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Note

Sn–S and Ru–Ru bonds cleavage reactions between [$Ph_3SnS(CH_2)_3SSnPh_3$] and $Ru_3(CO)_{12}$: X-ray crystal structures of [$Ph_3SnS(CH_2)_3SSnPh_3$] and *trans*-[$Ru(CO)_4(SnPh_3)_2$]

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ABSTRACT

The organotin complex [$Ph_3SnS(CH_2)_3SSnPh_3$] (**1**) was synthesized by $PdCl_2$ catalyzed reaction between Ph_3SnCl and disodium-1,3-propanedithiolate which in turn was prepared from 1,2-propanedithiol and sodium in refluxing THF. Reaction of **1** with $Ru_3(CO)_{12}$ in refluxing THF affords the mononuclear complex *trans*-[$Ru(CO)_4(SnPh_3)_2$] (**2**) and the dinuclear complex [$Ru_2(CO)_6(\mu\text{-}\kappa^2\text{-}SCH_2CH_2CH_2S)$] (**3**) in 20 and 11% yields, respectively, formed by cleavage of Sn–S bond of the ligand and Ru–Ru bonds of the cluster. Treatment of $pymSSnPPh_3$ (pymS = pyrimidine-2-thiolate) with $Ru_3(CO)_{12}$ at 55–60 °C also gives **2** in 38% yield. Both **1** and **2** have been characterized by a combination of spectroscopic data and single crystal X-ray diffraction analysis.

1. Introduction

Organotin complexes have become versatile reagents in organic and organometallic chemistry [1]. Ruthenium compounds derived from metal carbonyl cluster complexes combined with group 14 elements have been found to exhibit interesting catalytic properties [2–4]. The coordination of SnR_3 fragments to trimetallic clusters of ruthenium and osmium by oxidative addition of Sn–H bond of R_3SnH to give many interesting compounds is well documented [5–8]. It has recently been demonstrated that coordinated $SnPh_3$ is an excellent ligand for the introduction of $SnPh_2$ ligands to polynuclear metal carbonyl complexes [9–12]. On the other hand, the introduction of thiolato group in trimetallic clusters by oxidative addition of S–H bond of thiols is well established [13–19]. Recently, aminostannanes have been shown to react with transition metal hydride complexes to form metal–metal bonds and taking the advantage of this methodology, Garate-Morales and Fernandez-G have synthesized amine-containing osmium–tin compounds [$Os_3(CO)_{10}(HE)(SnBu_3)(\mu\text{-}H)$] (HE = amine) [20]. The organotin and thiolato fragments in organic compounds can be incorporated into organometallic cluster compounds resulting in the formation of M–Sn and M–S bonds (M = Os, Ru) and in this context, we have very recently reported some unusual bimetallic Ru–Sn, Ru–Ge clusters, e.g. [$Ru_3(CO)_8(\mu\text{-}SPh)_2(\mu_3\text{-}SnPh_2)(SnPh_3)_2$] and [$Ru_3(CO)_8(\mu\text{-}SPh)_2(\mu_3\text{-}GePh_2)(GePh_3)_2$], from the reactions of Ph_3SnSPh or Ph_3GeSPh with $Ru_3(CO)_{12}$, respectively (Scheme 1) [20]. We also reported the Os–Sn compound [$Os_3(CO)_9(\mu\text{-}SPh)(\mu_3\text{-}SnPh_2)(NCMe)(\eta^1\text{-}C_6H_5)_2$] from the reaction of [$Os_3(CO)_{10}(NCMe)_2$] with Ph_3SnSPh [21]. Given the potential of dithioorganotin compounds in the synthesis of triruthenium clusters containing bridging dithiolato and $SnPh_2$ ligands we have performed the synthesis of $Ph_3SnS(CH_2)_3SSnPh_3$. This paper provides an account of the synthesis, structure and reactivity of this ligand with $Ru_3(CO)_{12}$.

2. Experimental

All reactions were carried out under a nitrogen atmosphere. Reagent grade solvents were dried using appropriate drying agents and were freshly distilled prior to use. $Ru_3(CO)_{12}$ was purchased from Strem Chemicals Inc. and used without further purification. Infrared spectra were recorded on a Shimadzu FTIR 8101 spectrophotometer. ^1H NMR spectra was recorded on a Bruker DPX 400 instrument. All chemical shifts are reported in δ units with reference to the residual protons of the deuterated solvents. Electrospray mass spectra were recorded on a Bruker MicrOTof machine, using MeOH as solvent and NaOMe added as an ionisation aid [22]. The compound $pymSSnPPh_3$ was prepared according to the literature procedure [23].

