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Crystal Structures of μ_2-Fluoro- and μ_2-Salicylaldoximato-Bridged μ_3-Oxo-tris(dimethyltin(IV)) Bis(salicylaldoximate)

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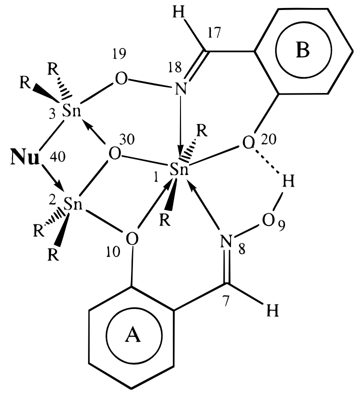
*Summary: Crystallographic data for [$(Me_2Sn)_2(Me_2SnO)$(**Nu**)(HONZO)(ONZO)] where HONZOH is ortho-HON═CH−C_6H_4−OH, salicylaldoxime, have been obtained for **Nu** = ONZOH (**2**) and F (**4**). Both **2** and **4** display one seven- and two five-coordinate diorganotin moieties as basic features. The key points of interest in **2** are the E-configuration for ONZOH and the observation of an intramolecular O−H···N hydrogen bond which is shown, by comparison with a related structure where such an interaction is precluded, not to impart a special stability in the structure. In **4**, a bridging F is present. The F atom forms a stronger interaction with one of the Sn atoms of the cyclic Sn_2OF unit, which provides a structural explanation to the observation of regioselective attack of nucleophiles onto one of the five-coordinate tin atoms in these systems.*

Introduction

Recent research has been devoted to the structure[1−4] and reactivity[3,4] of trinuclear tin clusters obeying the general formula [$(R_2Sn)_2(R_2SnO)$(**Nu**)(HONZO)(ONZO)] (Figure 1),[1−4] where **Nu** represents a nucleophilic group μ_2-bridging two five-coordinate tin atoms, R is a *n*-butyl[1] or methyl[2−4] group, and HONZOH represents symbolically *ortho*-HON═CH−C_6H_4−OH, salicylaldoxime.



	Nu	R
1	o-HOC$_6$H$_4$CHNO-	n-Bu
2	o-HOC$_6$H$_4$CHNO-	Me
3	CH$_3$O-	Me
4	F-	Me
5	C$_6$H$_5$CHNO-	Me

Figure 1. General structure of trinuclear tin salicylaldoximate complexes.

Compounds **1** (R = n-Bu) and **2** (R = Me) are of special interest in this series because they are invariably obtained as a primary reaction product in the condensation of salicylaldoxime with diorganotin oxide and play a key role in the generation of any other such trinuclear cluster. The stability and reactivity of **2** (as well as **1**) are believed to be related to the existence of a hydrogen bridge evidenced by IR and solution NMR data.[2,3] So far, neither **1** nor **2** could be isolated in crystalline form, as attempts at crystallization provided crystalline compounds with a different structure.[1−3] Thus, the μ_2-hydroxy-bridged analogue is obtained from **1**,[1] whatever the solvent used, indicating the involvment of trace amounts of atmospheric and/or residual solvent water. The μ_2-alkoxy-bridged analogues are obtained in crystalline form from **2**,[1] being isolated only when **2** is treated with low molecular weight alcohols, e.g., methanol in the preparation of **3**.[2]

On the other hand, **4** was recently isolated as the first trinuclear cluster of this type in which the μ_2-bridging atom in the **Nu** ligand (Figure 1) is not oxygenated.[4] Thus, **4** contains an *intra*molecularly μ_2-bridging fluoride ligand, a structural feature that remains uncommon in organic and inorganic tin fluorides,[5] μ_2-bridging fluoride being often *intermolecular* in the crystalline

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state.[6] The trinuclear clusters undergo facile reversible nucleophilic substitutions of their **Nu** ligand, provided the entering nucleophile is associated with exchangeable hydroxylic protons.[2-4] Compound **4** was particularly suitable to demonstrate that these nucleophilic substitutions with **Nu** ligands take place through an addition−elimination reaction.[4] Thus, the entering nucleophile first adds onto the Sn2 atom, generating an intermediate with a F−Sn3 bond, fluoride leaving as HF after transfer of a proton from the entering nucleophile, combined with formation of a new μ_2-bridged configuration involving the oxygen of the entering **Nu** ligand.[4] The conversion from **4** to **2** as well as to **3** was proven to take place through this mechanism.

