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$Et_4N[NO_3(SnClPh_3)_2(SnPh_3NO_3)]$: a trinuclear organostannate complex and related derivatives

Abstract: Four new di- and triorganonitrato stannate complexes and related derivatives have been synthesized and characterized by infrared and Mössbauer spectroscopy. The structure of $Et_4N[NO_3(SnClPh_3)_2(SnPh_3NO_3)]$ (**1**) has been determined by single-crystal X-ray diffraction analysis. In the trinuclear organostannate complex, each Sn^{IV} atom is five-coordinated and adopts a trigonal-bipyramidal *trans*-$OXSnC_3$ (X=O or Cl) geometry. The overall coordination environment of the Sn^{IV} atoms is trans-octahedral for **2** and **3** or cis-trigonal-bipyramidal for **4**. In the related derivatives, the NO_3^- anions behave as monodentate ligands.

Keywords: di- and triorganonitrato stannate; Mössbauer; octahedral; trigonal-bipyramida; trinuclear.

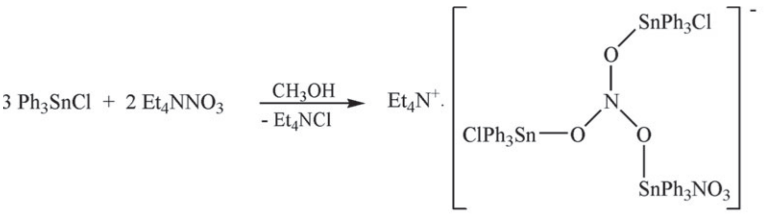
Introduction

The various applications of organotin(IV) compounds in many fields such as agriculture, medicine, antifouling paints, and wood preservatives are the main reasons several research groups are involved in the synthesis of new organotin(IV) compounds (Růžička et al., 2002; Thoonen et al., 2004; Basu et al., 2005; Hadjikakou and Hadjiliadis, 2009). Reports on structure determinations or spectroscopic characterizations of triphenyltin(IV) derivatives with mono- and polybasic oxyanions (XO_m^{n-}; X=Cr, Se, S, As; m=3, 4; n=1, 2, 3) show that the oxyanions behave mainly as polydentate ligands involving a one-dimensional polymeric, bi- or tridimensional network structure (Molloy et al., 1989; Diop et al., 2002; Diassé-Sarr et al., 2004; Fall et al., 2010; Boye et al., 2012). Some nitrato derivatives (Jurkschat et al., 2003a; García-Cuesta et al., 2004; Seward et al., 2007; Anjaneyulu et al., 2010; Hakimi et al., 2012) have also been reported. Our groups have published some articles dealing with $SnMe_3$- and $SnPh_3$-containing derivatives with mono- and polybasic oxyanions [SO_4^{2-}, $C_2O_4^{2-}$, $PhP(H)O_2^-$, $HAsO_4^{2-}$] (Sall et al., 1995; Diallo et al., 2009; Gueye et al., 2011; Diop et al., 2013). In this paper, we report on the reactions between tetraethylammonium nitrate (Et_4NNO_3) or dicyclohexylammonium nitrate ($Cy_2NH_2NO_3$) and mixing triphenyltin(IV) chloride ($SnPh_3Cl$) or SnR_2Cl_2 (R=Ph, Bu), which have yielded a trinuclear organostannate complex and related derivatives. The structure in the solid state of $Et_4N[NO_3(SnClPh_3)_2(SnPh_3NO_3)]$ has been determined by single-crystal X-ray diffraction analysis. All compounds have been characterized by Mössbauer and infrared spectroscopy.

Results and discussion

Molecular structure and spectroscopic characterization of $Et_4N[NO_3(SnClPh_3)_2(SnPh_3NO_3)]$ (1)

The reaction of triphenyltin chloride (Ph_3SnCl) with Et_4NNO_3 in the molar ratio 3:2 provided the trinuclear organostannate complex $Et_4N[NO_3(SnClPh_3)_2(SnPh_3NO_3)]$ (**1**) as a colorless crystalline material that shows good solubility in ethanol [Eq. (1)].



