Main Group Metal Directed Self-Assembly in a Tetrameric Trimethyltin(IV) *N*-nitroso-*N*-phenylhydroxylaminato Complex Containing an Unprecedented 20-Membered Inorganic (Carbon-Free) Metallamacrocycle

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Tetrakis[trimethyl(*N*-nitroso-*N*-phenylhydroxylaminato)tin(IV)] was synthesized by reaction of cupferron ([PhN(O)NO]NH_4) with trimethyltin(IV) chloride. The complex was characterized by X-ray diffraction analysis, FT-IR and FT-Raman spectroscopy. An X-ray structural analysis of the title complex $[Me_3Sn(O_2N_2Ph)]_4$ reveals the first example of a bridging coordinated cupferronato ligand. This coordination pattern leads to an unprecedented inorganic 20-membered (carbon-free) metallamacrocycle $Sn_4O_8N_8$ in which four Me_3Sn and four PhN_2O_2 subunits are self-assembled.

Introduction

The rational design and construction of inorganic and organometallic metallamacrocycles by transition metal directed multicomponent self-assembly has had a major impact on supramolecular chemistry.[1−3] The incorporation of metal centres into supramolecular systems gives rise to novel electronic and/or magnetic properties[1] as well as fascinating structural features. Metallamacrocycles have been widely used in host-guest chemistry to understand self-assembly phenomena in natural systems.[3][4] Transition metal centres present a large variety of preferred coordination geometries (square-planar, tetrahedral or octahedral) which have been systematically used in the construction of supramolecular assemblies such as molecular racks,[5] ladders,[5] grids,[6] cylinders,[7] metallamacrocycles,[4][8] metallacyclic polygons[9] and helicates.[10] In contrast, the use of main group metals in supramolecular chemistry has been less well explored. Tin(IV) complexes are also known to display a wide range of coordination geometries around the metal centre, the most common being the penta- and hexacoordinate environments.[11] In the case of pentacoordination, the trigonal-bipyramidal (TBP) configuration is more common than square-pyramidal (SP). For example, the axial connections of nearly planar Me_3Sn(IV) units, with suitable bridging ligands, offer ready access to the preferred TBP coordination and may generate metallamacrocycles. This can be seen in the tetranuclear complexes $[Me_3Sn(O_2PPh_2)]_4$[12] and $\{Me_3Sn[C_5Me_5W(O)(\mu\text{-}O)_2]\}_4$[13] which contain 16-membered inorganic metallamacrocycles $Sn_4O_8E_4$ (E = P and/or W). In addition, the ligand subunits must have two binding sites in a correct arrangement and must be as rigid as possible in order to predispose the coordinating groups to the desired geometry.

The aim of this work is to design new metallamacrocycles exploring the structural features of the cupferronato ligand as well as the ability of the Me_3Sn(IV) unit to exhibit TBP geometry in inorganic ring systems.[12][13] We report an unprecedented 20-membered inorganic (carbon-free)[14][15] metallamacrocycle in the trimethyltin(IV) cupferronato complex $[Me_3Sn(O_2N_2Ph)]_4$. The compound was characterized by analytical and spectroscopic (FT-IR, FT-Raman) methods.[16] To elucidate the three dimensional structure of the macrocycle, X-ray diffraction analysis was performed.[16] The ammonium salt of *N*-nitroso-*N*-phenylhydroxylamine, [PhN(O)NO]NH_4 (cupferron) is a well-known analytical reagent for the quantitative determination of several metal ions.[17] The *N*-nitroso-*N*-phenylhydroxylaminato (cupferronato) anion, $[PhN(O)NO^-]$ has been extensively used as a ligand in transitional metal chemistry behaving as a μ^3-bridging or, in most cases, as a bidentate chelating ligand.[18] In all cases, the X-ray diffraction analysis of the investigated cupferronato metal complexes has revealed a rigid and planar structure of the N(O)NO unit.[18] To the best of our knowledge, however, no bidentate bridging coordination pattern of the cupferronato ligand has been described.

Results and Discussion

Reaction of the cupferron with Me_3SnCl (1:1 ratio) in water yields the new tetranuclear trimethyltin(IV) cupferronato complex, $[Me_3Sn(O_2N_2Ph)]_4$ as a colourless crystalline solid. Single crystals suitable for an X-ray diffraction analysis were obtained by slow concentration of an *n*-heptane solution at ambient temperature.

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The molecular structure with atom-labeling scheme of $[Me_3Sn(O_2N_2Ph)]_4$ is shown in Figure 1 and relevant geometrical parameters are summarized in Table 1.