This paper addresses two key structural issues concerning these trinuclear salicylaldoximates. First, the structural features of the hydrogen bond as well as of the *Z*- or *E*-configuration of the μ_2-bridging H−C=N−O moiety of compound **2** are still unresolved,[2,3] as no X-ray diffraction data are available to detail the exact bonding modes involved; these data should provide insight into the influence of the intramolecular hydrogen bridge on the overall stability of compound **2**. Second, for the same reason, no structural basis exists to explain the regiospecific attack of the entering ligand onto tin atom Sn2 rather than Sn3 of **4** during the generation of **2** and **3**. The present paper provides an answer to each of the above questions on the basis of X-ray diffraction data.

Results and Discussion

Crystal Structure of 2. Crystals of compound **2** suitable for X-ray diffraction analysis could be finally obtained as a result of a complex crystallization procedure, differing markedly from previous ones used for similar clusters.[2,3] They were isolated after crystallization of **2** under darkness and moisture-free conditions, by slow room-temperature evaporation from a 1/1 (v/v) mixture of dry benzene and hexane.

The overall structure of **2**, illustrated in Figure 2, is, up to the bonding mode of the μ_2-bridging salicylaldoximate moiety, basically that previously[3] proposed from earlier solution NMR studies.[1-3] Selected interatomic parameters are collected in Table 1.

The trinuclear structure features tridentate HONZO and ONZO anions, a trans-C_2 pentagonal bipyramidal Sn(1) center, two trans-O_2 trigonal bipyramidal Sn(2) and Sn(3) atoms, and a tricoordinate O(30) atom that links the three tin centers.[1-3] The μ_2-salicylaldoximate ligand is orientated in an approximately perpendicular position with respect to the trinuclear cluster, with the dihedral angle between the Sn_2O_2 atoms and the 10 atoms comprising the oxime function being 84.9°. Two intramolecular hydrogen bonds are found in the structure with O(9)−H···O(20) of 1.86 Å and O(50)−H···N(38) of 1.92 Å. The closest non-hydrogen intermolecular contact in the lattice of 3.373(9) Å occurs between the O(9) and $C(25)^{ii}$ atoms (O(9)···H(25) is 2.84 Å and symmetry operation *ii* is $-1 + x, +y, +z$). The presence of the intramolecular O(50)−H···N(38) contact clearly rules out an early bonding mode proposal,[1,2] based on inconclusive IR and solution NMR data, for the μ_2-bridging H−C=N−O moiety, where the intramolecular phenolic hydrogen bridge was believed to be oriented toward the O40 oxygen, with a cis oximic configuration. The present result indicates therefore that this intramolecular hydrogen bridge plays no significant role in the overall stabilization of the μ_2-bridging H−C=N−O(Sn)$_2$ moiety, explaining, at least in part, the sensitivity of this unit to hydrolysis[1,2] and to nucleophilic attacks.[2,3] This statement is consistent with the similar bonding mode found for the μ_2-benzaldoximato analogue of **2**, compound **5**, where such an intramolecular O(50)−

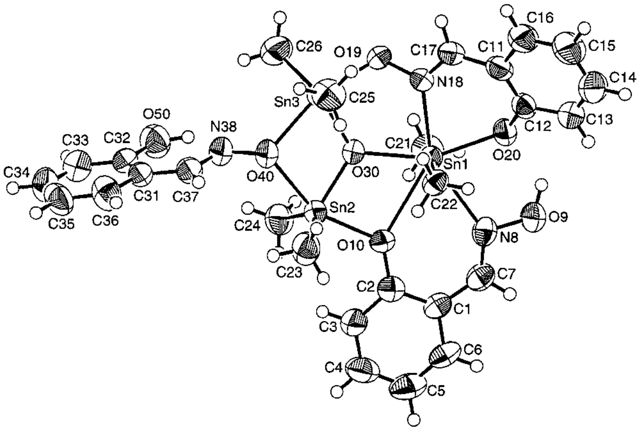

Figure 2. Molecular structure and atomic numbering scheme for **2**.