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The trinuclear organostannate complex consists of a discrete $[NO_3(SnClPh_3)_2(SnPh_3NO_3)]^-$ anion and a tetraethylammonium cation Et_4N^+ (Figure 1). The $[NO_3(SnClPh_3)_2(SnPh_3NO_3)]^-$ anion consists of two crystallographic independent $SnClPh_3$ moieties and one $SnONO_2Ph_3$ moiety that each show a distorted trigonal-bipyramidal environment as a result of their simultaneous coordination to the central nitrate anion. The axial positions are occupied by O(1), O(4) (Sn1), Cl(1), O(2) (Sn2), and Cl(2), O(3) (Sn3), whereas the equatorial positions are taken by the ipso carbon atoms of the corresponding phenyl substituents. The central nitrate anion is μ_3-bridging to three $SnPh_3$ moieties, and the second nitrate anion coordinates the Sn(1) atom in a monodentate mode. A similar arrangement has been observed in the crystal structure of poly[μ_2-chlorido-nonamethyl-μ_3-nitrato-tritin(IV)], $[(CH_3)_3Sn]_3NO_3Cl$, (Rehman et al., 2007) with a nitrate anion coordinated to three Sn atoms. The Sn-O distances are in the accepted range [Sn-O: 2.216(10) and 2.459(5) Å] and are shorter than the Sn-O distances in $[Ph_4P]^+[(Ph_2ClSn)_2CH_2.NO_3]^-$ [Sn-O: 2.510(5) and 2.545(5) Å] (Jurkschat et al., 2003b), which are very close to those observed in $Et_4NNO_3.SnPh_2Cl_2$ [Sn-O: 2.292(11) Å] (Diop et al., 2011). The sum of the O-N(1)-O angles subtended at NO_3^- μ_3-bridging [119.7° (6), 121.0° (6), and 119.3° (6)] is 360°, reflecting its trigonal-planar geometry. The C-N(70)-C angles of the cation are close to 109°, in agreement with the expected sp^3 hybridization.

Between the Et_4N^+ cations and the $[NO_3(SnClPh_3)_2(SnPh_3NO_3)]^-$ anion, the interactions are mainly of electrostatic nature. The infrared (IR) spectrum of the complex in the 4000–400 cm^{-1} region shows the presence of bands due to the nitrate ring vibrations at 1070 cm^{-1} (s) and 1061 cm^{-1} (s), (ν_1NO_3); 1334 cm^{-1} (vs), 1362 cm^{-1} (vs), and 1392 cm^{-1} (vs) ($\nu_3 NO_3^-$). The presence of two bands of $\nu_1NO_3^-$ reflects the two types of nitrates.

Selected bond distances (Å)

Sn(1)-O(4) 2.216(10); Sn(1)-O(1) 2.459(5); C(19)-Sn(2) 2.128(5); C(31A)-Sn(2) 2.188(12); Sn(2)-O(2) 2.502(5); Sn(2)-Cl(1) 2.512(4); C(37)-Sn(3) 2.134(5); C(43)-Sn(3) 2.125(4); C(49A)-Sn(3) 2.045(11); Sn(3)-Cl(2) 2.453(3); N(1)-O(2) 1.242(7); N(1)-O(1) 1.248(7); N(1)-O(3) 1.251(7); N(2)-O(4) 1.081(9); N(2)-O(5) 1.270(10); N(2)-O(6) 1.296(10); N(70)-C(77)#1 1.492(15); N(70)-C(71)#1 1.500(14); N(70)-C(73)#1 1.513(15).

Selected bond angles (°)

C(7B)-Sn(1)-C(1A) 119.8(6); C(1B)-Sn(1)-C(7A) 123.9(6); C(13)-Sn(1)-C(7A) 121.7(5); C(1A)-Sn(1)-C(7A) 116.5(6);