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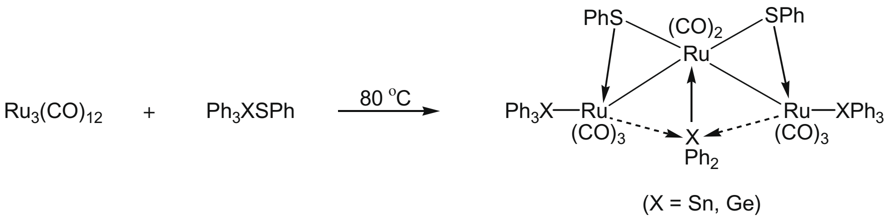


Scheme 1. Reaction of $Ru_3(CO)_{12}$ with Ph_3XSPh (X = Sn, Ge).

*2.1. Preparation of [$Ph_3SnS(CH_2)_3SSnPh_3$] (**1**)*

A mixture of 1,3-propanedithiol (243 mg, 2.25 mmol) and Na (103 mg, 4.51 mmol) in THF (40 mL) was heated to reflux for 4 h during which time all the Na reacted. To this was added dropwise a THF solution (10 mL) of Ph_3SnCl (1736 mg, 2.25 mmol) at 0 °C followed by 10 mg of $PdCl_2$ and the mixture was slowly warmed to room temperature and stirred overnight. After filtering the mixture, the filtrate was evaporated to dryness and the residue chromatographed by TLC on silica gel. Elution with hexane/CH_2Cl_2 afforded [$Ph_3SnS(CH_2)_3SSnPh_3$] (**1**) (600 mg, 34%) as colorless crystals from EtOH/THF at 4 °C. *Anal.* Calc. for $C_{39}H_{36}S_2Sn_2$: C, 58.11; H, 4.51. Found: C, 58.33; H, 4.59%. ^1H NMR ($CDCl_3$): δ 2.55 (t, 4H, J = 6.8 Hz), 1.70 (q, 2H, J = 6.8 Hz), 7.59 (m, 10H), 7.38 (m, 20H). ESI-MS: *m*/*z* 1155.048 [M+Ph_3Sn]$^+$ (calc. 1155.050); *m*/*z* 829.022 [M+Na]$^+$ (calc. 829.020).

*2.2. Reaction of $Ru_3(CO)_{12}$ with **1***

A THF solution (20 mL) of $Ru_3(CO)_{12}$ (100 mg, 0.15 mmol) and **1** (126 mg, 0.156 mmol) was heated to reflux for 3 h. The solvent was removed by rotary evaporation and the residue chromatographed by TLC on silica gel. Elution with hexane/CH_2Cl_2 (7:3, v/v) developed three bands. The first band was unreacted $Ru_3(CO)_{12}$ (trace). The second band gave *trans*-[$Ru(CO)_4(SnPh_3)_2$] (**2**) (28 mg, 20%) as colorless crystals after recrystallization from hexane/CH_2Cl_2 at 4 °C. *Anal.* Calc. for $C_{40}H_{30}O_4RuSn_2$: C, 52.62; H, 3.32. Found: C, 52.86; H, 3.38%. IR (νCO, CH_2Cl_2): 2032 vs cm^{-1}. ^1H NMR ($CDCl_3$): δ 7.62–7.49 (m, 12H), 7.37–7.34 (m, 18H). ESI-MS: *m*/*z* 944.948 [M+OMe]$^-$ (calc. 944.943). The third band afforded [$Ru_2(CO)_6(\mu$-κ^2-$SCH_2CH_2CH_2S)$] (**3**) (8 mg, 11%) as pale yellow crystals after recrystallization from hexane/CH_2Cl_2 at 4 °C. IR (νCO, hexane): 2086 s, 2055 vs, 2018 vs, 2004 s, 1994 s, 1966 w cm^{-1}. ^1H NMR ($CDCl_3$): δ 2.20 (m, 4H), 1.91 (m, 2H).

