

Review

# Supramolecular aspects of tin and lead chemistry

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**Cross-fertilization between organometallic and supramolecular chemistry opens new ways of understanding and interpreting the solid state structures, leading to a distinct new discipline. Intermolecular interactions of various types occur in solid state, leading to self-assembly and self-organization of organometallic molecular species. Self-assembly through hydrogen bonds, dative (coordinate) bonds, soft-soft (secondary bond) and pi-bond interactions, illustrate the great diversity of the bonding motifs available for supramolecular self-assembly in organotin and organolead chemistry. Copyright © 2007 John Wiley & Sons, Ltd.**

**KEYWORDS:** supramolecular organometallic chemistry; self-assembly; secondary bonds; soft-soft interactions; organotin; organolead.

## INTRODUCTION

Contemporary chemistry has two continuously growing areas: organometallic chemistry and supramolecular chemistry. While *organometallic chemistry* is by now a classical field, *supramolecular chemistry*<sup>1–3</sup> is a young area, whose birth was certified through the Nobel prize awarded in 1987 to D.J. Cram, J.-M. Lehn and C.J. Pedersen ([http://nobelprize.org/nobel\\_prizes/chemistry/laureates/1987/](http://nobelprize.org/nobel_prizes/chemistry/laureates/1987/)). The two fields seem to grow independently, but cross-fertilization between organometallic and supramolecular chemistry opens new ways of understanding and interpreting solid-state structures, leading to a distinct new discipline.<sup>4,5</sup> Supramolecular structures have been identified even before the concepts of this new area of chemistry were defined and the new paradigms have not been used until more recently. Therefore, much of the earlier chemistry can be reconsidered in terms of the new discipline.

Supramolecular chemistry is defined as ‘the chemistry beyond the molecule’ dealing with ‘organized entities of higher complexity that result from the association of two or more chemical species held together by intermolecular forces’.<sup>2</sup> It deals with two types of ‘objects’: supermolecules, i.e. ‘well-defined oligomolecular species that result from the intermolecular association of a few components’, and

molecular assemblies or supramolecular arrays, which are ‘polymolecular systems that result from the spontaneous association of a non-defined number of components.’ Both are formed through noncovalent intermolecular forces of various types and are based upon one of two possible processes: host–guest complexation and self-assembly. This article will deal only with self-assembled structures.

The main types of intermolecular interactions leading to self-assembly and self-organization of molecular species, include hydrogen bonds,<sup>6–9</sup> dative coordinate bonds,<sup>10–15</sup> secondary bonds (or soft–soft interactions),<sup>16</sup> pi-bonds,<sup>17</sup> electrostatic (ionic) interactions, pi–pi stacking<sup>18</sup> and others.<sup>19</sup> Cooperativity, i.e. the simultaneous presence of different types of intermolecular interactions, is also observed, e.g. hydrogen bonds and dative bonds or electrostatic interactions (charge-assisted hydrogen bonds).<sup>20–23</sup> They will be illustrated with selected examples, mostly from the organometallic chemistry of Group 14 elements, and, when possible, from our own work.<sup>24</sup>

In Group 14 there is a marked difference between silicon and germanium (non-metals) on one side and tin and lead (soft metals) on the other hand. In organosilicon (just briefly mentioned) and organogermanium (less explored) chemistry, supramolecular self-assembly through hydrogen bonds (e.g. in silanols, germanols) and electrostatic interactions (e.g. in siloxanates) is determined by appropriate side groups and is predominant. Similar self-assembly occurs in organotin and organolead compounds, but these also tend

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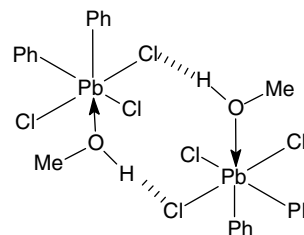
to self-assemble through dative (coordinate) bonds<sup>25,26</sup> and soft–soft (secondary bond) interactions, with participation of the central atom (tin, lead).