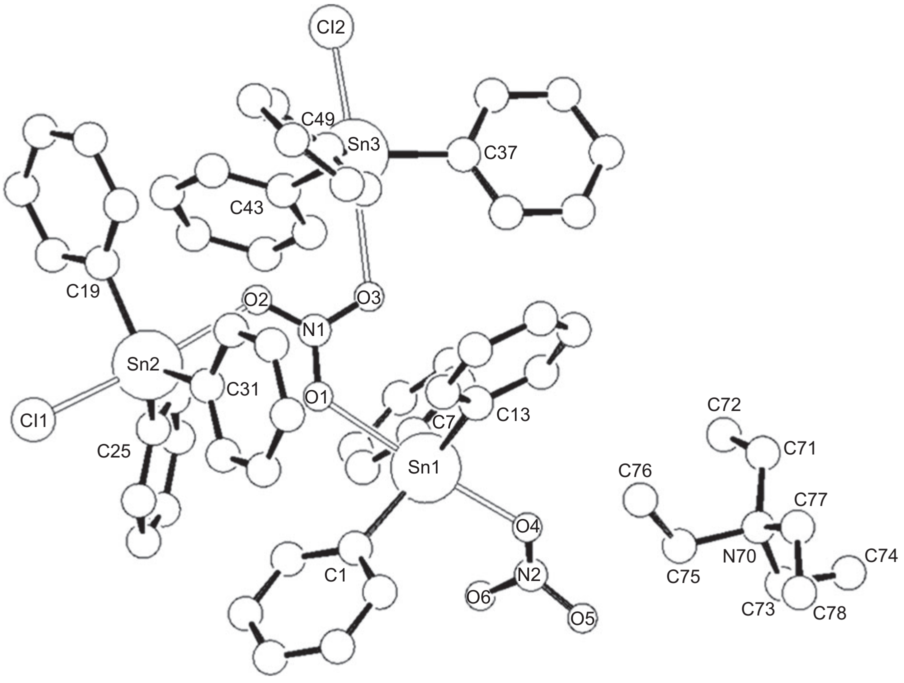


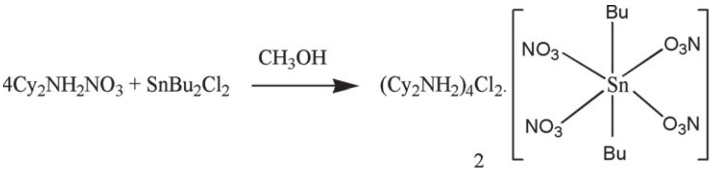
Figure 1 Molecular structure of $Et_4N[NO_3(SnClPh_3)_2(SnPh_3NO_3)]$ (**1**) showing the labeling scheme (the hydrogen atoms have been omitted for clarity). Symmetry codes: (′) *x*, *y*, *z* (″) -*x*, -*y*, -*z*; displacement ellipsoids are drawn at the 50% probability level.

C(7B)-Sn(1)-O(4) 96.8(5); O(4)-Sn(1)-O(1) 171.5(3); C(25A)-Sn(2)-C(31B) 117.1(8); C(25A)-Sn(2)-C(19) 116.8(5); C(31B)-Sn(2)-C(19) 124.2(5); C(25A)-Sn(2)-C(31A) 123.2(7); (49A)-Sn(3)-C(43) 122.9(5); O(2)-N(1)-O(1) 119.7(6); O(2)-N(1)-O(3) 121.0(6); O(1)-N(1)-O(3) 119.3(6); N(1)-O(1)-Sn(1) 125.4(4); N(1)-O(2)-Sn(2) 122.0(4); O(4)-N(2)-O(5) 125.3(11); O(4)-N(2)-O(6) 122.8(10); O(5)-N(2)-O(6) 111.8(10); N(2)-O(4)-Sn(1) 117.6(8); C(77)-N(70)-C(71) 110.1(12); C(77)-N(70)-C(73) 113.0(12); C(71)-N(70)-C(73) 112.9(11); C(77)-N(70)-C(75) 109.1(12).

A and B refer to the disorder of the molecular structure at the temperature measured.

Spectroscopic characterization of $(Cy_2NH_2)_4Cl_2SnBu_2(NO_3)_4$ (2)

Methanolic solutions containing $Cy_2NH_2NO_3$ and $SnBu_2Cl_2$ were mixed and stirred at room temperature for more than 1 h. The solution was allowed to evaporate to give powder of the title compound [Eq. (2)].



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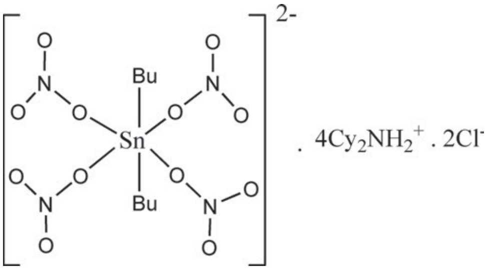
(2)

In comparison with the data reported for other nitrato adducts or derivatives (Addison et al., 1967; Nakamoto, 1997), we suggest the following IR band assignments to be made for the compound. The bands located at 1061 cm^{-1} (s, ν_1NO_3), 1335 cm^{-1} (vs), and 1370 cm^{-1} (vs), (ν_3NO_3) are assigned to the vibrations of the NO_3 groups.