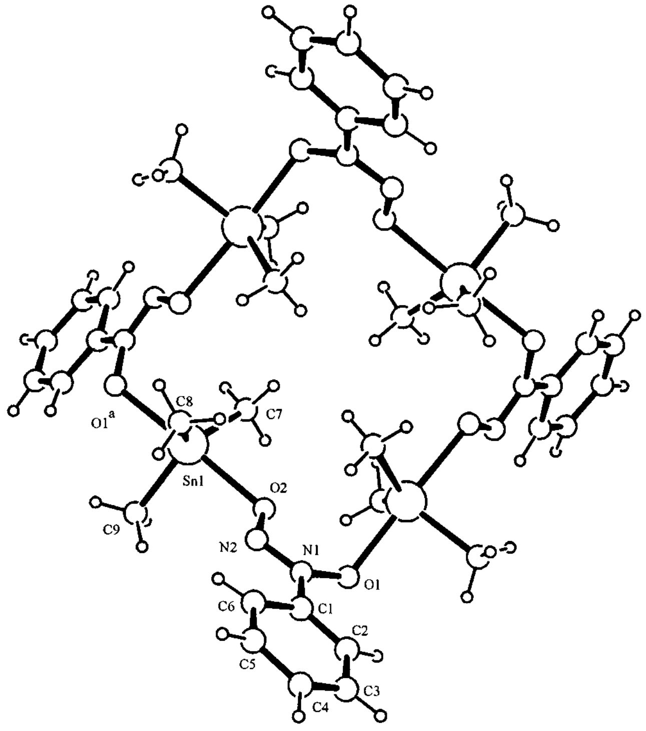

Figure 1. Molecular diagram of $[Me_3Sn(O_2N_2Ph)]_4$ with atom labeling for the asymmetric unit.

Table 1. Selected bond lengths (Å) and bond angles (°) for $[Me_3Sn(O_2N_2Ph)]_4$

Sn(1)−O(2)	2.3321(16)	O(1)[a]−Sn(1)−O(2)	175.79(6)
Sn(1)−O(1)[a]	2.2433(15)	O(1)−N(1)−N(2)	124.38(18)
O(1)−N(1)	1.321(2)	N(1)−N(2)−O(2)	112.63(18)
N(1)−N(2)	1.285(2)	C(7)−Sn(1)−C(8)	127.48(12)
N(2)−O(2)	1.293(2)	C(7)−Sn(1)−C(9)	116.83(13)
N(1)−C(1)	1.436(3)	C(8)−Sn(1)−C(9)	115.43(13)

[a] Equivalent atom generated by $1 - x$, $-1 + y$, $2 - z$.

The molecular structure of $[Me_3Sn(O_2N_2Ph)]_4$ consists of a novel 20-membered macrocyclic inorganic ring system $Sn_4O_8N_8$, self-assembled from four Me_3Sn units, connected by four bridging *N*-nitroso-*N*-phenylhydroxylaminato anions. It provides the first example of the bridging coordination pattern of the cupferronato ligand. Each metal centre occupies an almost perfect TBP coordination site defined by the oxygen atoms of two cupferronato ligands in a *trans* axial array [O(1a)−Sn(1)−O(2) = 175.75(7)°] and three methyl groups in the equatorial plane [average C−Sn−C = 119.91(12)°]. The distortion from the idealized TBP geometry can be quantified by the τ factor, where the limits of $\tau = 0$ or 1 are indicative[19] of perfect SP or TBP geometries, respectively. The calculated value of $\tau = 0.81$ reveals a strong TBP character of the Sn(IV) coordination environment. The Sn(1)−O(1) bond length is shorter by 0.089 Å than that of Sn(1)−O(2), but both are significantly longer than the normal Sn−O covalent bond.[11] The intramolecular nonbonded Sn(1)···O(2a) distance of 3.273(2) Å suggests a weak secondary interaction.

In each cupferronato anion the N(O)NO moieties are essentially planar [r.m.s. deviation from the best plane is 0.0127 Å; torsion angle: O(1)−N(1)−N(2)−O(2) = 4.0(3)°] and nearly coplanar with that of the phenyl ring [O(1)−N(1)−C(1)−C(2) = 5.6(3)°]. These structural features reveal a rigid arrangement of all the atoms in the cupferronato moieties. A significant electron delocalisation along the N(O)NO group can be predicted, all N−N and N−O bond lengths being intermediate between that of the corresponding single and double bond lengths.[16]

