the highest TON with aryl chlorides at room temperature reported so far. Various *para* and *meta* substituted substrates can be quantitatively converted within a few hours.

Experimental Section

3b: **1b** (1000 mg, 1.81 mmol) was dissolved in *n*-hexane (30 mL). A solution of **2** (1400 mg, 4.16 mmol) in *n*-hexane (30 mL) was added. The mixture was stirred at ambient temperature for 24–48 h during which a yellow solid precipitated. After filtration and drying in vacuo the product was obtained as an analytically pure bright yellow solid. X-ray suitable crystals were obtained by recrystallization from diethyl ether. Yield: 1177 mg, 1.51 mmol, 83 %; m.p. >285 °C (decomp.); ^{1}H NMR (400 MHz, [D$_8$]toluene, 25 °C): $\delta = 1.47-1.80$ (m, 24 H, C*H*$_2$ of $C_{10}H_{15}$), 2.06–2.12 (m, 12 H, C*H* of $C_{10}H_{15}$), 3.05 (s, 24 H, C*H*$_2$ of $C_{10}H_{15}$), 6.69 (s, 4 H, NC*H*C*H*N); ^{13}C{^{1}H} NMR (100.5 MHz, [D$_8$]toluene, 25 °C): $\delta = 31.0$, 36.8, 44.0 ($C_{10}H_{15}$), 57.2 (ipso-*C* of $C_{10}H_{15}$), 112.7 (N*C*H*C*HN), 191.8 (N*C*N); CI-MS: *m*/*z* (%): 778 (3) [*M*$^+$], 336 (100) [NHC$^+$], 281 (33), 207 (27), 203 (40); elemental analysis calcd (%) for $C_{46}H_{64}N_4Pd$ (779.44): C 70.88, H 8.28, N 7.19; found: C 70.80, H 8.24, N 7.22.

Alternative synthesis: **2** (343 mg, 1.02 mmol), [(η^3-C_3H_5)PdCl]$_2$ (100 mg, 0.254 mmol), and sodium dimethylmalonate (78 mg, 0.506 mmol) were suspended in toluene (30 mL) in a Schlenk tube. The stirred mixture was heated at 90 °C for 16 h after which the solution was brown. After cooling to room temperature, the mixture was filtered to remove some free palladium metal and the solution was concentrated. Crystallization at −50 °C yielded the product as bright yellow solid. Yield: 219 mg, 0.28 mmol, 55 %.

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[11] Current prices according to *STREM* catalogue 2001–2003: **1b** (2 g): 752 €; allylpalladium chloride dimer (2 g): 123 €.

[12] X-ray structure for **3b·H$_2$O**: bright yellow crystals, $C_{46}H_{64}N_4OPd$, crystal dimensions 0.38 × 0.32 × 0.24 mm; $M_r = 797.45$; monoclinic, space group *C*2/*c* (no. 15), $a = 23.436(2)$, $b = 13.213(1)$, $c = 12.999(1)$ Å, $\beta = 104.645(9)°$, $V = 3894.5(6)$ Å^3, $Z = 4$, $\lambda(\mathrm{Mo}_{K\alpha}) = 0.71073$ Å, $\mu = 0.518$ mm^{-1}, $\rho_{\mathrm{calcd}} = 1.360$ g cm^{-3}, $T = 199(1)$ K, $F(000) = 1696$. Preliminary examination and data collection were carried out on a Stoe IPDS area detecting diffraction system at the window of a rotating anode (Nonius, FR591). The unit cell parameters were obtained by full-matrix least-squares refinement of 4977 reflections. A total number of 23284 reflections were collected ($\theta_{\max}$: 25.66°, exposure time: 300 s per image, 310 images, $\Delta\phi$: 1.0°, dx: 70.0 mm). After merging ($R_{\mathrm{int}} = 0.0860$) a sum of 3527 (all data) independent reflections remained and were used for all calculations. 2307 of them were observed data with $I > 2\sigma(I)$. The structure was solved by direct methods (SIR-92)[15] and refined by full-matrix least-squares on F^2 (SHELXL-97).[16] All non-hydrogen atoms of the asymmetric unit were refined anisotropically. All hydrogen atoms were located in the difference Fourier map and refined with individual isotropic temperature parameters, except those located at the water oxygen. They were calculated in ideal positions (riding model). $R1 = 0.0451$ (observed data), $wR2 = 0.1185$ (all data), GOF = 0.934, 365 parameters, $\Delta\rho_{\max/\min} = 1.11/-0.97$ e Å^{-3}. No correction for decay and absorption was applied. CCDC-175167 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or deposit@ccdc.cam.ac.uk).