## HYDROGEN BOND SELF-ASSEMBLY

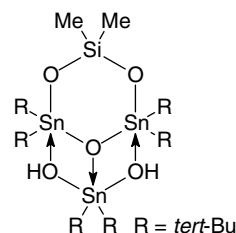
Silanols,  $R_3SiOH$ , silanediols,  $R_2Si(OH)_2$  and silanetriols,  $RSi(OH)_3$ , are all associated in solid state to form cyclic supermolecules, e.g. the tetramer  $[Ph_3SiOH]_4$ ,<sup>27</sup> hexamer  $[FcSiMe_2OH]_6$ ,<sup>28,29</sup> and many polymeric supramolecular arrays.<sup>30,31</sup> Associated supramolecular organogermanols (or hydroxoorganogermanes), e.g. tetrameric  $[Ph_3GeOH]_4$ ,<sup>32</sup> and polymeric  $[Bu_2^tGe(OH)_2]_x$  or  $[(HO)Fc(Bu^t)GeOGe(Bu^t)Fc(OH)]_x$  (dimeric or polymeric depending on the recrystallization solvent)<sup>33</sup> can be cited as examples of organogermanium supramolecular compounds formed through hydrogen bond self-assembly.

Organotin hydroxides, e.g.  $R_3SnOH$  ( $R = Me, Et, Ph$ ), behave differently. They are associated through hydroxo bridges, i.e. dative  $HO \rightarrow Sn$  bonds, to form supramolecular chains,<sup>34–36</sup> but those with bulky groups (e.g. mesityl) are monomeric. In many cases, however, the bridging hydroxo groups  $Sn(\mu-OH)Sn$  and other functional groups attached to tin can participate in additional hydrogen bonding schemes, leading to more complex supramolecular architectures.<sup>37</sup>

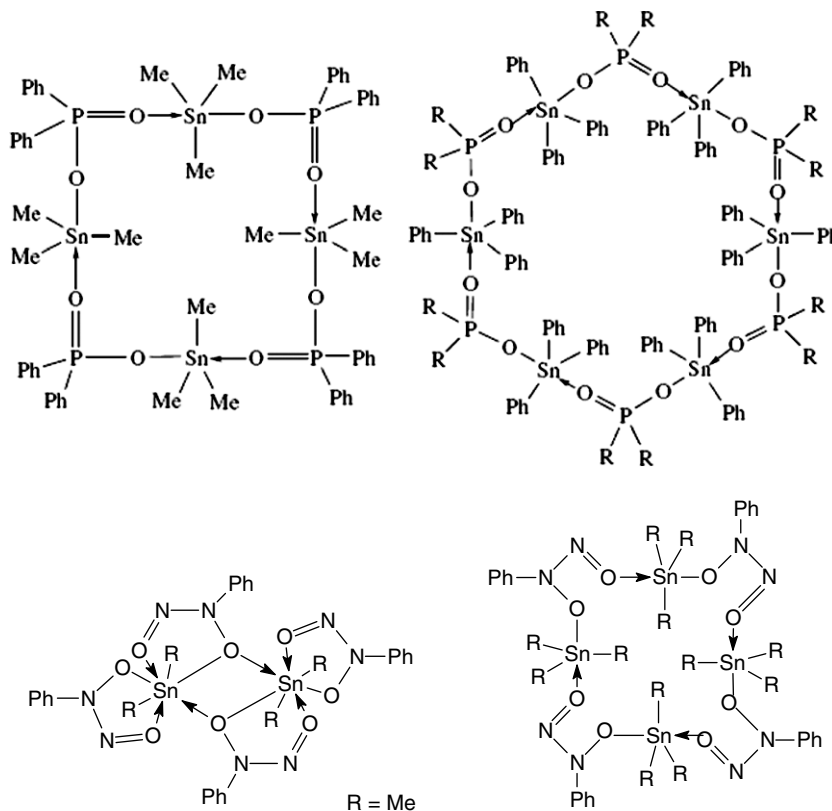
In lead compounds hydrogen bonding can be due to coordinated ligands, like methanol in the  $[Ph_2PbCl_3(MeOH)]^-$  anion<sup>38</sup> (Scheme 1).