Table 1. Selected Interatomic (Å, deg) Parameters for 2 and 4

	2[a]	**4**[b]
Sn(1)−O(10)	2.729(4)	2.706(8)
Sn(1)−O(20)	2.186(4)	2.169(9)
Sn(1)−O(30)	2.191(4)	2.207(9)
Sn(1)−N(8)	2.614(5)	2.57(1)
Sn(1)−N(18)	2.282(5)	2.31(1)
Sn(2)−Y(40)	2.282(5)	2.231(8)
Sn(2)−O(10)	2.127(4)	2.126(9)
Sn(2)−O(30)	2.001(4)	1.998(8)
Sn(3)−Y(40)	2.239(4)	2.185(7)
Sn(3)−O(19)	2.089(4)	2.07(1)
Sn(3)−O(30)	2.019(4)	1.986(8)
O(9)−N(8)	1.404(6)	1.38(1)
O(19)−N(18)	1.390(5)	1.39(1)
N(8)−C(7)	1.276(7)	1.28(2)
N(18)−C(17)	1.281(7)	1.32(2)
O(10)−Sn(1)−O(30)	65.0(1)	64.3(3)
O(10)−Sn(1)−N(8)	65.4(1)	66.7(3)
O(20)−Sn(1)−N(8)	73.8(2)	74.7(3)
O(20)−Sn(1)−N(18)	77.3(2)	77.1(4)
O(30)−Sn(1)−N(18)	78.4(2)	77.3(4)
C(21)−Sn(1)−C(22)	166.9(2)	167.8(5)
Y(40)−Sn(2)−O(10)	154.0(1)	153.2(3)
C(23)−Sn(2)−C(24)	129.9(3)	132.8(6)
Y(40)−Sn(3)−O(19)	157.2(2)	161.8(3)
C(25)−Sn(3)−C(26)	130.8(3)	129(1)

[a] Y = O. [b] Y = F.

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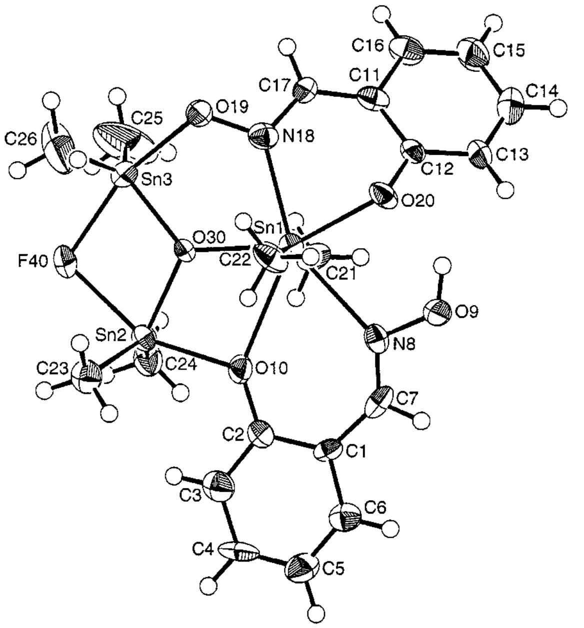

Figure 3. Molecular structure and atomic numbering scheme for **4**.

