3)₂ (4) or the heteroleptic arylstannylplumbylene Pb(Ar)SnAr₃ (5), a valence isomer of a stannaplumbene.

Mixed valence homo- or heterometallic compounds of the heavier group 14 elements with direct metal to metal bonding received growing attention in recent years.¹ Compounds of type $RM-MR_3$ (M = Ge, Sn; R = organic group) have long been anticipated as transient valence isomers of dimetallenes and initially been proven by trapping reactions.² Only a few stable homobimetallic compounds have been prepared to date by either (i) combination of organic groups with high or low/high steric requirements $[Ar'GeGe(^{t}Bu)_{3} (Ar' = C_{6}H_{3}Mes_{2}-2,6;$ $Mes = C_6H_2Me_3-2,4,6$; Ar*SnSn(Ph)₂Ar*, Ar*SnSn(Me)₂Ar* $(Ar^* = C_6H_3Trip_2-2,6; Trip = C_6H_2^{i}Pr_3-2,4,6)],^{3,4}$ (ii) incorporating the M–M unit into stabilizing organic ring structures $(M = Sn, {}^{5a}Pb^{5b})$, or (iii) employing additional coordination of the divalent metal atoms.⁶ Heterobimetallic or homo-/heterotrimetallic compounds are even scarce and include $Ar'SnGe(^{t}Bu)_{3}^{3}$ or Sn(SnPh₂Ar*)₂,⁷ apart from the hypersilylated bisgermylstannylene and -plumbylene M[Ge(SiMe₃)₃]₂ (M = Sn, Pb),⁸ or N-heterocyclic carbene stabilized analogues, e.g. [{(Me)-C(ⁱPr)N₂C]Sn[Sn(SiMe₃)₃]₂.⁹ In contrast, dicoordinated plumbylenes with Sn-Pb bonds have not been described so far. Moreover, for organometallic compounds structurally authenticated bonding between tin and lead in general is limited to few hexaarylstannylplumbanes (e.g. Ph₃SnPbPh₃).¹⁰

Recently, we described the unforeseen isolation of the brownishred distannylstannylene $Sn(SnAr_3)_2 \mathbf{1} [Ar = C_6H_3(O^iPr)_2-2,6]$, which we obtained in a straightforward salt metathesis reaction of $SnCl_2$ with two equivalents of LiAr in Et₂O and crystallisation from hexane.¹¹ The formation of $\mathbf{1}$ was surprising and relevant, since the employment of only one type of aryl ligand in comparable reactions did thitherto not yield mixed valence

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tin compounds with direct metal to metal bonding, but led to homoleptic stannylenes SnR_2 ,¹² distannenes $(SnR_2)_2$ ¹³ or cyclotristannanes $(SnR_2)_3$,¹⁴ depending on steric bulk of R, respectively. Furthermore, compound 1—being monomeric and V-shaped in solid state—in solution appears to reversibly disintegrate into arylstannylstannylene Sn(Ar)SnAr₃ **2**. This was corroborated by ¹¹⁹Sn NMR solution studies of **1** in C₆D₆, revealing two signals in a 1 : 1 signal ratio at δ –14 and 1296 ppm only with ¹¹⁹Sn–¹¹⁹Sn coupling constants (¹J_{SnSn} = 9011 Hz).

The interconversion between the mixed valence compounds Sn(SnAr₃)₂ 1 and Sn(Ar)SnAr₃ 2 presumably occurs via an invertible aryl rearrangement and elimination/addition process of a transient stannylene [SnAr₂], which has not been detected so far. However, pertinent results to this work involving a phenyl group migration have been obtained by Power et al.: they described a reversible valence isomer equilibrium between the stannylene Sn(Ph)Ar* and the valence isomer of its dimer, the stannylstannylene Ar*SnSn(Ph)₂Ar* in solution.^{4c} Aryl migration as a part of ligand exchange between two different homoleptic stannylenes has also been exploited in the synthesis of heteroleptic stannylenes.¹⁵ Furthermore, our results parallel observations from Masamune and Sita, who established an interconversion between the cyclic trimer c-(SnTrip₂)₃ I in solid state and the corresponding distannene (SnTrip₂)₂ II in solution, ^{14b} considering $Sn(SnAr_3)_2$ 1 and $Sn(Ar)SnAr_3$ 2 as valence isomer analogues of I and II, respectively (Scheme 1).