**Scheme 1.** Chemical diagram of  $[Ph_2PbCl_3.MeOH]^-$  dimeric supermolecule.



**Scheme 2.** Chemical diagram of  $Me_2SiO_2Sn_2R_4.R_2Sn(OH)_2$ .



**Scheme 3.** Cyclic organotin supermolecules.

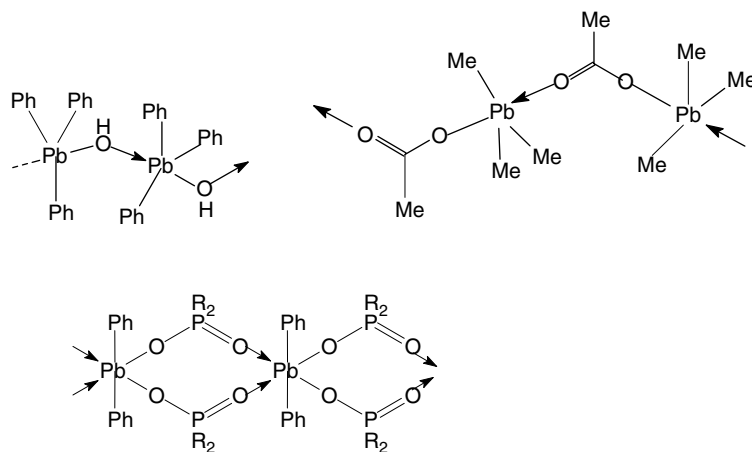
## DATIVE COORDINATE BOND SELF-ASSEMBLY

Organotin chemistry is a rich source of supramolecular structures. Compounds in which tin is connected to a hard donor atom (fluorine, oxygen), such as organotin fluorides, distannoxanes, hydroxides, alkoxides, carboxylates, phosphinates and other inorganic acid salts, all tend to self-assemble into supramolecular architectures through dative coordinate  $F \rightarrow Sn$  or  $O \rightarrow Sn$  bonds.<sup>39–41</sup> This process is sterically controlled and is prevented by bulky substituents at tin and compounds containing such large groups are monomeric. Similarly, organolead hydroxides, carboxylates and phosphinates also form supramolecular assemblies through  $O \rightarrow Pb$  dative coordinate bonds.<sup>42</sup> From the large number of such compounds we mention here only the triorganotin fluorides,  $[R_3SnF]_x$  ( $R = Me, n-Bu, Cy, Ph$ ),<sup>43–45</sup> the tricyclic oxide–hydroxide  $R_2SiO_3Sn_2R_4R_2Sn(OH)_2$  ( $R = tert-Bu$ )<sup>46</sup> (Scheme 2), polymeric di- and triorganotin carboxylates,  $[R_3SnOCOR']_x$ ,<sup>47,48</sup> tetrameric  $[Me_3SnO_2PPh_2]_4$ ,<sup>49</sup> hexameric  $[Ph_3SnO_2PR'_2]_6$ ,<sup>50,51</sup> and also the cupferronato dimer  $[Me_2Sn(ONNOPh)_2]_2$ <sup>52,53</sup> and tetramer  $[Me_3SnONNOPh]_4$ <sup>54</sup> (Scheme 3).

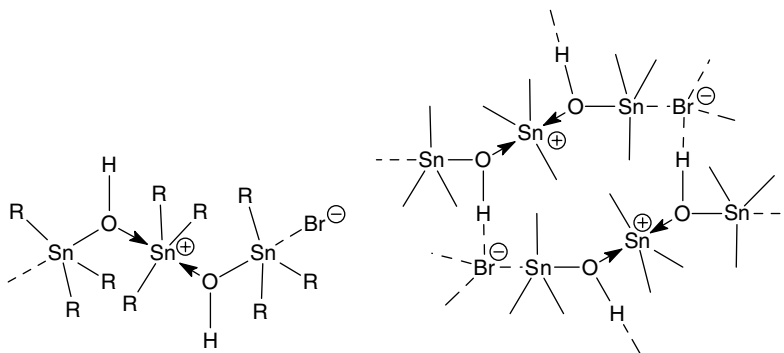
Among lead compounds, chain-like triphenyllead hydroxide,  $[Ph_3PbOH]_x$ ,<sup>55</sup> trimethyllead acetate,  $[Me_3PbOCOME]_x$ ,<sup>56</sup> tetrameric trimethyllead diphenylphosphinate,  $[Me_3PbO_2PPh_2]_4$ ,<sup>57</sup> double-chain phosphinates  $[Ph_2Pb(O_2PR_2)_2]_x$  ( $R = Me, Ph$ ),<sup>58</sup> can be mentioned (Scheme 4).