H···N(38) contact, by definition, cannot exist. Further evidence for the above conclusion is found in the invariance of the dihedral angles formed between the Sn_2O_2 units and the **Nu** groups, i.e., 84.9° for **2** and 84.6° for **5**. The absence of significant role of the intramolecular O(50)−H···N(38) contact in the overall stability of such trinuclear tin salicylaldoximates is in contrast with that of the O(9)−H···O(20) contact, found in all compounds of this type,[1−3] which obviously contributes to the overall stability of the seven-coordinate Sn1 atom. The latter appears indeed never to be affected directly by such nucleophilic attacks, unlike the five-coordinate Sn2 and Sn3 atoms.[1−4]

Crystal Structure of 4. Crystals of **4** suitable for X-ray diffraction could only be obtained after a tedious purification and crystallization procedure[4] (see Experimental Section). The molecular structure of **4** is shown in Figure 3, and selected interatomic parameters are collected in Table 1.

The general features of the structure are similar to those described for related compounds[1−3] and for **2**, with the key difference being the presence of a μ_2-F atom that forms a slightly dissymmetric bridge with Sn(2)−F and Sn(3)−F distances of 2.231(8) and 2.185(7) Å, respectively. The intramolecular hydrogen bond O(9)−H···O(20) is 1.90 Å. The structure is essentially molecular, with the most significant intermolecular contact of 2.49 Å occurring between the F(40) and $H(7)^i$ atoms [F(40)···$C(7)^i$ is 3.40(1) Å and symmetry operation i is $-0.5 + x, -y, 0.5 - z$]. The structure of compound **4** reveals a novel example of a μ_2-F atom involved in a four-membered ring structure of the type Sn−O−Sn−F.

Some comments can be made from the Sn(2)−F and Sn(3)−F distances above. First, the observation that the Sn(2)−F bond is longer and hence weaker than the Sn(3)−F one offers some indirect support to the regiospecific attack of nucleophiles onto tin atom Sn2 rather than Sn3.[4] However, this finding should not be overinterpreted because there is no evidence favoring or disfavoring the proposal that the Sn(2)−F and Sn(3)−F bond lengths are identical in the solid and solution states. Second, the corresponding $^1J(^{119}Sn-^{19}F)$ coupling constants, being 1285 and 1271 Hz[4] for the Sn(2)−F and Sn(3)−F bonds, respectively, display too low a difference for being of any significant use, neither in geometrical considerations as to the Sn−F bond lengths in solution nor as a support to the regiospecific attack onto Sn2 rather than Sn3.[4]

Actually, a literature overview confirms that no clear relationship can be established so far between $^1J(^{119}Sn-^{19}F)$ coupling constants and Sn−F bond distances alone, whenever NMR data are available, and that the substituent pattern plays a major role in addition to whether the Sn−F bond is terminal or bridging. Also a wide spread is observed in Sn−F bonds. In $[Me_2N(CH_2)_3]_2SnF_2$, where no bridging fluorine exists in solution, the $^1J(^{119}Sn-^{19}F)$ coupling constant amounts to ca. 2780 Hz, with the Sn−F bond distance being 2.084(6) Å.[6a]