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X-Ray Structural Characterization of a Monoorganotin Acid[**]

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Dedicated to Sir John Meurig Thomas
on the occasion of his 70th birthday

The hydrolysis of monoorganotin trihalides has been a topic of interest in the chemistry of tin for a long time. Methylstannonic acid [Me(Sn(OH)O] was first reported in 1922.[1] It was proposed that monoorganotin acids exist either as cyclic trimers or as chainlike hydroxytin ethers; however, these structural elements could not be confirmed unambiguously.[2] Functionalized organotin compounds are used in organic synthesis[3] and catalytic reactions.[4] Very recently Chandrasekhar et al. synthesized a hexaferrocene–tin–oxygen cluster containing a *cyclo*-tristannoxane-like structure by the reaction of *n*-butylstannonic acid with ferrocene monocarboxylic acid.[5] Basic hydrolysis of diorganotin dihalides also yields tin–oxygen heterocycles,[6, 7] which show no additional sub-

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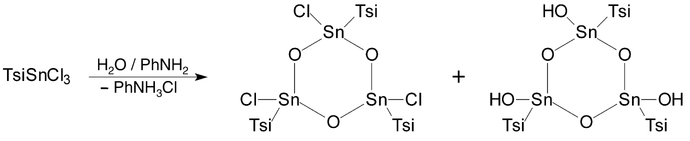
stituents on the ring apart from the organic ligands. Further derivatization of these compounds is rather limited due to the strong tin–carbon bond.[8]

A formal hydrolysis product of a monoorganotin trihalide was isolated for the first time in 1999: The reaction of $TsiSnBr_3$ (Tsi = $(Me_3Si)_3C$) with Na_2O in liquid ammonia gave a product of the composition $[(TsiSn)_4O_6]$ with a heteroadamantane-like structure. In this compound the tin atoms possess tetrahedral geometry.[9] In contrast, basic hydrolysis of nBuSnCl$_3$ results in amorphous products, for which a polymeric structure of the composition $[\{n\text{BuSn(OH)O}\}_n]$ with only four-coordinate tin atoms was proposed.[10] Mössbauer spectroscopic investigations by Davies et al. confirmed the tetrahedral coordination sphere of the tin atoms.[11] Subsequent ^{119}Sn MAS NMR measurements, however, suggest only 80% of the tin atoms in [nBuSn(OH)O] display tetrahedral geometry and nearly 20% are five-coordinate.[12]

Starting from $TsiSnCl_3$ (**1**),[13] we attempted controlled hydrolysis reactions under mild conditions in the absence of alkali metal hydroxides, because the latter are known to lead to decomposition reactions at the silicon-substituted organic ligands.[6] We used weakly basic aniline to trap the hydrochloric acid formed during the reaction, to avoid reactions between the nitrogen-containing base and the Lewis acidic tin atom. Surprisingly, the reaction of **1** with water yields the trimeric chloro compound [{TsiSn(O)Cl}$_3$] (**2**) as the main product, whereas the expected stannonic acid [{TsiSn(OH)O}$_3$] (**3**) is formed only as a by-product (Scheme 1).



1 **2** **3**

Scheme 1. Reaction of **1** with H_20 in the presence of aniline. Tsi = $C(SiMe_3)_3$.

The tetrahedral coordination of the tin atoms in compounds **2** and **3** was confirmed by characteristic resonance signals in the ^{119}Sn NMR spectra (**2**: $\delta = -133, -141$; **3**: $\delta = -156$). These data are consistent with ^{119}Sn NMR resonances of tin compounds displaying similar coordination.[14] Single crystals of the amine adduct of **3** were obtained from a solution of the crude product in a mixture of n-pentane and neopentyldimethylamine (4:1) and were characterized by X-ray crystallography.[15, 16]

The reaction of **1** with water yields **3** in a yield of about 2%, which indicates that the Sn–Cl bond in **2** is very stable towards hydrolysis. Compound **3** can be obtained in a 50% yield by treatment of **2** with LiOH in n-hexane under reflux [Eq. (1)].

$$\mathbf{2} + \text{LiOH} \xrightarrow[n\text{-hexane}]{\Delta/28\,\text{days}} \mathbf{3} \qquad (1)$$

NMR spectroscopic investigations of the compounds **2** and **3** indicate that the trifunctionalized *cyclo*-tristannoxanes exist as different conformers. The NMR spectra recorded for **2** show two resonance signals for each of the nuclei (^{1}H, ^{13}C, ^{29}Si, ^{119}Sn). The ^{1}H NMR spectrum recorded at 80 °C shows that **2** is stable to thermal-induced rearrangement reactions up to this temperature.

