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[4-*t*Bu-2,6-{P(O)(O*i*Pr)$_2$}$_2$C$_6$H$_2$SnL]$^+$: An NHC-Stabilized Organotin(II) Cation and Related Derivatives

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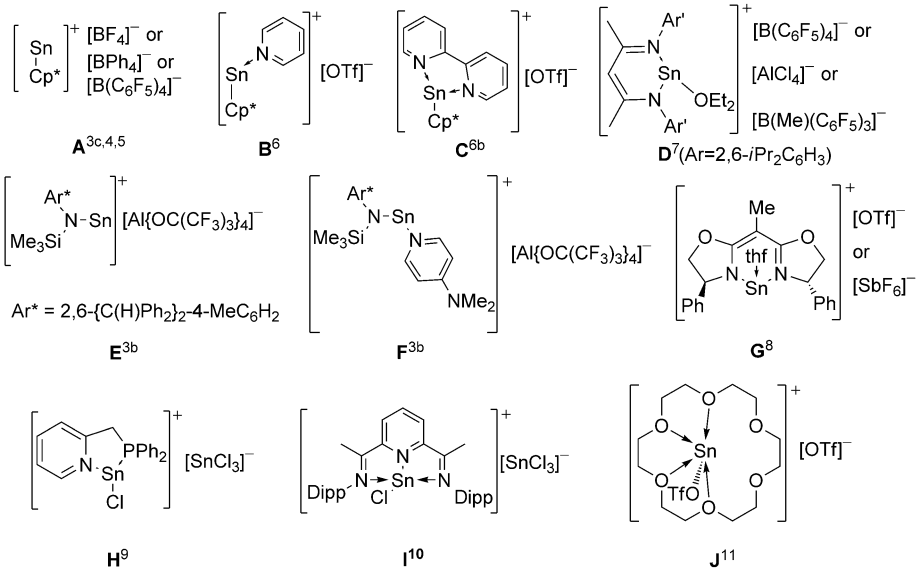
Dedicated to the Bayer company on the occasion of its 150th anniversary

The chemistry of stable, heavier carbene homologues is a topic of continuing interest,[1] especially because low-valent tin cations are targets in inorganic and organometallic chemistry.[2] Their potential as highly Lewis acidic catalysts has been explored.[3] Selected tin(II) monocations and their Lewis base adducts are depicted in Scheme 1. Transition-metal-substituted derivatives of such cations are known as well.[12] N-heterocyclic carbenes (NHCs) are known for their extraordinary capacity to stabilize exotic species such as dichlorosilylene, $SiCl_2$,[13] compounds with terminal Si=O bonds[14] and even molecular Si_2[15] and Sn_2.[16] The addition of nucleophilic carbenes to stannylenes provides the tin adducts.[17] In particular, complexes of the type (NHC)$SnCl_2$ were screened as catalysts for polyurethane formation.[18] Preliminary efforts to isolate NHC-stabilized Sn^{II} cations have also been reported.[19] Herein, we present attempts to obtain salt-like compounds containing organotin(II) cations stabilized by combined intra- and intermolecular donor–acceptor interactions. These investigations are undertaken as part of our ongoing studies of the synthesis and reactivity of intramolecularly coordinated heteroleptic organostannylenes[20] of the type RSnX (hereafter R = 4-*t*Bu-2,6-{P(O)(O*i*Pr)$_2$}$_2$C$_6$H$_2$, X = electronegative substituent).



A[3c,4,5] B[6] C[6b] D[7](Ar=2,6-*i*Pr$_2$C$_6$H$_3$) E[3b] F[3b] G[8] H[9] I[10] J[11]

Scheme 1. Tin(II) monocations and their Lewis base adducts.

The NMR spectra of a mixture of sodium tetraphenylborate ($NaBPh_4$) and organostannylene (RSnCl) in CH_2Cl_2 showed decomposition of half of the tetraphenylborate to triphenylboron (see the Supporting Information). No well-defined tin-containing products were isolated. The reaction of RSnCl with sodium tetrakis(3,5-bis-trifluoromethyl)phenylborate (NaB[3,5-(CF$_3$)$_2$C$_6$H$_3$]$_4$) and 1,3-bis(2,6-diisopropylphenyl)imidazol-2-ylidene (IPr), or with $NaBPh_4$ and *para*-dimethylaminopyridine (DMAP) in CH_2Cl_2 furnished the Lewis base adducts **1** and **2**, respectively, as crystalline materials in good yields (Scheme 2). Compounds **1** and **2** show good solubility in CH_2Cl_2, but are sparingly soluble in diethyl ether and almost insoluble in benzene, toluene, or hexanes. They are sensitive to moisture (see the Supporting Information).