Bearing in mind that 1 is subject to disintegrative processes in solution, we reacted 1 with the diarylplumbylene $PbAr_2 3$ in different stoichiometries. Whereas the reaction of 1 with 3 in a 2 : 3 ratio afforded the dark green crystalline distannylplumbylene $Pb(SnAr_3)_2 4$, the purple arylstannylplumbylene $Pb(Ar)SnAr_3 5$ was isolated from a reaction in a 1 : 3 ratio. Starting from either 5 or 4, both compounds may also be interconverted by addition of 1 or 3 in corresponding amounts, respectively (Scheme 2).§

Formally, the generation of 5 (or 4) can be rationalized by the reaction of a transient stannylene $[SnAr_2]$ with the plumbylene PbAr₂ 3 (or 5), yielding the corresponding



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[†] This communication is dedicated to Professor Reinhard Kirmse on the occasion of his 65th birthday.

[‡] Electronic supplementary information (ESI) available: Experimental details, analytical and crystal data with structure refinement for **3**. CCDC 864029 (**3**), 864030 (**4**) and 864028 (**5**). For ESI and crystallographic data in CIF or other electronic format see DOI: 10.1039/c2cc30687e



Scheme 3

stannylene-plumbylene adduct, which suffers aryl migration from lead to tin to yield the mixed valence compounds (Scheme 3 for **5**).

It is noteworthy to mention that attempts to synthesize 4 via the conventional salt metathesis route with two equivalents of lithium stannate Ar_3SnLi^{11} and $PbCl_2$ failed and resulted in the formation of elemental lead and Ar_3SnCl .

Compounds 3-5 have been characterised by multinuclear NMR-spectroscopy. Each of their ²⁰⁷Pb NMR spectra exhibit signals at $\delta = 5859$ (3), 7853 (4) and 6905 ppm (5); the ¹¹⁹Sn NMR spectra of the latter two compounds comprise resonances at $\delta = 1273$ (4) and 1270 ppm (5) relating to the tetravalent tin atoms, respectively. While for **5** an ¹¹⁹Sn-²⁰⁷Pb coupling $({}^{1}J_{\text{SnPb}} = 19971 \text{ Hz})$ is revealed, for **4** the ${}^{119}\text{Sn}{-}^{207}\text{Pb}$ coupling $({}^{1}J_{\text{SnPb}} = 22560 \text{ Hz})$ and additional ${}^{117}\text{Sn}{-}^{119}\text{Sn}$ satellites (${}^{2}J_{\text{SnSn}} = 3254$ Hz) are observed, confirming its structural integrity in solution. These ${}^{1}J_{\text{SnPb}}$ coupling values are markedly larger than those determined for hitherto isolated stannylplumbanes, e.g. $Ph_3SnPbPh_3$ (${}^1J_{SnPb} = 3469$ Hz).¹⁰ The ¹¹⁹Sn NMR stannyl group signals for 4 and 5 are located strikingly downfield ($\delta = ca.$ 1270 ppm) and in sharp contrast to the corresponding ¹¹⁹Sn NMR shift of Sn(Ar)SnAr₃ 2 $(\delta = -14 \text{ ppm})$, which is indicative of considerably deshielded lead-bonded tetracoordinated tin centres.