## SECONDARY BOND (SOFT–SOFT) INTERACTIONS

Tin (to some extent) and lead are soft metals and their compounds containing soft donor atoms (heavier halogens, sulfur, selenium binding atoms) can engage in secondary bonding or soft-soft interactions, leading to supramolecular self-assembly. The secondary bonds were described by N.W. Alcock<sup>59</sup> as 'interactions characterized by interatomic distances longer than single covalent bonds but shorter than van der Waals interatomic distances'. In energy terms this corresponds to weaker than covalent or dative bonds but stronger than van der Waals interactions. They are strong enough to influence the coordination geometry of the central metal atoms and to hold together associated molecules, either as distinct supermolecules



**Scheme 4.** Organolead supramolecular chain-like arrays.



**Scheme 5.** Charge assisted self-assembly.

(dimers, trimers, tetramers) or as supramolecular polymeric arrays. The secondary bonds are explained as  $X-A \cdots Y$  asymmetric three-center systems, with three  $s$  symmetry atomic orbitals on A, X and Y, combined to form three molecular orbitals: one filled bonding MO located between A and X, one filled non-bonding or weakly bonding MO located between A and Y and one empty antibonding orbital.<sup>60,61</sup>

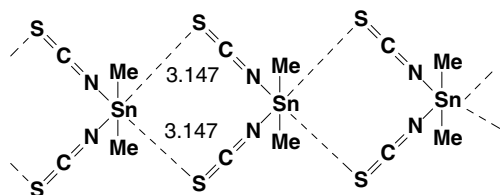
In organotin chemistry, the halides (other than fluorides, mostly chlorides),<sup>62–68</sup> cyclic dioxastannolanes,<sup>69</sup> oxathiastannolanes,<sup>70,71</sup> dithiastannolanes,<sup>72,73</sup> and other cyclic thio derivatives,<sup>74</sup> are often associated into supramolecular structures via  $\text{Sn} \cdots \text{S}$  secondary bonds.

An interesting supramolecular architecture is observed in the compound  $[(\text{Me}_3\text{Sn})_3(\mu - \text{OH})_2]^+ \text{Br}^-$ , containing dative coordinate  $\text{HO} \rightarrow \text{Sn}$  bonds and charge-assisted  $\text{OH} \cdots \text{Br}^-$  hydrogen bonds and  $\text{Sn} \cdots \text{Br}^-$  secondary bonds<sup>75</sup> (Scheme 5).

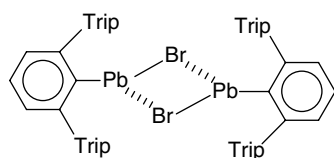
Secondary bond interactions have been often ignored in earlier crystal structure determinations. The mining of the Cambridge Crystallographic Database allows sometimes the identification of new secondary bond interactions. Thus, dimethyltin dithiocyanate  $\text{Me}_2\text{Sn}(\text{NCS})_2$  molecules,<sup>76</sup> are self-organized into chain-like supramolecular arrays through  $\text{Sn} \cdots \text{S}$  secondary bonds ( $\text{Sn} \cdots \text{S}$  3.147 Å, van der Waals distance 3.97 Å) (Scheme 6).

Dimethyltin dimethyldithiophosphate (BABPID) was described as a monomeric compound,<sup>77</sup> but a structure analysis with the aid of MERCURY program reveals supramolecular self-assembly through secondary  $\text{S} \cdots \text{S}$  interactions ( $\text{S} \cdots \text{S}$  3.594 Å) (Scheme 7).