Compound **1** is the first example containing a carbene-stabilized tin(II) cation, whereas compound **2** is comparable to the salt **F** (Scheme 1) reported by Jones and co-workers.[3b] Notably, carbene-stabilized germanium cations of the types $[(NHC)_3Ge]^{2+}$ and $[(NHC)_2GeCl]^+$ (NHC = :C(*i*PrNCMe)$_2$) have been reported.[21] The molecular structures of com-

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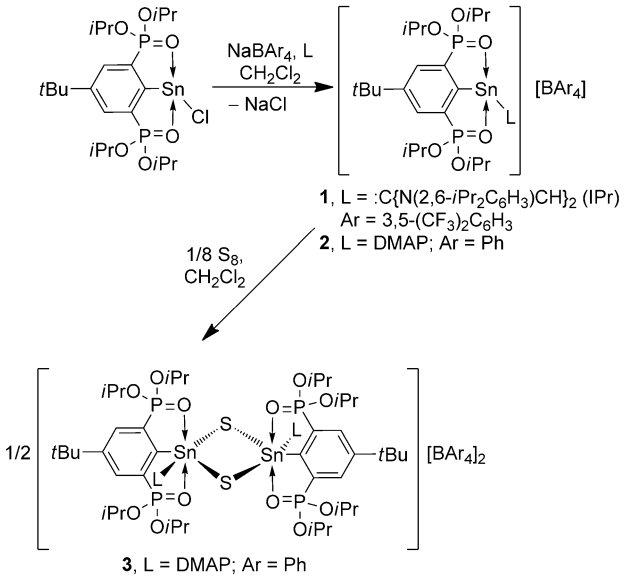


Scheme 2. Syntheses of the organotin(II) salts **1** and **2**, which contain Lewis base stabilized cations, and oxidation with sulfur to give compound **3**.

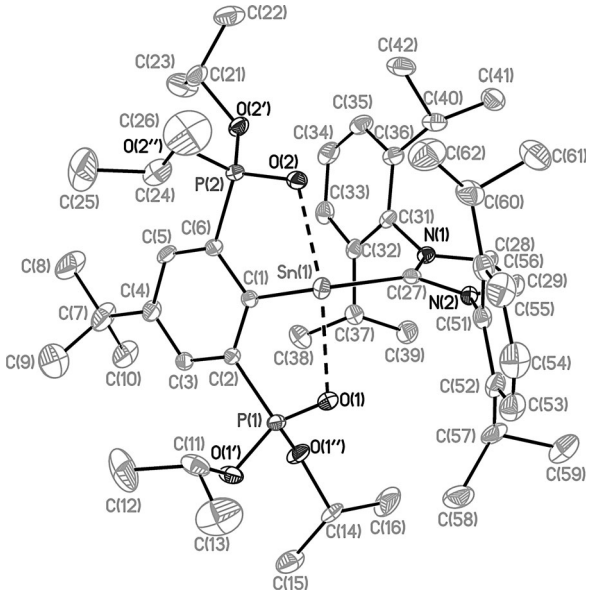


Figure 1. Molecular structure of compound **1**·0.5CH_2Cl_2 with thermal ellipsoids showing 30% probability. The tetrakis(3,5-trifluoromethyl)phenylborate anion, the CH_2Cl_2 solate molecule and the hydrogen atoms are omitted for clarity.

pounds **1**, as its dichloromethane solvate **1**·0.5CH_2Cl_2, and **2** are shown in Figures 1 and 2, respectively, and selected bond lengths and angles are given in Table 1. The tin atoms in compounds **1** and **2** each exhibit a distorted pseudo-trigonal-bipyramidal environment with O(1) and O(2) occupying the axial positions. The equatorial positions are occupied by C(1), the lone pair and the basic site of the stabilizing ligand (C(27) in **1** and N(1) in **2**). The axial O(1)-Sn(1)-O(2) angles of 149.3(1)° (**1**) and 150.3(1)° (**2**), differ from 180° as a