The ²⁰⁷Pb NMR resonance for **3** at $\delta = 5859$ ppm is in agreement with the generally observed range of ²⁰⁷Pb NMR chemical shifts for organolead(II) compounds, although at the upper end but still downfield of PbMes*[CH₂CMe₂C₆H₃(¹Bu)₂-2,5] (5067 ppm; Mes* = C₆H₂(¹Bu)₃-2,4,6),¹⁶ or Pb[C₆H₃(NMe₂)₂-2,6]₂ (3919 ppm),¹⁷ with the latter reflecting a higher than twocoordinate Pb(II) environment. Substitution of each aryl-ligand in **3** by SnAr₃-groups leads to a stepwise (*ca.* 1000 ppm) downfield shifting of the ²⁰⁷Pb NMR signal from $\delta = 5859$ (**3**) to 6905 (**5**) and 7853 ppm (**4**). The latter two values may be compared with the ²⁰⁷Pb NMR shift of the related arylsilylplumbylene Pb(R')Ar* [R' = Si(SiMe₃)₃],¹⁸ which is located more downfield at $\delta = 10745$ ppm, but consistent with $\delta = 7545$ ppm for Pb(R')(C₆HMe₃-4,5,6-¹Bu-2), being dimeric in solid state.¹⁶

Single crystals of complexes 4 and 5 suitable for X-ray diffraction were obtained from benzene (4) and hexane (5) solutions; their molecular structures are provided in Fig. 1 (4) and 2 (5) with selected bond lengths and bond angles given in the figure caption. The molecular structure of the distannylplumbylene 4 (Fig. 1) shows it to be mononuclear with a V-shaped



Fig. 1 Thermal ellipsoid plot (40% probability surface) of 4 (hydrogen atoms and methyl groups are omitted for clarity). Selected bond lengths (Å) and angles (°): Pb(1)–Sn(1) 2.9283(4), Pb(1)–Sn(2) 2.9483(6), Sn(1)–Pb(1)–Sn(2) 114.26(1), C_{ipso}–Sn(1)/Sn(2)–Pb(1) 97.65(8)–123.65(8).

Sn(1)–Pb(1)–Sn(2) skeleton and an interligand angle of 114.26(1)°. The Pb(1)–Sn(1)/Sn(2) distances amount to 2.9283(4)/2.9483(6) Å and the geometries at the two tetra-coordinated Sn(1)/Sn(2) atoms are heavily distorted with C_{ipso} -Sn(1)/Sn(2)–Pb(1) angles ranging from 97.65(8)° to 123.65(8)°. A peculiar feature—similar to the structural array of the tritin analogue **1**—is the distorted transoid arrangement of two oxygen atoms in proximity to the central lead atom with Pb(1)···O(6)/O(7) separations of 2.968(2) and 2.965(2) Å [O(6)/O(7)–Pb(1)–Sn(1)/Sn(2): 66.82(4)/66.31(4)°; O(6)–Pb(1)–O(7): 178.57(6)°].

Heteroleptic plumbylene **5** is also monomeric in solid state (Fig. 2) with a more relaxed Sn(1)–Pb(1)–C(37) interligand angle of 102.94(12)° and a slightly diminished Sn(1)–Pb(1) bond length of 2.8784(4) Å compared with **4**. In contrast, the Sn–Pb bond lengths determined for stannylplumbanes Ph₃SnPbPh₃ are significantly shorter with 2.809(2) and 2.848(2) for two independent molecules, respectively.¹⁰ The three innerligand angles $[C_{ipso}$ –Sn(1)–Pb(1)] at the tetracoordinated tin atom Sn(1) vary from 99.87(11)° to 125.60(12)°. In **5** only one intramolecular Pb(1)···O(1) separation becomes apparent with 2.876(3) Å.