2.3. Preparation of pyrimidine-2-thiotriphenyltin, pymSSnPh$_3$

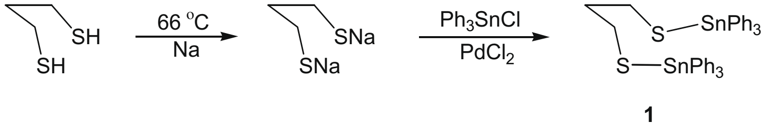
KOH (51 mg, 0.91 mmol) was added to a C_2H_5OH solution (20 mL) of 2-mercaptopyrimidine (100 mg, 0.89 mmol) and the reaction mixture was stirred for 30 min at room temperature. The color of the solution slowly changed from orange to colorless. Then Ph_3SnCl (343 mg, 0.89 mmol) was added to the reaction mixture and stirred for further 30 min at room temperature. The solution was filtered through a filter paper. The solvent was removed under reduced pressure and the residue chromatographed by TLC on silica gel. Elution with CH_2Cl_2 developed one band which afforded pymSSnPh$_3$ (280 mg, 68%) as colorless crystals from hexane/CH_2Cl_2 at 4 °C. ^1H NMR ($CDCl_3$): δ 8.28 (d, J = 4.8 Hz, 2H), 7.76–7.59 (m, 6H), 7.37 (m, 9H), 6.85 (t, J = 4.8 Hz, 1H).

2.4. Reaction of $Ru_3(CO)_{12}$ with pymSSnPh$_3$

A mixture of $Ru_3(CO)_{12}$ (100 mg, 0.016 mmol) and pymSSnPh$_3$ (148 mg, 0.32 mmol) in THF (25 mL) was heated for 5 h at 55–60 °C. The solvent was removed under reduced pressure and the residue separated by TLC on silica gel. Elution with hexane/CH_2Cl_2 (7:3 v/v) developed three bands. The first band gave unreacted $Ru_3(CO)_{12}$ (20 mg). The second band afforded **2** (41 mg, 38%) as

Table 1
Crystallographic data and structure refinement for **1**, **2a**, **2b** and **2c**.

	1	**2a** and **2b**	**2c**·$C_2H_4Cl_2$
Empirical formula	$C_{39}H_{36}S_2Sn_2$	$C_{40}H_{30}O_4RuSn_2$	$C_{40}H_{30}O_4RuSn_2 \cdot C_2H_4Cl_2$
Formula weight (Å)	806.18	913.09	1012.04
Temperature (K)	89(2)	90(2)	90(2)
Crystal system	monoclinic	monoclinic	monoclinic
Space group	$P2_1/c$	$C2/c$	$P2_1/n$
Unit cell dimensions			
a (Å)	12.9769(1)	33.5198(5)	8.4910(1)
b (Å)	14.8376(2)	9.1535(1)	11.8888(1)
c (Å)	18.9431(2)	26.0496(4)	19.8906(2)
β (°)	106.533(1)	117.403(1)	100.821(1)
V (Å^3)	3496.62(7)	7095.78(17)	1972.21(4)
Z	4	8	2
Density (calculated) (Mg/m^3)	1.531	1.709	1.704
Absorption coefficient (mm^{-1})	1.574	1.857	1.810
F(0 0 0)	1608	3568	992
Crystal size (mm)	0.34 × 0.23 × 0.23	0.30 × 0.20 × 0.15	0.32 × 0.20 × 0.10
θ Range for data collection (°)	1.77–36.30	1.37–27.97	2.01–27.91
Reflections collected	42 758	82 690	24 013
Independent reflections	15 714 [R_{int} = 0.0280]	8522 [R_{int} = 0.0308]	4712 [R_{int} = 0.0449]
Data/restraints/parameters	15 714/0/388	8522/0/427	4712/0/232
Goodness-of-fit (GOF) on F^2	1.043	1.037	1.026
Final R indices [$I > 2\sigma(I)$]	R_1 = 0.0289, wR_2 = 0.0586	R_1 = 0.0198, wR_2 = 0.0494	R_1 = 0.0261, wR_2 = 0.0531
R indices (all data)	R_1 = 0.0465, wR_2 = 0.0630	R_1 = 0.0247, wR_2 = 0.0520	R_1 = 0.0376, wR_2 = 0.0569
Largest difference in peak and hole (e Å^{-3})	0.899 and −0.611	0.899 and −0.444	0.610 and −0.409



Scheme 2. Synthesis of $Ph_3SnS(CH_2)_3SSnPh_3$.

colorless crystals after recrystallization from hexane/$C_2H_4Cl_2$ at 4 °C. The third band was too small for complete characterization.