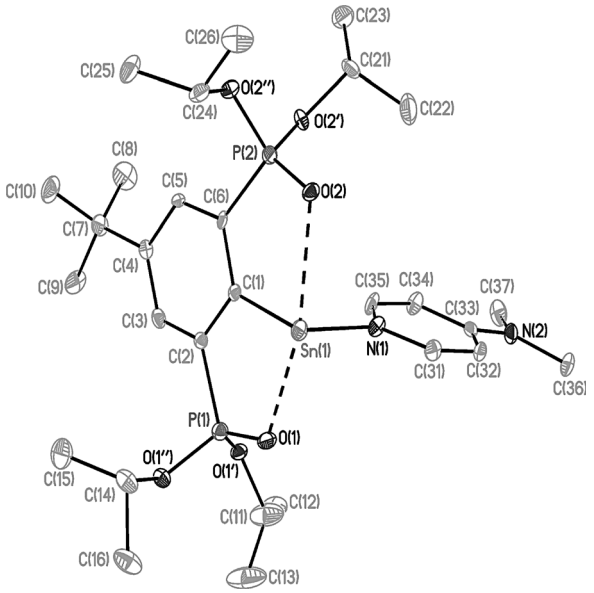


Figure 2. Molecular structure of compound **2** with thermal ellipsoids showing 30% probability and the hydrogen atoms and the tetraphenylborate anion omitted for clarity.

Table 1. Selected bond lengths [Å] and angles [°] in the compounds **1**–**3**.

	1·0.5 CH_2Cl_2 X = C(27)	**2** X = N(1)	**3**·3CH_3CN X = N(1)
Sn(1)–C(1)	2.202(4)	2.234(2)	2.143(2)
Sn(1)–O(1)	2.423(3)	2.407(2)	2.250(1)
Sn(1)–O(2)	2.360(2)	2.482(2)	2.227(1)
Sn(1)–X(1)	2.287(4)	2.243(2)	2.297(2)
Sn(1)–S(1)			2.3472(5)
Sn(1)–S(1A)			2.5515(5)
O(1)-Sn(1)-O(2)	149.3(1)	150.33(5)	158.36(5)
C(1)-Sn(1)-X(1)	101.2(1)	91.73(8)	91.19(6)
Sn(1)-S(1)-Sn(1A)			90.87(2)
N(1)-Sn(1)-S(1)			90.66(4)

result of geometric constraints imposed by the structure of the ligand. They are smaller than the analogous angles in the parent organotin(II) chloride RSnCl (152.01(6)°)[20c] and the corresponding triorganotin(IV) cation $[RSnPh_2]^+$ (159.01(9)°).[22] The C(1)-Sn-C(27) angle of 101.2(1)° in **1** clearly shows the Sn–$C_{Carbene}$ interaction is not of the stannene-type,[23] which is consistent with the Lewis basic nature of the IPr ligand. The angle is, however, as result of steric hindrance, larger than the C(1)-Sn-N(1) angle of 91.73(8)° in **2**, which, in turn, is similar to the C(1)-Sn-Cl(1) angle in RSnCl (94.23(6)°).[20c] The geometries observed imply high s character of the lone pairs at the Sn atoms in **1** and **2**, as substantiated by NBO analysis (see discussion below). The Sn(1)–C(27) distance (2.287(4) Å) is similar to that observed in (IPr)Sn=Sn(IPr) (2.280(3) Å).[16] The N(1)-C(27)-N(2) angle of 104.4(3)° is slightly larger than in the free carbene (101.4°).[24] The Sn–O distances vary between 2.360(2) (Sn(1)–O(2) in **1**) and 2.482(2) Å (Sn(1)–O(2) in **2**), which are comparable to the corresponding distances found for RSnCl (2.430(2) and 2.427(2) Å),[20c] but longer than in

$[RSnPh_2]^+$ (2.241(3) and 2.249(2) Å).[22] The Sn(1)–N(1) distance of 2.234(2) Å in compound **2** is shorter than the Sn–N distance reported for compound **F** (2.286(6) Å; Scheme 1). One phenyl ring (C61–C66) of the tetraphenylborate anion shows a π-type interaction with the Sn(1) cation at a distance of 3.6955(2) Å. This interaction, although much weaker, resembles the situation reported for the Sn···Ph interaction at a distance of 3.110(2) Å reported for Cp^*SnBPh_4 ($Cp^* = C_5Me_5$).[5]