The observed Pb···O distances for **4** and **5** are clearly within the sum of the van der Waals radii $(3.54 \text{ Å})^{19}$ but exceed the sum of their covalent radii $(2.14 \text{ Å})^{19}$ and also go beyond comparable



Fig. 2 Thermal ellipsoid plot (40% probability surface) of **5** (hydrogen atoms and methyl groups are omitted for clarity). Selected bond lengths (Å) and angles (°): Pb(1)–Sn(1) 2.8784(4), Pb(1)–C_{ipso} 2.285(4), Sn(1)–Pb(1)–C_{ipso} 102.94(12), C'_{ipso}–Sn(1)–Pb(1) 99.87(11)–125.60(12).

Pb···O distances described earlier, *e.g.* 2.518(2) Å for $[Pb\{C_6H_2[P(O)OEt_2]_2-2,6^{-t}Bu-4\}Cl]_n^{20}$ or 2.598(12) Å for $[Pb\{C(SiMe_3)_2(SiMe_2OMe)\}Cl]_2^{21}$ with the latter Pb···O contact regarded as not being particularly strong. Nevertheless—despite the long Pb···O distances in **4** and **5**, a distorted Pb···O arrangement and ²⁰⁷Pb NMR shifts adequate for dicoordinated lead(II) atoms—some weak interactions can possibly not be denied. Moreover, they may be supportive for the formation of mixed valence compounds with only one ligand-type, provided that three aryl groups can join together at the tetravalent tin atoms without steric constraints.

In conclusion, the reaction of the distannylstannylene $Sn(SnAr_3)_2 \mathbf{1}$ with the monomeric plumbylene $PbAr_2 \mathbf{3}$ afforded the dark green distannylplumbylene $Pb(SnAr_3)_2 \mathbf{4}$ and a purple arylstannylplumbylene $Pb(Ar)SnAr_3 \mathbf{5}$ depending on stoichiometric ratios, respectively. Their molecular structures feature unique bonding between tetracoordinated Sn and dicoordinated Pb. We deduce that 2,6-dialkoxyphenylene-based ligands have potential in heavy group 14 element chemistry²² and the presented compounds may be the forerunners of a series in mixed valence chemistry.

Notes and references

§ All manipulations were performed using Schlenk line techniques under an atmosphere of dry nitrogen. Synthesis of 4: a solution of 1 (1.44 g, 2.42 mmol) in benzene (20 ml) was added to a solution of 3 (2.45 g, 1.62 mmol) in an equal amount of benzene at 0 °C. The reaction mixture became dark green and was stirred for 30 min at 0 °C and further 5 h at room temperature. After filtration the filtrate was concentrated upon crystallization and set aside at room temperature and at 6 °C to afford dark green crystals of 4. A further crop of 4 was isolated from the mother liquid to give in total 3.6 g (93%). Characterization: mp (nitrogen, sealed capillary): 191 °C (decomp.); NMR (C₆D₆, TMS, 300 K): ¹H (400 MHz), δ = 0.81 (d, J = 6.0 Hz, 18H, CHMe₂), 0.93 $(d, J = 6.0 \text{ Hz}, 18\text{H}, \text{CH}Me_2), 1.01 (d, J = 6.0 \text{ Hz}, 18\text{H}, \text{CH}Me_2),$ 1.18 (d, J = 6.0 Hz, 18H, CHMe₂), 4.05 (q, J = 6.0 Hz, 4H, CHMe₂), 4.19 (q, J = 6.0 Hz, 8H, $CHMe_2$), 6.45 (d, J = 8.0 Hz, 6H_{meta}), 6.57 (d, J = 8.0 Hz, 6H_{meta}), 7.08 (t, J = 8.0 Hz, 6H_{para}); ¹³C{¹H} (100.6 MHz), $\begin{array}{l} (4,5) = 0.51 \, \text{H}_{2}, \text{M}_{\text{metal}}, \text{M}_{\text{metal}}, \text{M}_{\text{s}}, \text{M}_{2}, \text{M}_{2$ analysis calcd (%) for C₇₂H₁₀₂O₁₂PbSn₂: C 53.91, H 6.41; found (%): C 54.18, H 6.53. Synthesis of 5: crystalline 5 was prepared from 1 and 3 in a 3:1 stoichiometry as described above for the synthesis of 4. After filtration and removal of benzene the crystalline residue was recrystallized from hexane to get purple 5 in a total of 89% yield. Characterization: mp (nitrogen, sealed capillary): 134 °C (decomp.); NMR (C₆D₆, TMS, 300 K): ¹H (400 MHz), $\delta = 0.95$ (d, J = 6.0 Hz, 36H, CHMe₂), 1.02 (d, J =6.0 Hz, 12H, CHM₂), 4.19 (q, J = 6.0 Hz, 8H, CHM₂), 6.52 (d, J = 8.0 Hz, 6H_{meta}), 6.03 (d, J = 8.0 Hz, 2H_{meta}), 7.08 (t, J = 8.0 Hz, 3H_{para}); 7.15 (t, J = 8.0 Hz, 1H_{para}); ¹³C{¹H} (100.6 MHz), $\delta = 21.9$, 22.2 (CH₂Me), 68.8, 69.0 (CH₂Me), 104.5, 109.2, 145.1 (C_{ipso}Sn), 161.4, 163.7, 212.9 (C_{ipso}Pb) (phenyl); ¹¹⁹Sn{¹H} (149.2 MHz), $\delta = 1270 ({}^{1}J_{\text{SnPb}} = 19971 \text{ Hz}); {}^{207}\text{Pb}{}^{1}\text{H} (83.7 \text{ MHz}), \delta = 6905;$ EI-MS: m/z (%): 699 (62) [Ar₃Sn]⁺. Elemental analysis calcd (%) for C48H68O8PbSn: C 52.46, H 6.24; found (%): C 52.63, H 6.38. ¶ Crystallographic data for 4: empirical formula C₉₀H₁₁₁O₁₂PbSn₂,