2.5. X-ray crystallography

Single crystals were mounted on fibres and diffraction data collected at low temperatures (see Table 1) on a Bruker APEX II CCD diffractometer using Mo Kα radiation (λ = 0.71073 Å). Data collection, indexing and initial cell refinements were all done using SMART [24] software. Data reduction was done with SAINT [25] software and the SADABS program [26] was used to apply empirical absorption corrections. The structures were solved by direct methods [27] and refined by full matrix least-squares on F^2. All non-hydrogen atoms were refined anisotropically and hydrogen atoms were included using a riding model. Crystal data and refinement details are summarized in Table 1.

3. Results and discussion

3.1. Synthesis and molecular structure of [$Ph_3SnS(CH_2)_3SSnPh_3$] (**1**)

The nucleophilic substitution reaction between triphenyltin chloride and disodium-1,3-propanedithiolate in the presence of $PdCl_2$, followed by usual workup and chromatographic separation, affords [$Ph_3SnS(CH_2)_3SSnPh_3$] (**1**) in 34% yield (Scheme 2). Apparently $PdCl_2$ catalyzes the formation of **1** as attempts to obtain **1** by the same procedure in the absence of $PdCl_2$ failed.

The aliphatic region of the ^1H NMR spectrum of **1** shows a triplet at δ 2.55 (J = 6.8 Hz; integrated to 4H) and a quintet at δ 1.70 (J = 6.8 Hz, integrated to 2H) while the aromatic region contains two multiplets at δ 7.59 and 7.38 with a relative intensity of 10:20 confirming the formulation of **1**. We were able to grow single crystals of **1** and undertook a solid-state investigation. An ORTEP diagram of the molecular structure of **1** is depicted in Fig. 1, and selected bond distances and angles are listed in the caption. The molecule contains two $SSnPPh_3$ units linked by a $CH_2CH_2CH_2$ chain. The molecule possesses a non-crystallographic two-fold axis of symmetry passing through the C(2) carbon. Both tin atoms are four coordinated and adopt a distorted tetrahedral geometry. The average C–Sn–C angle {112.81(5)° for Sn(1) and 111.80(5)° for Sn(2)} is greater than 109.5° (in the idealized polyhedron) while the average C–Sn–S angle {105.83(4)° for Sn(1) and 107.04(4)° for Sn(2)} is smaller at both tin centres, as expected on steric arguments. The two Sn–S bond distances in **1** are equal [av. 2.4084(3) Å] and are towards the shorter end of the range reported in literature for Sn–S bonds [28,29]. Molecules are linked in the crystal by centrosymmetric, reciprocal *ortho*-C–H···S hydrogen-bonds, H···S distances 2.9 Å.