The ^{119}Sn NMR spectra in CD_2Cl_2 of compounds **1** and **2** displayed sharp triplet resonances at $\delta = -169$ and -170 ppm, respectively, showing that the tin atoms in both compounds are in similar electronic environments, as confirmed by natural charge calculations (+1.11 for **1** and +1.21 for **2**). The ^{31}P NMR spectra showed resonances shifted to lower frequency with larger $J(^{31}P–^{117/119}Sn)$ coupling constants [**1**, $\delta = 30.0$ ppm (s, $J(^{31}P–^{117/119}Sn) = 137/141$ Hz); **2**, $\delta = 35.6$ ppm (s, $J(^{31}P–^{117/119}Sn) = 129/135$ Hz] than those observed for the organostannylene RSnCl ($\delta = 37.8$ ppm, $J(^{31}P–^{117/119}Sn) = 113/118$ Hz)[20c]. The ^1H and the ^{13}C NMR spectrum of compound **1** showed two sets of signals for the isopropyl groups, whereas the spectra of compound **2** showed only one set of signals. A ^1H NMR spectrum of compound **2** at −84 °C failed to resolve the isopropyl group. This is attributed to a fast Sn–N dissociation–association process in compound **2**, whereas no Sn–C dissociation is observed for carbene complex **1** as evidenced by the ^{13}C NMR spectrum of **1**. This spectrum showed $J(^{13}C–^{117/119}Sn)$ couplings for the imidazole *C*H carbon ($\delta = 126.6$ ppm, $^3J(^{13}C–^{117/119}Sn) = 23.0$ Hz) and a triplet resonance for the carbene carbon atom at $\delta = 183.2$ ppm ($J(^{13}C–^{31}P) = 1.5$ Hz). The chemical shift of the latter is comparable to the value reported for $(IPr)SnCl_2$ ($\delta = 184.2$ ppm).[25] The ^{15}N NMR spectrum of compound **2** showed two resonances at $\delta = -166$ (HC*N*CH) and -307 ppm (NMe_2), which are shifted in comparison to free DMAP ($\delta = -107$ and -327 ppm).

The reaction of compound **2** with elemental sulfur provided the novel four-membered-ring species $[R(L)SnS]_2[BPh_4]_2$ (**3**: L = DMAP) containing a dinuclear dication. Its molecular structure, determined from single crystals of its acetonitrile solvate **3**·3CH_3CN, is shown in Figure 3. In compound **3** the tin atom adopts a distorted octahedral geometry. The structure of **3** strongly resembles that of the chlorido-substituted analogue $[R'(Cl)SnS]_2$ (R′ = 4-*t*Bu-2,6-{P(O)(OEt)$_2$}$_2$C$_6$H$_2$).[20b] The Sn(1)–S(1)/S(1A) bond lengths are 2.3472(5)/2.5515(5) Å. The Sn(1)–N(1) distance of 2.297(2) Å is elongated in comparison to compound **2**, whereas the Sn–O distances are shortened to 2.250(1) and 2.227(1) Å.

The ^{31}P and ^{119}Sn NMR spectra showed a singlet at $\delta = 22.9$ ppm ($J(^{31}P–^{117/119}Sn) = 105/110$ Hz) and a triplet resonance at $\delta = -423$ ppm ($J(^{119}Sn–^{31}P) = 108$ Hz, $^2J(^{119}Sn–^{117}Sn) = 341$ Hz), respectively, that resemble the data reported for $[R'(Cl)SnS]_2$ [^{119}Sn NMR: $\delta = -439$ ppm (t, $J(^{119}Sn–^{31}P) = 84$ Hz); ^{31}P NMR: $\delta = 26.7$ ppm ($J(^{31}P–^{119/117}Sn) = 86/82$ Hz)].[20b] A $^{119}Sn–^{117}Sn$ coupling larger than that observed for $[Sn(Ar^{N2})_2(\mu\text{-}S)]_2$ (277 Hz, $Ar^{N2} = 2,6\text{-}(Me_2N)_2C_6H_3$)[26] or $[L'(Ph)Sn(\mu\text{-}S)]_2$ (226 Hz, L′ = 2,6-(*t*BuOCH$_2$)$_2$C$_6$H$_3$)[27] shows that the four-membered ring is retained in solution. The ^1H NMR spectrum of compound **3** showed two broad unresolved signals ($\nu_{1/2}$ ca. 250 Hz) for the C*H*(CH$_3$)$_2$ protons and a broad unresolved signal for the CH(C*H*$_3$)$_2$ protons. Lowering the temperature to −84 °C resulted in sharpening of the two C*H* signals and resolution into four doublets for the CH(C*H*$_3$)$_2$ protons. This indicates that the Sn–N dissociation has become slow on the ^1H NMR time scale. The ^{15}N NMR spectrum showed two higher frequency resonances, with respect to **2**, at $\delta = -152$ and -309 ppm. An electrospray ionization mass spectrum of compound **3** (positive ion mode) in CH_3CN showed a mass cluster centered at $m/z = 612.2$ corresponding to a dicationic structure resulting from loss of both dmap ligands.