The title complex with $P\bar{4}2_1$ symmetry consists of a 20-membered cup-shaped metallamacrocycle $Sn_4O_8N_8$ (see Figure 2) in which the four Sn(1)−O(2) bonds lie in the best plane of the symmetry related Sn(1) and O(2) atoms [r.m.s. deviation from the best plane is 0.212 Å; torsion angles: Sn(1)−O(2)−Sn(1a)−O(2a) = 1.12(7)°, O(2)−Sn(1)−O(2b)−Sn(1b) = 20.68(9)°, Sn(1a)−O(2a)−Sn(1b)−O(2b) = 1.12(7)°, O(2c)−Sn(1c)−O(2a)−Sn(1a) = 20.68(9)°]. The bridging ligands are placed alternatively above and below this plane. In the same manner, two of the three methyl groups on each tin atom protrude alternately above and below the $\{\cdots Sn(1)-O(2)\cdots\}_4$ plane. This arrangement leads to two narrow rings defined by the C(7), C(7a), C(7b), C(7c) and C(8,) C(8a), C(8b), C(8c) atoms, which are concentric with the 20-membered metallamacrocycle. There are weak intramolecular hydrogen bonds such as C−H···O [C(8)···O(2), O(2)···H(8A) distances, 3.368(3), 2.899(12) Å, C(8)−H(8A)···O(2) angle 115.04°] and C−H···N [C(7)···N(1), N(1)···H(7A) distances 3.282(3), 2.732(10) Å, C(7)−H(7A)···N(1) angle 122.60°; C(7)···N(2), N(2)···H(7A) distances 3.412(3), 2.874(14) Å, C(7)−H(7A)···N(2) angle 121.88°] between the methyl hydrogen atoms and an adjacent cupferronato group and weak Me···Ph [C(8)···π, H(8A)···π distances 3.793, 3.054 Å, C(8)−H(8A)···π angle 142.63°] interactions between neighbouring groups.

The molecules self-organise in the solid state leading to a column arrangement along the shortest unit cell edge of 7.383(1) Å, analogous to the β-type packing observed in crystalline aromatic hydrocarbons (see Figure 3). If we rescale the short axis ranges given by Desiraju and Gavezotti for crystalline aromatic hydrocarbons[20] by the ratio of the covalent radii of Sn/C (1.82), the short axis for a β-packing must be less than 7.6 Å.[21] Indeed, the shortest axis of the title compound falls in this range in agreement with the β-packing assumed by the cup-shaped metallamacrocycles. These symmetry-related columns then interact with each other through weak C−H···π contacts [C(4)···π, H(4)···π distances 3.854, 3.150 Å, C(4)−H(4)···π angle 133.36°].

The spectral data are consistent with the determined structure of the title compound. The FT-IR and Raman spectra were recorded for the $[Me_3Sn(O_2N_2Ph)]_4$ complex and its corresponding neutral ligand $[PhN(O)NO]NH_4$ (cupferron) in the 3500−100 cm^{-1} range. Selected bands and assignments are listed in Table 2.

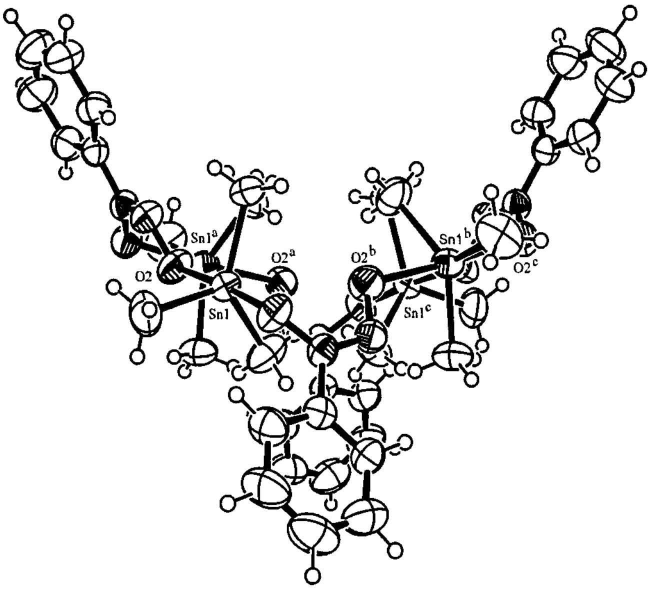

Figure 2. The side view of the molecular structure in $[Me_3Sn(O_2N_2Ph)]_4$; equivalent atoms are generated by: ^{a}: $1 - x$, $-1 + y$, $2 - z$; ^{b}: $-x$, $2 - y$, z; ^{c}: $1 + x$, $1 - y$, $2 - z$