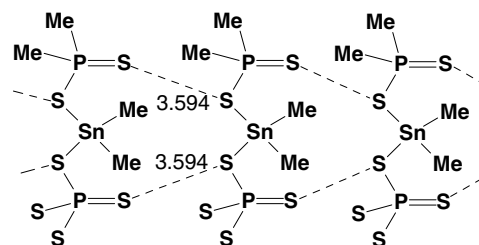
A soft metal lead is particularly able to form secondary bonds (soft–soft interactions), either with halogens or sulfur, leading to supramolecular self-assembly. Thus, organolead(II) monohalides with bulky substituents form supermolecules, like the dimers  $[\text{BrPbC}_6\text{H}_4(\text{trip})_2 \cdot 2, 6]_2$ ,<sup>78</sup>



**Scheme 6.** Supramolecular self-assembly of  $\text{Me}_2\text{Sn}(\text{NCS})_2$ .



**Scheme 8.** Cyclic organolead supermolecules.

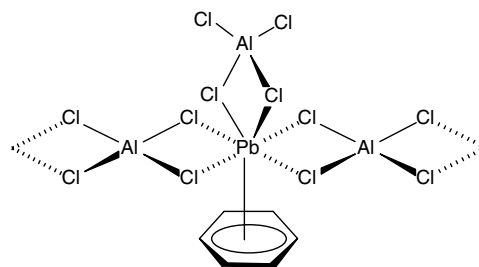


**Scheme 7.** Supramolecular self-assembly of  $\text{Me}_2\text{Sn}(\text{S}_2\text{PMe}_2)_2$ .

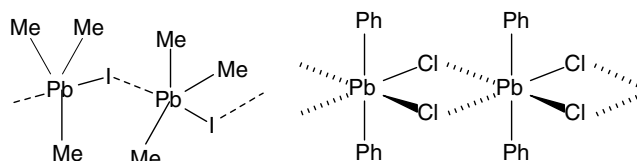
and  $[\text{ClPbC}(\text{SiMe}_3)_2\text{CPhNSiMe}_3]_2$  (with alternating short  $\text{Pb} \cdots \text{Cl}$  2.609 Å and long  $\text{Pb} \cdots \text{Cl}$  3.276 Å, van der Waals distance 3.77 Å),<sup>79</sup> whereas  $\text{ClPbC}(\text{SiMe}_3)_3$  forms cyclic trimers with  $\text{Pb} \cdots \text{Cl}$  secondary bonds ( $\text{Pb} \cdots \text{Cl}$  2.71–2.76 Å) based upon six-membered  $\text{Pb}_3\text{Cl}_3$  rings<sup>80</sup> (Scheme 8).

A pi-arene complex  $\eta^6 - \text{C}_6\text{H}_6\text{Pb}(\text{AlCl}_4)_2$  forms chain-like supramolecular arrays due to charge-assisted  $\text{Pb} \cdots \text{Cl}$  secondary bonds and contains both chelating and bridging  $\text{AlCl}_4$  units<sup>81</sup> (Scheme 9).

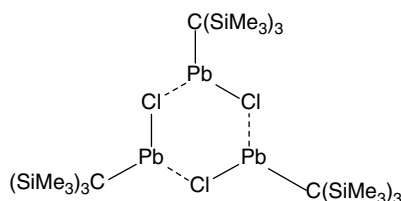
Organolead(IV) halides also self-assemble into supramolecular arrays. Thus, trimethyllead chloride,  $\text{Me}_3\text{PbCl}$ , forms