For Me_3SnF,[6g−i] where two unequal Sn···F contacts are found, with bond distances of 2.15 and 2.45 Å, a single $^1J(^{119}Sn-^{19}F)$ coupling constant of 1300 Hz is found in the solid state. For Ph_3SnF,[6b,c] a single coupling constant of 1530 Hz is likewise found for a polymeric structure in which both Sn···F contacts are found identical, with a Sn−F bond distance of 2.1458(3) Å. In the case of Bz_3SnF,[6b] for which contacts of 2.12 and 2.21 Å are reported,[6f] as well as in $(cHex)_3SnF$,[6e] where the difference between the two bond lengths, respectively 2.051(10) and 2.303(10) Å, is somewhat larger, no coupling data are reported. Without structures known so far, the coupling constants of 1291 and 1260 Hz for i-Bu_3SnF and n-Bu_3SnF,[6b] respectively, suggest similar polymeric structures as for Ph_3SnF. By contrast, for Mes_3SnF (Mes = mesityl),[6b] two unequivalent molecules with single, very short Sn−F bonds (1.957(4) and 1.965(4) Å) were found in the crystal lattice, indicating a monomeric structure with the closest intermolecular contact being as high as 4.461 Å. Accordingly, two solid-state coupling constants of 2300 and 2256 Hz, characteristic for terminal Sn−F bonds, have been found.[6b] For $N(CH_2CH_2CH_2)_3Sn-F$,[6d] appearing as a dimer in the solid state with Sn−F bonds of 2.121(5) and 2.115(6) Å, two individual molecules display an intermolecular Sn···F contact of 2.797(6) Å. In solution, a temperature-dependent $^1J(^{119}Sn-^{19}F)$ coupling constant of ca. 2500 Hz indicates loss of the latter intermolecular contact. Other works on R_3SnF compounds provide $^1J(^{119}Sn-^{19}F)$ coupling constants, (*cis*-3-(benzyloxy)cyclohexyl)-fluorodimethylstannane, 2095 Hz;[6j] $(PhMe_2Si)_3CSnFMe_2$, 2367 Hz;[6k] $(Me_3Si)_3CSnFMe_2$, 2376 Hz;[6k] $(Me_3Si)_3CSnFPh_2$, 2463 Hz[6k]), and Sn−F bond lengths of bridging fluorines in dimeric $SnFMe_2$−1,4-cyclohexadiene−COOMe, with a strong Sn−F bond of 1.974(8) Å and a weak Sn′−F contact of 3.641(7) Å,[6l] as well as terminal fluorines in [2-(fluorodimethylstannyl)ethyl]diphenylphosphine oxide, 2.035(2) Å,[6m] the latter two compounds being six- and five-coordinate, respectively.

In the fluorinated ladder compound $[(t\text{-}Bu_2FSn)_2O]_2$ recently described,[5d] where likewise terminal and bridging fluorine atoms are found, coupling constants on the order of 2500 and 700−900 Hz, respectively, are found.

In the seven-coordinate (tris(2,6-dimethoxyphenyl)-methyl)tin trifluoride,[6n] with three very short Sn−F bonds (1.956(6), 1.948(7), and 1.975(6) Å), an exceptionally high $^1J(^{119}Sn-^{19}F)$ coupling constant of 3390 Hz is found for these terminal fluorines.

In Sn_4OF_6,[5c] a polymeric network of bridging F and O atoms is found in which each F atom bridges two Sn atoms unsymmetrically with average $Sn-F_{short,long}$ contacts of 2.20 and 2.45 Å, respectively, distances that are significantly longer than in **4**.

In anionic compounds of the type $\{[(Ph_2XSn)_2(CH_2)_n]F\}^-$ ($n = 1-3$; X = F, Cl, Br, I),[5a] $^1J(^{119}Sn-^{19}F)$ coupling constants are generally smaller than in neutral compounds. They range from 2178 to 2211 Hz for terminal Sn−F bonds and 780 to 879 Hz for bridging Sn−F−Sn units, when $n = 1$ (X = F, Br, I), with bond distances of 2.004(2) and 1.995(2) Å for the terminal bonds, and ranging from 2.204(2) to 2.274(5) Å for the bridging Sn−F−Sn bonds, that is, much smaller couplings than in **4** for comparable bond distances (2.231(8) and 2.185(7) Å).[5a] The size of the ring generated by the bridging fluorine obviously plays a role, since for $n = 2$, X = Cl, the bridging Sn−F bond distances reduce to 2.178(4) and 2.197(4) Å, reflecting, on average, stronger bonds than in **4**, with a single solution $^1J(^{119}Sn-^{19}F)$ coupling constant of 1180 Hz.[5a] For other similar compounds, with $n = 2$ and 3, for which X-ray data are not available, terminal $^1J(^{119}Sn-^{19}F)$ coupling constants range from 2030 to 2100 Hz, bridging ones, from 1120 to 1270 Hz,[5a] showing a rather high variabilty in these values, evidencing the multiparameter dependence of such couplings.