FW = 1829.36, crystal system triclinic, space group PI, $\alpha = 12.272(2)$, $b = 15.377(3), c = 25.439(4) \text{Å}, \alpha = 96.850(3)^{\circ}, \beta = 102.309(3)^{\circ}, \gamma = 109.506(3)^{\circ}, V = 4325.5(12) \text{Å}^3, Z = 2, \rho_{calcd} = 1.405 \text{ Mg m}^{-3}, 53.958$ reflections collected, 22.639 independent reflections ($R_{int} = 0.0284$), final R indices ($I > 2\sigma(I)$): $R_1 = 0.0301, wR_2 = 0.0706, R$ indices (all data): $R_1 = 0.0490, wR_2 = 0.0801$. Data were collected on a Siemens CCD diffractometer (SMART) at 208(2) K with Mo-K_{\alpha} radiation ($\lambda = 0.71073$ Å). Crystallographic data for 5: empirical formula C₅₁H₇₅0₈PbSn, FW = 1141.99, crystal system monoclinic, space group P2(1)/n, $a = 26.9800(17), b = 12.5178(6), c = 31.719(2) \text{ pm}, \alpha = 90^{\circ},$ $\beta = 97.596(8)^{\circ}, \gamma = 90^{\circ}, V = 10618.3(11) \text{ Å}^3, Z = 8, \rho_{calcd} = 1.429 \text{ Mg m}^{-3}, 61.579$ reflections collected, 18.514 independent reflections $(R_{\rm int} = 0.0580)$, final *R* indices $(I > 2\sigma(I))$: $R_1 = 0.0296$, $wR_2 = 0.0574$, *R* indices (all data): $R_1 = 0.0575$, $wR_2 = 0.0608$. Data were collected on a Stoe-IPDS imaging plate diffractometer (ϕ scan mode) at 213(2) K with Mo-K_{α} radiation ($\lambda = 0.71073$ Å). All data were solved by direct methods and refined by full-matrix least squares on $F^{2,23}$

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