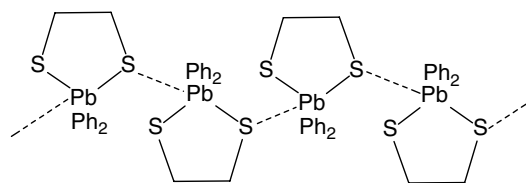


**Scheme 9.** Supramolecular self-assembly in  $\text{C}_6\text{H}_6\text{Pb}[\text{AlCl}_4]_2$ .

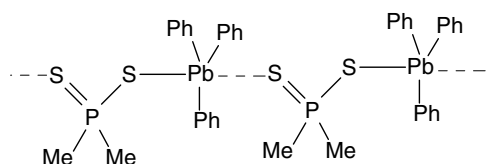


**Scheme 10.** Supramolecular self-assembly of organolead halides.





**Scheme 11.** Supramolecular self-assembly of dithia-plumbolanes.



**Scheme 12.** Supramolecular self-assembly of  $\text{Ph}_3\text{Pb}(\text{S}_2\text{PMe}_2)$ .

zig-zag chains of trigonal bipyramids with alternating axial covalent (short  $\text{Pb}-\text{Cl}$  2.764 Å) and secondary (long  $\text{Pb}\cdots\text{Cl}$  2.814 Å, van der Waals distance 3.77 Å) bonds.<sup>82</sup> Similarly,  $\text{Ph}_3\text{PbCl}$  ( $\text{Pb}-\text{Cl}$  2.706,  $\text{Pb}\cdots\text{Cl}$  2.947 Å),  $\text{Ph}_3\text{PbBr}$  ( $\text{Pb}\cdots\text{Br}$  2.852 Å,  $\text{Pb}\cdots\text{Br}$  3.106 Å, van der Waals distance 3.87 Å),<sup>83</sup> benzyldiphenyllead bromide,  $\text{BzPh}_2\text{PbBr}$  ( $\text{Pb}-\text{Br}$  2.885 and  $\text{Pb}\cdots\text{Br}$  2.985 Å),<sup>84</sup> and  $\text{Me}_3\text{PbI}$  ( $\text{Pb}-\text{I}$  3.038,  $\text{Pb}\cdots\text{I}$  3.360 Å, van der Waals distance 4.00 Å)<sup>85</sup> are self-assembled into supramolecular single-strand chains, whereas diphenyllead dichloride,  $\text{Ph}_2\text{PbCl}_2$ , forms double bridged chain-like arrays made of octahedral units with the phenyl groups in axial positions (Scheme 10).

Lead-sulfur compounds show a marked tendency to self-assemble through  $\text{Pb}\cdots\text{S}$  secondary bonds. A typical example is diphenyllead dithiaplumbolane,  $\text{Ph}_2\text{Pb}(\text{SCH}_2)_2$ , which forms chain-like supramolecular arrays through weak  $\text{Pb}\cdots\text{S}$  secondary bonds ( $\text{Pb}\cdots\text{S}$  3.55 Å, van der Waals distance 3.82 Å)<sup>86</sup> (Scheme 11).

Quite a number of lead(II) dithiophosphates,  $\text{Pb}[\text{S}_2\text{P}(\text{OR})_2]_2$ , are associated in solid state through  $\text{Pb}\cdots\text{S}$  secondary bonds. Dimeric dithiophosphate supermolecules ( $\text{R} = \text{Bu}^i$ ,  $\text{Ph}^{87}$ ) or chain-like supramolecular arrays

[ $\text{R} = \text{Me}^{88}$ ,  $\text{Et}$  (Lönnecke P. *private communication* 2006),  $\text{iso-Pr}^{89}$ ,  $n\text{-Pr}$ ,  $\text{Cy}^{90}$ ), have been reported. The compound  $\text{Pb}[\text{S}_2\text{P}(\text{OEt})_2]_2$  was initially described as monomeric,<sup>91</sup> but it was found that the structure contains  $\text{Pb}\cdots\text{S}$  (3.469 and 3.469 Å, van der Waals distance 3.82 Å) and  $\text{Pb}\cdots\text{O}$  (2.998 and 3.041 Å, van der Waals distance 3.54 Å) intermolecular secondary bonds.