In conclusion, the structural interpretation of $^1J(^{119}Sn-^{19}F)$ couplings should be done with extreme care, and the very high similarity of the two $^1J(^{119}Sn-^{19}F)$ coupling constants of **4**, associated with two bonds with different lengths and chemical reactivities,[4] may therefore appear somewhat surprising.

Experimental Section

Syntheses. The synthesis and basic characteristics of **4** have been described earlier.[4] The crystals of **4** used in this X-ray diffraction study were obtained by refluxing first crude **4** (existing as an equilibrium mixture of **4** and **2**),[4] in boiling hexane. Subsequently, the nondissolved fraction is filtered off, the filtrate being reduced at 50 °C to one-third of its volume at the rotavapor. The oil that separates off is removed, the remaining solution being allowed to stand for 24 h in the refrigerator, after which a precipitate separates off. Recrystallization of the latter precipitate after refluxing in hexane provided the crystals suitable for X-ray analysis.

The synthesis of **2**[2,3] was modified as follows. Ten grams (6.07 mmol) of dimethyltin oxide are added to a solution containing 8.32 g (6.07 mmol) of salicylaldoxime in 600 mL of benzene. The mixture is refluxed for 24 h, ca. 300 mL of solution being distilled off with a Dean−Stark apparatus as a water/benzene azeotrope. After 24 h, the undissolved suspension is filtered off warm; the clear, yellow filtrate is then evaporated at the rotavapor, yielding a foamy solid, crude **2**. Analytically pure crystals appropriate for X-ray analysis (ca. 230 mg) were obtained by allowing a solution of crude **2** (ca. 500 mg) to slowly evaporate from a mixture of dry benzene/hexane (v/v 1/1) under strict conditions of dryness and darkness. The characteristics of **2** were provided elsewhere.[2]

Table 2. Crystallographic Data for 2 and 4

	2	4
formula	$C_{27}H_{33}N_3O_7Sn_3$	$C_{20}H_{29}FN_2O_5Sn_3$
fw	867.6	752.5
cryst size, mm	0.16 × 0.27 × 0.32	0.03 × 0.40 × 0.44
color	pale-orange	colorless
temp, K	293	200
cryst syst	monoclinic	orthorhombic
space group	$P2_1/c$	*Pccn*
a, Å	9.71(1)	17.99(1)
b, Å	21.188(9)	25.434(7)
c, Å	16.195(5)	11.45(3)
β, deg	103.18(5)	90
V, Å^3	3245(3)	5238(8)
Z	4	8
D_{calcd}, g cm^{-3}	1.776	1.908
$F(000)$	1688	2896
μ, cm^{-1}	23.35	28.76
transmission factors	0.698−1	0.353−1
no. of data colld	8150	6688
θ_{max}, deg	27.5	25
no. of unique data with $I \geq 3.0\sigma(I)$	4384	3078
R	0.034	0.062
R_w	0.035	0.065
residual electron density, e Å^{-3}	0.43	1.18

Crystal and Molecular Structures of 2 and 4. Intensity data were measured on a Rigaku AFC6R diffractometer fitted with graphite-monochromatized Mo Kα radiation, $\lambda = 0.710\ 73$ Å, and employing the $\omega-2\theta$ scan technique. The data sets were corrected for Lorentz and polarization effects,[7] and an empirical absorption correction was applied in each case.[8] Relevant crystal data are given in Table 2.

The structures were solved by direct methods employing DIRDIF[9] and refined by a full-matrix least-squares procedure based on F.[7] Non-H atoms were refined with anisotropic displacement parameters, and H atoms were included in the models in their calculated positions; the O−H atoms in **2** were located from a difference map, but their positions were not refined. Final refinement details are collected in Table 2, and the numbering schemes employed are shown in Figures 2 and 3, which were drawn with ORTEP[10] at 50% probability ellipsoids. The teXsan[8] package, installed on an Iris Indigo workstation, was employed for all calculations.

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Supporting Information Available: Further details of the structure determination including atomic coordinates, bond distances and angles, and thermal parameters (15 pages). Ordering information is given on any current masthead page.

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