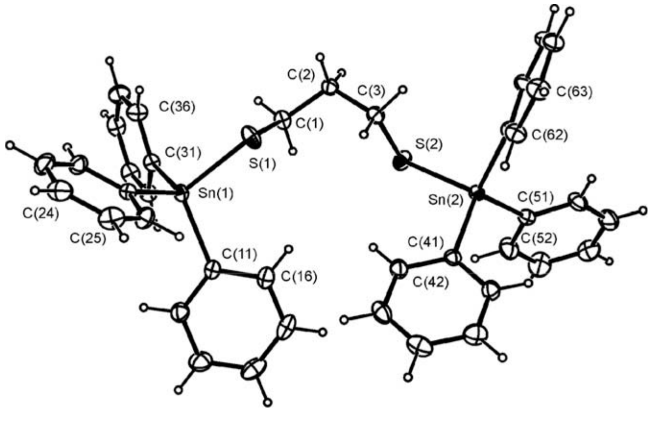


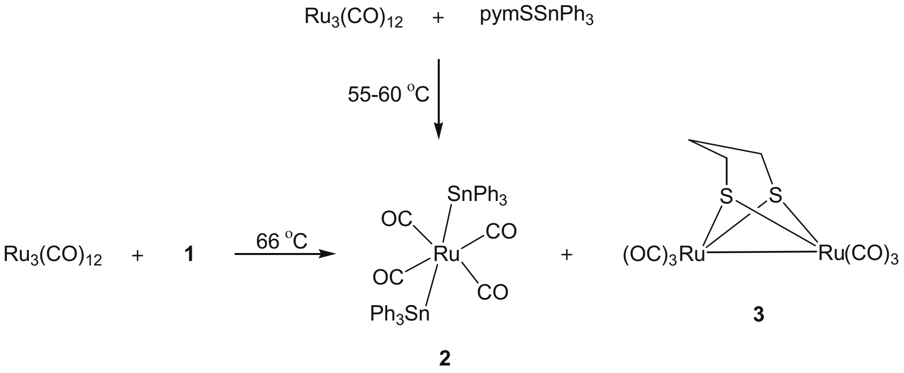
Fig. 1. Molecular structure of [$Ph_3SnS(CH_2)_3SSnPPh_3$] (**1**) showing 35% probability thermal ellipsoids. Selected bond distances (Å) and angles (°): Sn(1)–S(1) 2.4077(3), Sn(2)–S(2) 2.4091(3), C(1)–S(1) 1.8319(14), C(3)–S(2) 1.8360(14), C(1)–C(2) 1.5234(17), C(2)–C(3) 1.5217(18), av. Sn(1)–C 2.1310(13), av. Sn(2)–C 2.1292(13), C(1)–S(1)–Sn(1) 102.99(4), C(3)–S(2)–Sn(2) 100.09(4), av. C–Sn(1)–C 112.81(5), av. C–Sn(1)–S(1) 105.83(4), av. C–Sn(2)–C 111.80(5), av. C–Sn(2)–S(2) 107.04(4), C(2)–C(1)–S(1) 108.80(9), C(2)–C(3)–S(2) 109.96(9), C(3)–C(2)–C(1) 114.90(12).

3.2. Reactions of $Ru_3(CO)_{12}$ with **1** and $pymSSnPh_3$

Treatment of $Ru_3(CO)_{12}$ with **1** at 66 °C furnishes *trans*-[$Ru(CO)_4(SnPh_3)_2$] (**2**) and [$Ru_2(CO)_6(\mu\text{-}\kappa^2\text{-}SCH_2CH_2CH_2S)$] (**3**) in 20 and 11% yields, respectively, whereas the reaction between $Ru_3(CO)_{12}$ and $pymSSnPh_3$ at 50–60 °C predominantly gives **2** in 38% yield (Scheme 3).

The dinuclear compound **3** was previously reported by us obtained from the reaction between $Ru_3(CO)_{12}$ with 1,3-propanedithiol [30]. Compound **2** was first described many years ago, from reactions of Ph_3SnH with $Ru_3(CO)_{12}$ [31], from Ph_3SnCl and $H_2Ru_3(CO)_{12}$ [32] or from Ph_3SnCl and $Ru(CO)_4{}^{2-}$ [33]. Very recently it was also isolated from the reaction between Ph_3SnH and [$Ru_3(CO)_{10}(CNMe)_2$] [8,34]. The iron analogue of **2** was reported by Graham et al. obtained from the reaction of $Na_2Fe(CO)_4\cdot 1.5C_4H_8O_2$ and Ph_3SnCl at −78 °C [35] while the osmium analogue was reported by Collman et al. synthesized from the reaction between $Na_2[Os(CO)_4]$ and Ph_3SnCl at −78 °C and also from $H_2Os(CO)_4$ and Ph_3SnCl at room temperature [36]. The iron and osmium analogues of compound **2** have been structurally characterized in detail, while the structure of **2** itself has been very recently reported, without discussion, in a triclinic form as the benzene solvate [34]. It is interesting to note that the iron complex has a *cis*-configuration whereas the osmium complex possesses *trans*-configuration. The Ph_3Ge analogue of **2**, [$Ru(CO)_4(GePh_3)_2$], is also found in *trans*-configuration [37] whereas [$Ru(CO)_4(GeCl_3)_2$] has been shown to exist in both *cis* and *trans*-configuration [38].

We have crystallised compound **2** from two different solvent systems, hexane/dichloromethane (CH_2Cl_2) or hexane/1,2-dichloroethane ($C_2H_4Cl_{l2}$) and obtained two crystalline modifications: monoclinic ($C2/c$) from CH_2Cl_2 and monoclinic ($P2_1/n$) from $C_2H_4Cl_2$. Both forms were investigated by single crystal X-ray diffraction analyses for comparison with the earlier triclinic version. The $C2/c$ form contains two independent molecules (**2a** and **2b**) in the asymmetric unit, each lying on an inversion centre. The bond lengths and bond angles in both molecules are very similar and an ORTEP diagram of **2a** is shown in Fig. 2, with selected bond distances and angles listed in the caption. The $P2_1/n$ form contains a $C_2H_4Cl_2$ solvent molecule in the unit cell in addition to a molecule of **2** (**2c**), both of which lie on crystallographic inversion centres. In each case, as well as in the previous triclinic version, compound **2**



Scheme 3. Reactions of $Ru_3(CO)_{12}$ with $Ph_3SnS(CH_2)_3SSnPh_3$ and pymSSnPh$_3$.