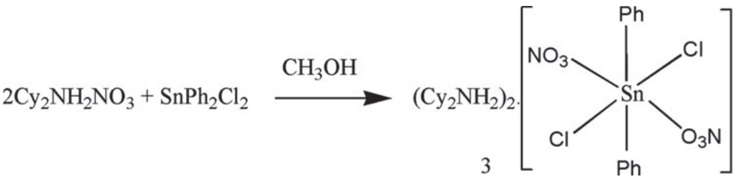
The absence of ν_sSnBu_2 in the IR spectrum indicates the *n*-Bu substituents to be trans. The Mössbauer spectrum of compound **2** shows a slightly asymmetric quadrupole split doublet with an isomer shift value (1.20 mm^{-1}) in the normal range for a diorganotin(IV) derivative (Davies and Smith, 1982). The value of the quadrupole splitting (3.78 mm/s) is consistent with a trans hexacoordinated $SnBu_2$ moiety (Scheme 1) according to Bancroft and Platt (1972). These $[SnBu_2(NO_3)_4]^{2-}$, including the Cl atom, anions are then connected by the cation through NH···O hydrogen bonds responsible for the strong IR absorptions at 3340 and 3215 cm^{-1}.



Scheme 1 Suggested structure for $(Cy_2NH_2)_4Cl_2SnBu_2(NO_3)_4$.

Spectroscopic characterization of $(Cy_2NH_2)_2SnPh_2Cl_2(NO_3)_2$ (3)

The title compound, $(Cy_2NH_2)_2SnPh_2Cl_2(NO_3)_2$ (**3**), was prepared by the condensation reaction of diphenyltin(IV) dichloride with $Cy_2NH_2NO_3$ in a 1:2 molar ratio [Eq. (3)].



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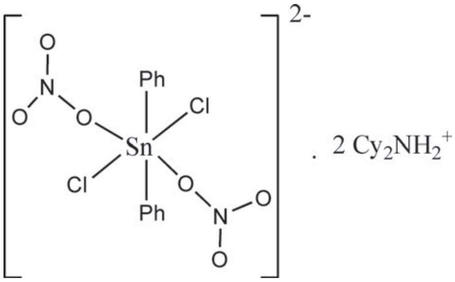
(3)

In the IR spectrum, the bands located at 1060 (s, ν_1NO_3), 1334, and 1375 (vs, ν_3NO_3) cm^{-1} are assigned to the stretching vibrations of the NO_3 groups. The value of the quadrupole splitting of (**3**) (QS=3.57 mm/s) is consistent

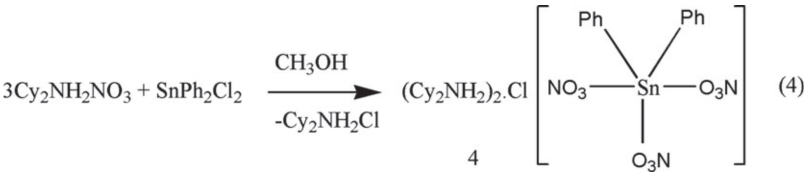


Scheme 2 Suggested structure for $(Cy_2NH_2)_2SnPh_2Cl_2(NO_3)_2$.

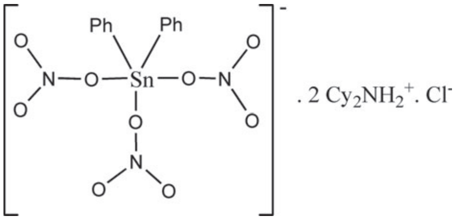
with the presence of a trans hexacoordinated $SnPh_2$ group according to Bancroft and Platt (1972). The suggested structure for (**3**) consists of a trans-octahedral geometry around the Sn^{IV} atom (Scheme 2). The dicyclohexylammonium cation connects adjacent anionic $[SnPh_2(NO_3)_2Cl_2]^{2-}$ layers through N-H···O hydrogen bonding into a three-dimensional network structure.

Spectroscopic characterization of $(Cy_2NH_2)_2ClSnPh_2(NO_3)_3$ (4)

$(Cy_2NH_2)_2ClSnPh_2(NO_3)_3$ (**4**) has been prepared by allowing $Cy_2NH_2NO_3$ to react with diphenyltin dichloride, Ph_2SnCl_2, in methanol in the molar ratio 1:3 [Eq. (4)] (Scheme 3).