The reaction of the carbene-stabilized compound **1** with sulfur appeared to be more complex than that of **2** and no reaction product could be identified unambiguously.

The calculation of the electronic structures of compounds **1** and **2** revealed the natural bond orbitals (NBOs) depicted in Figure 4. In both **1** and **2** the Sn(1)–C(1) bond is the only covalent bond, whereas all other interactions involving the

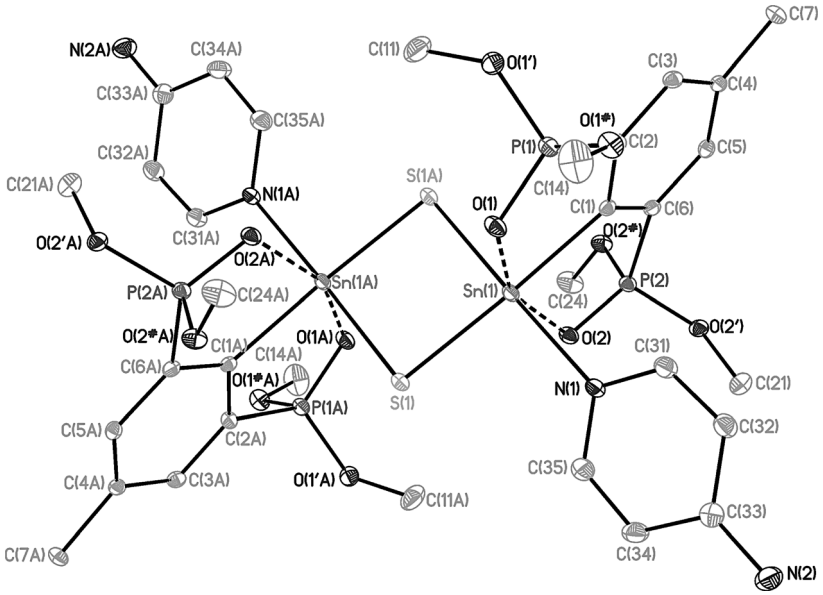


Figure 3. Molecular structure of compound **3** with thermal ellipsoids showing 30% probability and the hydrogen atoms, the methyl groups, the acetontrile solvate molecule and the tetraphenylborate anions omitted for clarity.

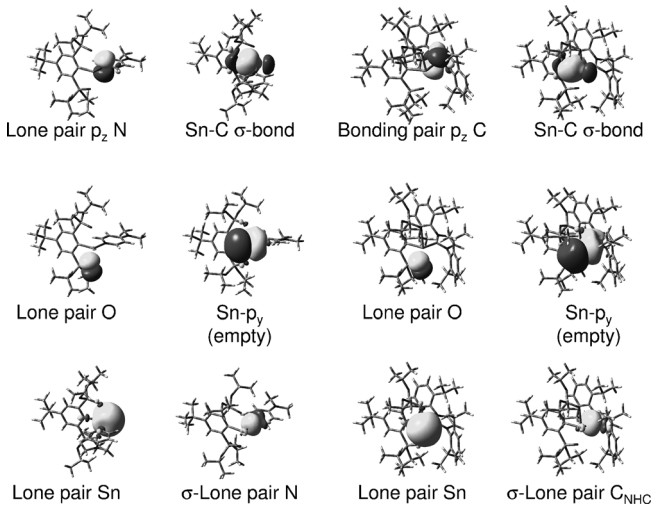


Figure 4. Natural bond orbitals for complexes **1** (right) and **2** (left).