Table 2. Selected FT-IR/Raman data and vibrational assignments for cupferron and $[Me_3Sn(O_2N_2Ph)]_4$[a]

$[PhN(O)NO]NH_4$ IR	Raman	$[Me_3Sn(O_2N_2Ph)]_4$ IR	Raman	Assignment
1335 s	1332 w	1344 s	1346 mw	ν(N−N)
1270 vs	1270 s	1224 vs	1221 vw	ν(N=O)
1220 vs	1226 m	1186 m	1187 w	
910 vs	910 mw	919 s	918 m	δ(ONNO)
		779 s		ρ(Me)
		555 m		$\nu_{as}(SnC_3)$
		542 m	546 mw	ν(Sn−C)
		520 w	516 s	$\nu_s(SnC_3)$
		387 vs		ν(Sn−O)
		142 s	141 m	$\delta(SnC_3$

[a] vw: very weak, w: weak, m: medium, mw: medium weak, s: strong, vs: very strong.

The assignments of the N−N and N=O stretching as well as ONNO bending modes − specific for the neutral ligand − in the 1500−800 cm^{-1} range of the IR and Raman spectra of the cupferron are in good agreement with similar IR data already published.[22][23] A comparison of IR and Raman spectra of the title complex with that of cupferron reveals significant changes in the position of the mentioned bands due to electron delocalisation over the coordinated ONNO unit. Thus, the ν(N−N) and δ(ONNO) modes are shifted to higher frequencies by 9 (IR)/14 (Raman) and 9 (IR)/8 (Raman) cm^{-1}, respectively, while the ν(N=O) mode is shifted to lower frequencies by 46/34 (IR) and 49/39 (Raman) cm^{-1}.

The vibrational modes specific to the coordination environment of tin may be assigned in the 800−100 cm^{-1} spectral range. The Me rocking vibration (779 cm^{-1} in the IR)[24] and the Sn−C stretching mode (542 in IR and 546 cm^{-1} in Raman)[24] are characteristic. Furthermore, the assignment of the three vibrational modes of the SnC_3 group (ν_{as}, ν_s and δ) in the IR spectrum of the complex are in good agreement with the vibrational behaviour proposed for a trigonal-plane arrangement of the Me groups about the tin(IV) centre.[24] The presence of the $\nu_s(SnC_3)$ − as a strong band − and $\delta(SnC_3)$ modes in the Raman spectrum support this structural feature.

Taking into account the coordination of the anionic cupferronato ligand to the metal centres through the oxygen atoms, the Sn−O stretching vibration, tentatively assigned at 565−560 cm^{-1},[22] was not found in the IR spectrum of the title complex. It is probably found at 387 cm^{-1}, as a very strong band.[23]

Experimental Section

General Remarks: All reagents and solvents were purchased from commercial suppliers and used without further purification. Elemental analyses were carried out in the laboratory of the University of Santiago de Compostela (Spain). FT-IR spectra were recorded on a Perkin−Elmer 2000 spectrometer. FT-Raman spectra

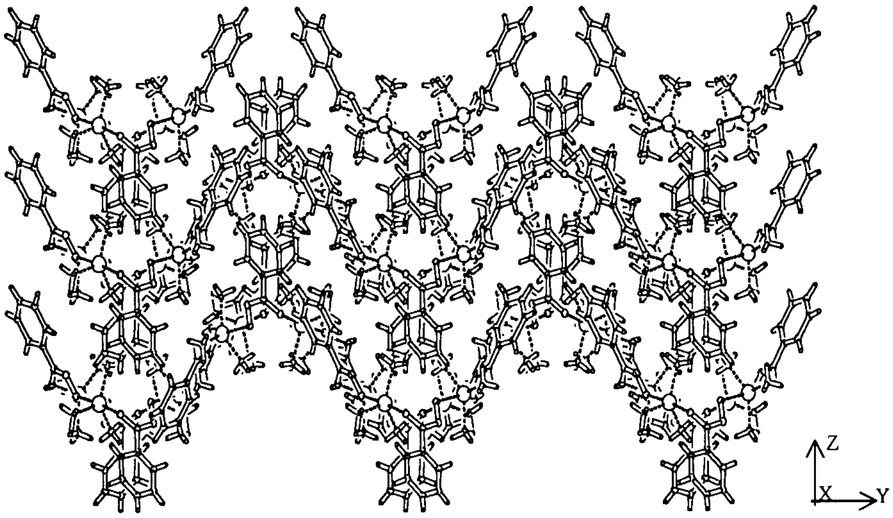

Figure 3. Crystal packing in $[Me_3Sn(O_2N_2Ph)]_4$; the molecules are stacked along the *c* axis

were recorded on a Bruker FT Raman spectrometer (IFS model 66).