Compound **3**·tBuCH$_2$NMe$_2$ has an ideal chair conformation (Figure 1). The three organic ligands are bonded to the Sn_3O_3 heterocycle in equatorial positions. The three axial hydroxy groups show a *cis* arrangement. The amine is located between two organic ligands (not shown). Interactions through hydrogen bonds between the amine tBuCH$_2$NMe$_2$ and **3** were not observed. Compounds **2** and **3** were compared by NMR spectroscopy. Unlike for **2** the NMR spectra for **3** show only one resonance signal for the relevant nuclei (^{1}H, ^{13}C, ^{29}Si, ^{119}Sn). The tin–oxygen bond lengths range from 1.958 to 1.970 Å within the six-membered ring of **3**·tBuCH$_2$NMe$_2$ and are comparable with those in known Sn_3O_3 heterocycles.[6, 7] The distances between tin and the axially bonded oxygen atoms (1.968 to 1.977 Å) are in the same range as those in hydroxy-substituted tin–oxygen clusters.[18] The tin–carbon bond lengths (2.145 to 2.156 Å) are similar to those in Tpsi-substituted tin compounds (Tpsi = $(PhMe_2Si)_3C$).[13a]

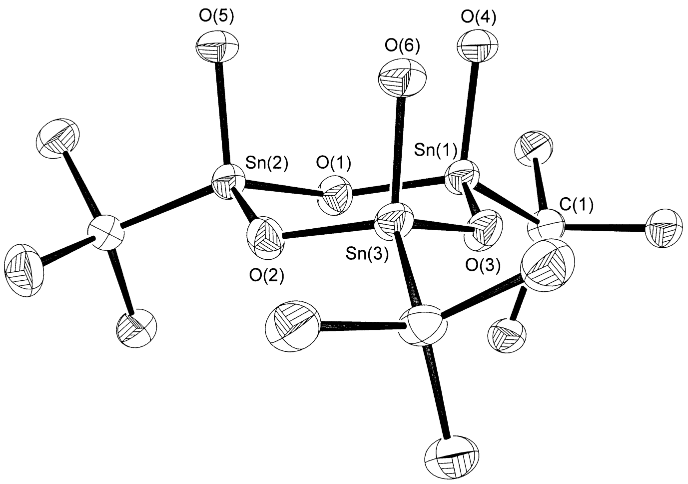


Figure 1. Molecular structure of **3**·tBuCH$_2$NMe$_2$ (ORTEP drawing,[20] thermal ellipsoid plot (50% probability)). The hydrogen atoms, methyl groups, and the amine were omitted for clarity. Selected bond lengths [Å] and angles [°]: Sn(1)-O(3) 1.965, Sn(1)-O(4) 1.968, Sn(1)-C(1) 2.156; O(3)-Sn(1)-O(1) 104.99, O(3)-Sn(1)-O(4) 106.06, O(3)-Sn(1)-C(1) 114.4, O(4)-Sn(1)-C(1) 110.5.

The presented synthesis and X-ray single-crystal structure analysis of the trimeric monoorganotin acid [{TsiSn(OH)O}$_3$] (**3**) provides a clarification of the much-discussed structure of this type of compounds. The structure is consistent in terms of the tetrahedral coordination of the tin atoms with recent ^{119}Sn MAS NMR investigations of [nBuSn(OH)O]. We eagerly anticipate a multitude of new products through the substitution of the oxygen-bonded hydrogen atoms.

Experimental Section

1: $SnCl_4$ (40.91 g, 157.0 mmol) was added slowly to Et_2O (1500 mL) at −78 °C. A solution of TsiLi·2THF (40.00 g, 104.5 mmol) in Et_2O (1500 mL) was added slowly under vigorous stirring to this freshly prepared suspension of $SnCl_4$–Et_20 adduct. After the addition was complete, the reaction mixture was allowed to warm to room temperature and was stirred for another 5 h. Finally all volatile components were removed in vacuo and the residue was dissolved in *n*-pentane (2000 mL). The insoluble components were filtered off and the colorless solution was concentrated under reduced pressure. Compound **1** crystallized as a colorless solid (43.42 g, 95.1 mmol, 91 %). The physical data of **1** are consistent with those reported by Eaborn et al.[13a]