Triorganolead dithiophosphates, e.g. triphenyllead dimethyldithiophosphate,  $[\text{Ph}_3\text{PbS}_2\text{PMe}_2]_x$ , forms supramolecular chains ( $\text{Pb}-\text{S}$  2.708,  $\text{Pb}\cdots\text{S}$  3.028 Å),<sup>92</sup> whereas lead(II) dithiophosphates,  $[\text{Pb}(\text{S}_2\text{PR}_2)_2]_x$  ( $\text{R} = \text{Me}$ ,  $\text{Pb}\cdots\text{S}$  3.298 Å;  $\text{Et}$ ,  $\text{Pb}\cdots\text{S}$  3.100 Å;  $\text{Ph}$ ,  $\text{Pb}\cdots\text{S}$  3.270 and 3.448 Å)<sup>93–95</sup> form intricate chain-like supramolecular arrays (Scheme 12).

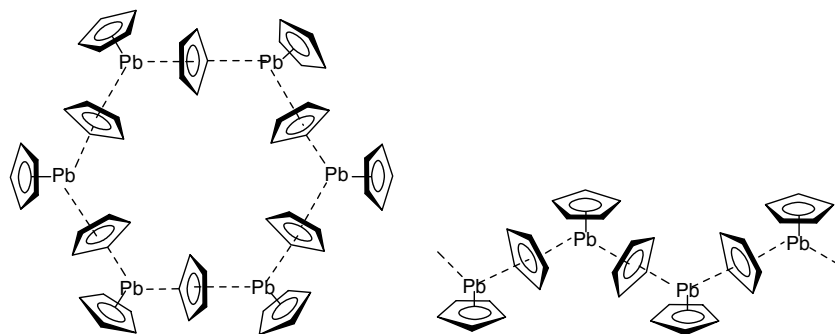
## PI-BOND SELF-ASSEMBLY

Supramolecular self-assembly through pi-bonds is observed in lead(II) cyclopentadienyl compounds. Thus, plumbocene, forms cyclic hexamer supermolecules, and supramolecular zig-zag and helical chains, through lead- $\text{C}_5\text{H}_5$  pi-bonds<sup>96,97</sup> (Scheme 13).

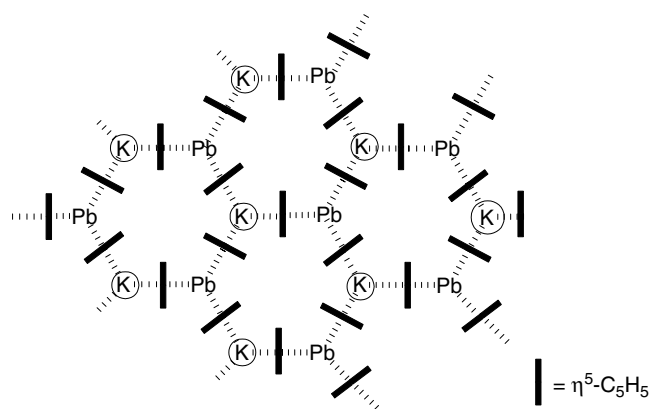
Shorter fragments, like  $[\text{Pb}_2(\eta^5-\text{C}_5\text{H}_5)_5]^-$  and  $[\text{Pb}_4(\eta^5-\text{C}_5\text{H}_5)_9]^-$  have also been discovered.<sup>98</sup> Charge-assisted pi-bonding interactions between  $\text{K}^+$  cations and  $[\text{Pb}(\eta^5-\text{C}_5\text{H}_5)_3]^-$  anions lead to the formation of a unique supramolecular bidimensional structure<sup>99</sup> (Scheme 14).

## CONCLUSIONS

This survey of selected tin and lead compounds (mainly organometallic) suggests that supramolecular self-assembly can be a frequent occurrence and should be not ignored. The current X-ray diffraction facilities afford now rather easy analysis of crystal packing and this is the way to identify supramolecular self-assembly. It can be expected in compounds containing groups able to form hydrogen bonds, or in compounds containing both donor and acceptor sites. It can be expected that this interdisciplinary area of modern chemistry will provide further interesting examples of



**Scheme 13.** Supramolecular self-assembly of plumbocene.



**Scheme 14.** Supramolecular self-assembly of  $K[Pb(C_5H_5)_3]$ .

intermolecular interactions and novel unexpected structures. The better understanding of supramolecular interactions can provide routes to important and useful new materials.

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