The value of the quadrupole splitting (QS=3.07 mm/s) is consistent with a cis-trigonal-bipyramidal coordinated $SnPh_2$ residue according to Bancroft and Platt (1972). The overall coordination environment of the Sn^{IV} atom is trigonal-bipyramidal defined by two phenyl C atoms in cis positions and three nitrate O atoms. The dicyclohexylammonium cation connects adjacent anionic $[SnPh_2(NO_3)_3]^-$ layers through N-H···O hydrogen bonding into a three-dimensional network structure. The Cl atom can be involving in the hydrogen bonds network of supramolecular architecture.



Scheme 3 Suggested structure for $(Cy_2NH_2)_2ClSnPh_2(NO_3)_3$.

Experimental

Materials and spectroscopic methods

$SnPh_2Cl_2$, $SnBu_2Cl_2$, $SnPh_3Cl$, $AgNO_3$, Et_4NCl, HNO_3, and Cy_2NH were purchased from Aldrich or Merck Chemical Company and used without further purification.

The infrared spectra were recorded at the laboratory of control medicine (Dakar) by means of a Bruker FT-IR type spectrometer; the samples were prepared as KBr pellets. Elemental analyses were performed at the University of Bath (UK) using an Exeter Analytical CE440 analyzer. Infrared data are given in cm^{-1} (abbreviations: vs, very strong; s, strong; m, medium; w, weak). ^{119}Sn Mössbauer spectra were obtained from a constant-acceleration spectrometer moving a $CaSnO_3$ source at room temperature. The samples were analyzed at liquid N_2 temperature, and the isomer shift values are given with respect to that source. All the Mössbauer spectra were computer-fitted assuming Lorentzian lineshapes. Mössbauer parameters are given in mm/s (abbreviations: QS, quadrupole splitting; IS, isomer shift).

Synthesis of ligands

Et_4NNO_3 (**L_1**) was obtained on mixing $AgNO_3$ (0.50 g, 2.95 mmol) with tetraethylammonium chloride (Et_4NCl) (0.82 g, 2.95 mmol) both in water and filtering off the AgCl precipitate (Sall et al., 1995).

$Cy_2NH_2NO_3$ (**L_2**) was obtained as a precipitate on mixing an aqueous solution of Cy_2NH with NO_3H in 1:1 ratio. Analytical data: % found (% calc. for ligand): % C, 58.76 (58.99); % H, 9.56 (9.90); % N, 11.00 (11.47).

Synthesis of $Et_4N[NO_3(SnClPh_3)_2(SnPh_3NO_3)]$ (1)

Regular crystals of $Et_4N[NO_3(SnClPh_3)_2(SnPh_3NO_3)]$ suitable for X-ray work were obtained after a slow solvent evaporation of the solution obtained on $SnPh_3Cl$ (0.23 g, 0.6 mmol) with Et_4NNO_3

(0.12 g, 0.40 mmol) in methanol in 3:2 ratio [melting point (mp), +260°C; yield, 82%].

Analytical data: [% found (% calc.) for $C_{62}H_{65}Cl_2N_3O_6Sn_3$, 1375.14 g]: % C: 54.32 (54.15); % H: 4.34 (4.76); % N: 2.95 (3.06). IR (cm^{-1}, KBr): 3078 m (ν CH), 1070 s, and 1061 s (ν_1NO_3); 1334 vs, 1362 vs, and 1392 vs (ν_3NO_3).

X-ray crystallographic data of $Et_4N[NO_3(SnClPh_3)_2(SnPh_3NO_3)]$ (1)

A crystal of approximate dimensions 0.13 mm×0.08 mm×0.07 mm was used for data collection. Data were collected at 298(2) K using Mo-kα radiation (λ=0.71073 Å).

Refinement of F^2 against ALL reflections

The weighted *R* factor *wR* and goodness of fit *S* are based on F^2, and conventional *R* factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2>2\sigma(F^2)$ is used only for calculating *R* factors (gt), etc. and is not relevant to the choice of reflections for refinement. *R* factors based on F^2 are statistically about twice as large as those based on *F*, and *R* factors based on all data will be even larger.

Data collection

CrysAlis CCD, Oxford Diffraction (2009); cell refinement: CrysAlis RED, Oxford Diffraction (2009); data reduction: CrysAlis RED, Oxford Diffraction; program used to solve structure: SHELXS97 (Sheldrick, 2008); program used to refine structure: SHELX97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: enCIFer (Allen et al., 2004), PlATON (Spek, 1990, 1998), WinGX (Farrugia, 1999).