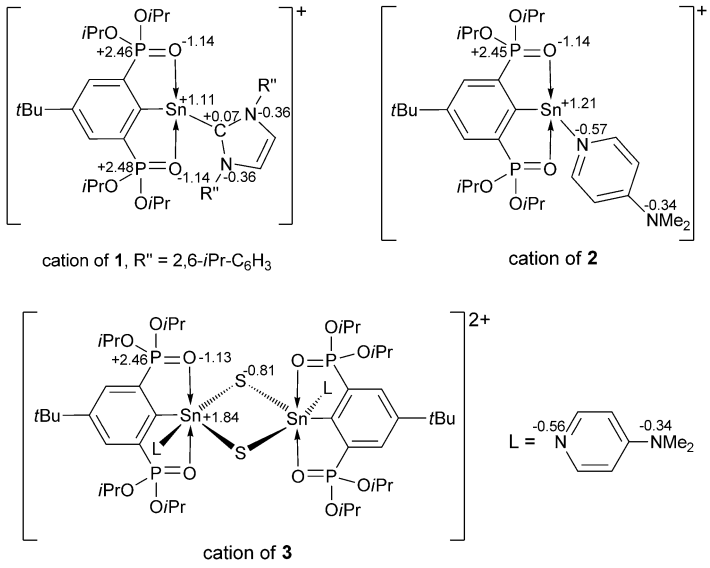
tin atom were calculated to be of donor–acceptor character within the NBO framework and criteria set by the program. From second-order perturbation analysis, the interaction in **1** between the carbene carbon atom (C(27)) and the tin atom is calculated to be 137 kcal mol^{-1}, whereas the interaction in **2** between the DMAP nitrogen donor atom (N(1)) and the tin atom is calculated to be 78 kcal mol^{-1}. The stronger donor character of the NHC ligand in **1** as compared to DMAP in **2** is also evident from the natural population analysis (NPA) charges of +1.11 (**1**) and +1.21 (**2**; Scheme 3).

The lone pairs at the Sn^{II} atoms in **1** and **2** occupy mainly 4s type orbitals with about 14 and 17% p character, respectively. They are slightly polarized away from the pincer-type ligand indicating their Lewis basic character. However, an empty p_y orbital at the Sn atoms, polarized away from the carbene and DMAP ligands, is accessible for coordination to the Sn atom and thus represents a Lewis acidic character that depends on the polarization of the tin atom by the Lewis base ligand. Overall, the positive charge in the cations $[RSnL]^+$ is unsymmetrically distributed (**1**: RSn +0.7, NHC +0.3; **2**: RSn +0.8, DMAP +0.2).

The packing of complexes **1** and **2** suggest that nonbonding tin–arene interactions contribute to their molecular structures. However, a full geometry optimization of the complexes revealed only slight geometric changes indicating the structural influence of these nonbonding interactions on the geometry is negligible.

As expected, in oxidized dicationic compound **3**, the tin atom carries most of the positive charge (+1.8, Scheme 3). Both Sn–S bonds and the Sn–C bond are highly covalent and contain 1.9, 1.85, and 1.81 e$^-$, respectively. However, they are different with respect to the contributions made by the orbitals of the tin and sulfur atoms (short Sn–S bond: Sn: 40% s/60% p, S: 16% s/82% p; long Sn–S bond: Sn: 20% s/72% p/5% d, S: 17% s/82% p; Sn–C bond: Sn: 34% s, 56% p, 5% d). Remarkably, for the shorter Sn–S bond there is an additional covalent bond (1.6 e$^-$) of π type that is 99% localized at the sulfur and only 1% at the tin atom. In fact, this representation supports the idea of the dication in **3** resulting from the dimerization of two $[R(dmap)Sn{=}S]^+$ moieties.

In summary, two compounds containing Lewis base stabilized organotin(II) cations have been prepared by simple one-pot reactions. Among these, **1** is the first of its type containing an NHC as a Lewis base. Although NHCs appear to be superior in stabilizing such organotin(II) cations, the "cheaper" DMAP-containing compound **2** is sufficiently stable to allow subsequent reactions, as shown by the formation of **3** with the retention of the positive charge, which in turn is attractive for applications in catalysis.



Scheme 3. Schematic drawings of the cations in **1**–**3** including the NPA charges at selected atoms.

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Keywords: carbene homologues · carbenes · cations · density functional calculations · tin

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