2: Compound **1** (35.00 g, 76.6 mmol) was dissolved in *n*-pentane (1000 mL), and water (8.70 g, 482.6 mmol) and aniline (25.83 g, 277.3 mmol) were added to this solution. The resulting reaction mixture was heated under reflux for 21 days. Subsequently, all volatile components were removed in vacuo, and the residue was dissolved in *n*-pentane (1000 mL). The resulting solution was filtered and the solution was concentrated continuously under an inert atmosphere. Compound **2** crystallized as large colorless crystals (23.14 g, 19.2 mmol, 75 %). M.p. 295–300 °C; ^{1}H NMR (C_6D_6, 300 K, 500 MHz): $\delta = 0.47$ (s, 54 H; $(H_3C)_3Si\{Sn(1,2)\}$), 0.50 (s, 27 H; $(H_3C)_3Si\{Sn(3)\}$); ^{1}H NMR (C_6D_6, 353 K, 500 MHz): $\delta = 0.47$ (s, 54 H; $(H_3C)_3Si\{Sn(1,2)\}$), 0.50 (s, 27 H; $(H_3C)_3SiSn(3)$); ^{13}C NMR (C_6D_6, 300 K, 125 MHz): $\delta = 5.32$ (s; $(H_3C)_3Si\{Sn(3)\}$), 5.36 (s; $(H_3C)_3Si\{Sn(1,2)\}$), 26.71 (s; CSn(1,2)), 26.99 (s; CSn(3)); ^{29}Si NMR (C_6D_6, 300 K, 100 MHz): $\delta = 0.61$ (s; *Si*Sn(1,2), $^2J(^{29}Si,^{119}Sn) = 81.3$ Hz, $^2J(^{29}Si,^{117}Sn) = 77.8$ Hz), 0.76 (s; *Si*Sn(3), $^2J(^{29}Si,^{119}Sn) = 81.3$ Hz, $^2J(^{29}Si,^{117}Sn) = 77.8$ Hz); ^{119}Sn NMR (C_6D_6, 300 K, 186 MHz): $\delta = -141.0$ (Sn(3), $^2J(^{117}Sn,^{119}Sn) = 829$ Hz), -133.0 (Sn(1,2), $^2J(^{117}Sn,^{119}Sn) = 822$ Hz); elemental analysis calcd for $C_{30}H_{81}Cl_3O_3Si_9Sn_3$ (1205.16 g mol^{-1}) (%): C 29.90, H 6.77, Cl 8.83; found: C 30.56, H 6.87, Cl 9.19; IR (CCl_4): $\tilde{\nu} = 599$ w, 619 w, 661 w, 680 w, 756 vs, 768 vs, 856 vs, 1023 w, 1096 w, 1256 vs, 1269 vs, 1867 w, 1934 w, 2902 s, 2951 s, 2981 s cm^{-1}; MS (70 eV): *m*/*z*: 1205 [M^+], 1190 [$M^+ - Me$], 944 [$M^+ - TMS_3C - 2\,Me$].

3: A solution of **2** (400.0 mg, 0.33 mmol) in *n*-hexane (50 mL) was added to a suspension of LiOH (50.0 mg, 1.98 mmol) in *n*-hexane (50 mL). The reaction mixture was heated under reflux for 28 days. After the reaction was complete, all volatile components were removed in vacuo. The colorless residue was extracted for 5 h with *n*-pentane (75 mL). Finally the insoluble solid was filtered off and the resulting colorless solution was concentrated under reduced pressure. Fractional crystallization at −26 °C yielded **3** as a colorless solid (195 mg, 0.17 mmol, 50 %). M.p. 145–147 °C; ^{1}H NMR (C_6D_6, 300 K, 500 MHz): $\delta = 0.28$ (s, 81 H; $(H_3C)_3Si$);[19] ^{13}C NMR (C_6D_6, 300 K, 125 MHz): $\delta = 5.19$ (s; $(H_3C)_3Si$), 26.55 (s; CSn); ^{29}Si NMR (C_6D_6, 300 K, 100 MHz): $\delta = 0.26$ (s, $^2J(^{29}Si,^{119}Sn) = 74.5$, $^2J(^{29}Si,^{117}Sn) = 70.4$ Hz); ^{119}Sn NMR (C_6D_6, 300 K, 186 MHz): $\delta = -156.3$ (s, $^2J(^{117}Sn,^{119}Sn) = 719$ Hz); elemental analysis calcd for $C_{30}H_{84}O_6Si_9Sn_3$ (1149.83 g mol^{-1}) (%): C 31.34, H 7.36; found: C 30.92, H 7.41; IR (CCl_4): $\tilde{\nu} = 629$ w, 662 w, 682 w, 981 w, 1013 s, 1098 s, 1550 vs, 1734 w, 1867 w, 1936 w, 2905 s, 2957 s, 2986 s, 3588 w cm^{-1}.

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