Tetrakis[trimethyl(*N*-nitroso-*N*-phenylhydroxylaminato)tin(IV)]: $NH_4[PhN(O)NO]$ (0.20 g, 1.29 mmol) was dissolved in 10 mL water and this solution was added to a solution of Me_3SnCl (0.26 g, 1.30 mmol) in 10 mL water. The product was formed immediately as a pale yellow precipitate. After stirring for 20 minutes at room temperature, the mother liquor was removed and the product was dried. Recrystallisation from n-heptane gave the title compound as colourless crystals. Yield: 0.24 g (62%); m.p. 81−85°C. − FT-IR (KBr): $\tilde{\nu} = 1594\ cm^{-1}$ (C−H), 1487 (C−H), 1463 (C−H), 1344 (N−N), 1299 (C−H), 1224 (N=O), 1186 (N=O), 919 (ONNO), 779 (C−H), 755 (C−H), 689 (C−H), 555 (SnC_3), 542 (Sn−C), 520 (SnC_3), 387 (Sn−O), 280 (C−H), 230 (C−H), 142 (SnC_3). − FT-Raman: $\tilde{\nu} = 1594\ cm^{-1}$ (C−H), 1491 (C−H), 1346 (N−N), 1288, 1221 (N=O), 1187 (N=O), 1003 (C−H), 918 (ONNO), 546 (Sn−C), 516 (SnC_3), 141 (SnC_3). − $C_{36}H_{56}N_8O_8Sn_4$ (1203.73): calcd. C 35.9, H 4.7, N 9.3; found C 36.0, H 4.4, N 9.4.

X-ray Crystallographic Study: Crystal data for $[Me_3Sn(O_2N_2Ph)]_4$: $C_{36}H_{56}N_8O_8Sn_4$, $M = 1203.73$, tetragonal, space group $P\bar{4}2_1c$ (No. 114), with $a = b = 18.084(3)$, $c = 7.383(1)$ Å, $\alpha = \beta = \gamma = 90°$, $V = 2414.5(7)$ Å^3, $Z = 8$, $\rho_{calcd} = 1.656$ Mg/m^3, $F(000) = 1184$, $\lambda = 0.71073$ Å, $T = 293(2)$ K, $\mu(\text{Mo-}K_\alpha) = 2.096$ mm^{-1}, crystal size 0.45 × 0.25 × 0.24 mm. Intensity data of 11745 reflections were measured ($2.25 \leq 2\Theta \leq 34.98°$) on an Enraf−Nonius CAD-4 diffractometer (ω-2Θ scans) of which 5245 were independent ($R_{int} = 0.0197$). The intensities of the standard reflections indicated a crystal decay of 28% which was corrected. A semi-empirical (ψ-scan) absorption correction was also applied (min/max transmission: 0.933/0.974). The structure was solved by direct methods (SHELXS-97)[25a] and refined by full-matrix least squares (SHELXL-97).[25b] 5245 reflections were employed in the structure refinement (138 parameters, 0 restraints). The final R values were $R1 = 0.0238$ [$I \geq 2\sigma(I)$] and $wR2 = 0.0486$ (all data); min/max residual electron density −0.467/0.262 eÅ^{-3}. All non-hydrogen atoms were refined anisotropically. Hydrogen atomic positions were generated from assumed geometries. A riding model refinement was applied for the hydrogen atoms. Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-114414. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK [Fax: int. code +44−1223/336−033; E-mail: deposit@ccdc.cam.ac.uk].

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[21] In principle *mutatis mutandis* the tin macrocycles may adopt one of the four packing types characterized by the rescaled ranges β [*sa* ≤ 7.6 Å], γ [8.4 ≤ *sa* ≤ 9.8 Å], herringbone [9.8 ≤ *sa* ≤ 14.5 Å] and sandwich herringbone [*sa* ≤; 14.5 Å] (*sa*: the shortest crystal axis). The applicability of the empirical rescaling was tested on a 16-membered inorganic tin macrocycles. The $[Me_3Sn(O_2PPh_2)]_4$[12] complex crystallizes in an orthorhombic system (space group *Pnma*) with a short crystal axis of 14.501 Å which suggests a herringbone or sandwich-herringbone packing. Indeed, in this complex a sandwich-herringbone packing can be recognized. In contrast, in the oblique unit cell (space group $P\bar{1}$) of $[Me_3Sn(C_5Me_5W(O)(\mu\text{-}O)_2)]_4$[13] complex no such packing rules can be realised.

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