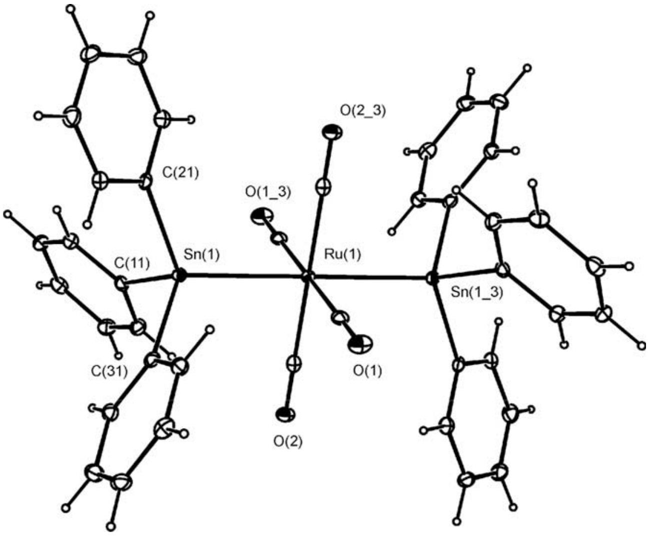


Fig. 2. Molecular structure of *trans*-[Ru(CO)$_4$(SnPh$_3$)$_2$] (**2c**) showing 35% probability thermal ellipsoids. Selected bond distances (Å) and angles (°): Sn(1)–Ru(1) 2.69558(14), Sn(1)#–Ru(2) 2.2.69558(14), Ru(1)–C(11) 1.943(2), Ru(1)–C(11)#1 1.943(2), Ru(1)–C(12) 1.950(2), Ru(1)–C(12)#1 1.950(2), C(11)–O(11) 1.130(3), C(12)–O(12) 1.135(3), Sn(1)–C 2.153(2), Sn(1)–Ru(1)–Sn(1)#1 180.000(5), C(11)–Ru(1)–C(11)#1 180.0(3), C(12)–Ru(1)–C(12)#1 180.0(15), av. C–Ru(1)–C 90.00(9), av. C–Ru(1)–Sn(1) 90.00(6), av. C–Ru(1)–Sn(1)#1 90.00(6).

contains a single ruthenium atom ligated by four CO and two SnPPh$_3$ ligands. The SnPh$_3$ ligands are *trans* to each other with the Sn–Ru–Sn angle being exactly 180° by symmetry constraints (in for the monoclinic forms) compared with 172.1° for the triclinic form **2d**. The Ru–Sn bond distances in **2** {2.6956(2) Å in **2a**; 2.6982 (2) Å in **2b**; 2.6892 (2) Å in **2c**;} are within the range found in literature [21,39], though marginally shorter than found for **2d** (2.709 Å). These values compare with the Sn–Os bond length of 2.712(1) Å in the osmium analogue of **2** [32]. The average Sn–Ru–C and C–Ru–C angles between *cis*-positioned ligands are 90° in both monoclinic forms but the SnRuSn axis is ~1.5° tilted with respect to the Ru(CO)$_4$ plane.

The ^1H NMR spectrum of **2** displays only aromatic resonances while the infrared spectrum shows only one very intense band, consistent with the solid-state geometry.

There is still no clear explanation for the distribution of *cis* and *trans* isomers of $(R_3E)_2M(CO)_4$ as E (Si, Ge, Sn) and M (Fe, Ru, Os) vary. The *trans* isomer must be favoured sterically, whereas the *cis* isomer would be preferred on electronic grounds for purely σ-bonded R$_3$E groups [40]. The surprising observation that it is the example with the smallest metal atom, Fe, that provides the *cis* example amongst the now complete series $(Ph_3Sn)_2M(CO)_4$ may suggest that π-bonding is more important in the Sn–Fe bond than in the Sn–Ru or Sn–Os ones. NMR studies which show that there is facile *cis–trans* isomerism in solution for these complexes suggest that energy differences are small [41].

Acknowledgement

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Appendix A. Supplementary material

CCDC 711279, 711277**,** and 711278 contain the supplementary crystallographic data for **1**, **2** (**2a** and **2b**) and **2** (**2c**), respectively. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.ica.2009.05.011.

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