Crystal data and structure refinement

Empirical formula: $C_{62}H_{65}Cl_2N_3O_6Sn_3$; formula weight=1375.14; crystal system: triclinic; space group: *P*-1; *a*=14.0465(12) Å; *b*=15.0816(10) Å; *c*=16.0431(13) Å; α (°) = 85.611(6), β (°)=85.599(7), γ(°)=63.381(7); *V* (Å^3)=3026.0(4); *Z*=2; ρ_{calc} (mg/m^3)=1.509; μ(Mo-k$_\alpha$) (mm^{-1})=1.368; *F*(000): 1380; reflections collected: 23,179; independent reflections [*R*(int)]: 12,346 [0.062]; reflections observed (>2σ): 6014; absorption correction: semiempirical from equivalents; maximum, minimum transmission: 0.9103, 0.8422. Refinement method, full-matrix least squares on F^2; goodness of fit, 0.825; final *R* indices [$I>2\sigma(I)$]: 0.0659, 0.1588; *R* indices (all data): 0.1502, 0.1832; largest difference peak and hole (eÅ^3): 1.763, -0.672. The structure itself is highly disordered owing to the temperature measurement [298(2) K]. The crystallographic data for CCDC 859692 contain the supplementary crystallographic data for this paper.

Synthesis of $(Cy_2NH_2)_4Cl_2SnBu_2(NO_3)_4$ (2)

$(Cy_2NH_2)_4Cl_2SnBu_2(NO_3)_4$ (**2**) has been obtained by reacting (**L_2**) (0.20 g, 0.80 mmol) with dibutyltin(IV) dichloride ($SnBu_2Cl_2$) (0.06 g, 0.20 mmol) in methanol. After a slow solvent evaporation a white powder was collected in the solvent (81%, mp 200°C).

Analytical data: [% found (% calc.) for $C_{56}H_{114}Cl_2O_{12}Sn$, 1280.7 g]: % C: 52.56 (52.50); % H: 8.92 (8.97); % N: 8.70 (8.75). IR (cm^{-1}, KBr): 3340 s and 3215 s (ν NH_2), 3160 m (ν NH), 3078 m (ν CH), 1061 s (ν_1NO_3); 1335 vs and 1370 vs (ν_3NO_3), 669w (ν_{as} $SnBu_2$). Mössbauer data (mm/s): IS=1.20, QS=3.78.

Synthesis of $(Cy_2NH_2)_2SnPh_2Cl_2(NO_3)_2$ (3)

An ethanolic solution containing 0.10 g (0.4 mmol) of **L_2** and 0.07 g (0.2 mmol) of diphenyltin(IV) dichloride ($SnPh_2Cl_2$) was stirred at room temperature for more than 1 h. After a slow solvent evaporation of the solution a white powder was obtained (78%, mp 160°C).

Analytical data: [% found (% calc.) for $C_{36}H_{58}Cl_2N_4O_6Sn$, 832.28 g]: % C: 52.00 (51.94); % H: 7.00 (7.02); % N: 6.70 (6.73). IR (cm^{-1}, KBr): 3321 and 3000 m (ν NH_2), 3160 m (ν NH), 3072 m (ν CH), 1060 s (ν_1NO_3); 1334 vs, and 1375 vs (ν_3NO_3). Mössbauer data (mm/s): IS=1.25, QS=3.57.

Synthesis of $(Cy_2NH_2)_2ClSnPh_2(NO_3)_3$ (4)

$(Cy_2NH_2)_2ClSnPh_2(NO_3)_3$ (**4**) has been obtained by reacting 0.10 g (0.4 mmol) of **L_2** with 0.05 g (0.13 mmol) of diphenyltin(IV) dichloride ($SnPh_2Cl_2$) in methanol. After a slow solvent evaporation a white powder was collected in the solvent (79%, mp 173°C).

Analytical data: [% found (% calc.) for $C_{36}H_{58}ClN_5O_9Sn$, 859.04 g]: % C: 50.23 (50.33); % H: 6.80 (6.81); % N: 8.40 (8.15). IR (cm^{-1}, KBr): 3340 and 3215 m (ν NH_2), 3160 m (ν NH), 3078 m (ν CH), 1061 s (ν_1NO_3); 1340 vs, and 1370 vs (ν_3NO_3). Mössbauer data (mm/s): IS=1.30, QS=3.07.

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