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# The synthesis and crystal structure of $Sn(C_6H_4CH_2NMe_2\text{-}o)_2$, and its reaction with $Co(\eta^5\text{-}C_5H_5)(\eta^2\text{-}C_2H_4)_2$

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## Abstract

Treatment of $SnCp_2$ or $SnCl_2$ with $o\text{-}LiC_6H_4CH_2NMe_2$ gave the new monomeric stannylene $Sn(C_6H_4CH_2NMe_2\text{-}o)_2$ (1). The reaction between $Co(\eta^5\text{-}C_5H_5)(\eta^2\text{-}C_2H_4)_2$ and the stannylene 1 yielded $Co(\eta^5\text{-}C_5H_5)(\eta^2\text{-}C_2H_4)[Sn(C_6H_4CH_2NMe_2\text{-}o)_2]$ (3). The crystal structure of 1 and 3 has been determined by X-ray diffraction. 1: $C_{18}H_{24}N_2Sn$, $a$ 10.096(1) Å, $b$ 10.056(1) Å, $c$ 17.665(1) Å, $\beta$ 95.37(1)°, space group $P2_1/c$ (No. 14), $Z = 4$, monoclinic; $R = 0.031$ for 3402 observed reflections. 3: $C_{25}H_{33}CoN_2Sn$, $a$ 9.735(2) Å, $b$ 19.917(4) Å, $c$ 12.429(1) Å, $\beta$ 90.36(1)°, space group $P2_1/a$ (No. 14), $Z = 4$, monoclinic; $R = 0.046$ for 4171 observed reflections. The geometry about tin in 1 is distorted $\psi$-trigonal bipyramidal, and that in 3 is distorted trigonal bipyramidal. Both of the $C_6H_4CH_2NMe_2$ ligands in 1 and 3 act as chelates by bonding of their nitrogen atom to tin.

## Introduction

We have a long-standing interest in the synthesis of reactive complexes or of organometallic compounds of novel structure based on the reductive abstraction of five-membered ring ligands by alkali metals from *d* group metallocenes or on the metathetical reactions of *d* group metallocenes with metal alkyls and aryls [1–5]. Jutzi and co-workers recently reported the metathetical reactions of the diphosphene $P_2(C_5Me_5)_2$ with lithium alkyls and amides to give $RPP(C_5Me_5)$ or RPPR species (R = alkyl) together with $Li(C_5Me_5)$ [6]. They also reported that $M(C_5Me_5)_2$ (M = Ge, Sn) can be reduced by sodium or potassium to give elemental $M^0$ and alkali metal cyclopentadienide [7]. We decided to find out whether the 'synthetic concept of degradation of metallocenes under mild conditions' [1] is applicable to main group metallocenes. It was previously found that $o\text{-}LiC_6H_4CH_2NMe_2$ is a useful reagent for the metathetical reactions of nickelocene, chromocene and manganocene [1]. We report below the synthesis of $Sn(C_6H_4CH_2NMe_2\text{-}o)_2$ (1), from *o*-

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$LiC_6H_4CH_2NMe_2$ and $SnCp_2$ or $SnCl_2$, and describe the ligating properties of this new stannylene towards cobalt(I).

## Results and discussion

Reaction of stannocene with butyllithium was previously found to give 1,1′-dilithiostannocene, which on treatment with organosilicon or organophosphorus halides gave substituted stannocenes [8]. We have found that reaction of stannocene with *o*-$LiC_6H_4CH_2NMe_2$ resulted in the exchange of both of the cyclopentadienyl ligands by $C_6H_4CH_2NMe_2$ groups, to give $Sn(C_6H_4CH_2NMe_2$-*o*$)_2$ (**1**) in good yield. The NMR spectrum of the reaction mixture showed that only **1** and lithium cyclopentadienide were present, and no dimethylbenzylamine could be detected (the deep red $Sn(LiC_5H_4)_2$ is isochronous with LiCp [9], and the colour of the reaction mixture suggested that little $Sn(LiC_5H_4)_2$, if any, is present). The stannylene **1** can also be prepared conveniently and in comparable yield, from $SnCl_2$ and *o*-$LiC_6H_4CH_2NMe_2$. The pale yellow crystalline **1** is air- and moisture-sensitive, but is thermally stable up to 120 °C, at which it melts with decomposition. It is monomeric in solution, as shown by cryoscopy in benzene, and in the solid state.

An X-ray diffraction study on **1** was carried out to ascertain whether the nitrogen atoms in **1** were bound to tin, information we could not obtain by spectroscopic methods. The stannylene **1** has a distorted $\psi$-trigonal bipyramidal structure, with two nitrogen atoms in the axial positions, and two carbon atoms and the 'lone pair' in the equatorial positions (Fig. 1). The distorted $\psi$-trigonal bipyramidal is known for tin(II) compounds: the complexes Sn(*O,O*′-$MeCOCHCOPh)_2$ [10], Sn[*O,O*′-$C_5(CO_2Me)_5]_2$ [11], $[Sn(SC_2H_4)_2NBu^t]_2$ [12] and Sn[*P,P*′-$C(PMe_2)_3]_2$ [13] all have this geometry about tin. A noteworthy feature of **1** is that one tin–nitrogen

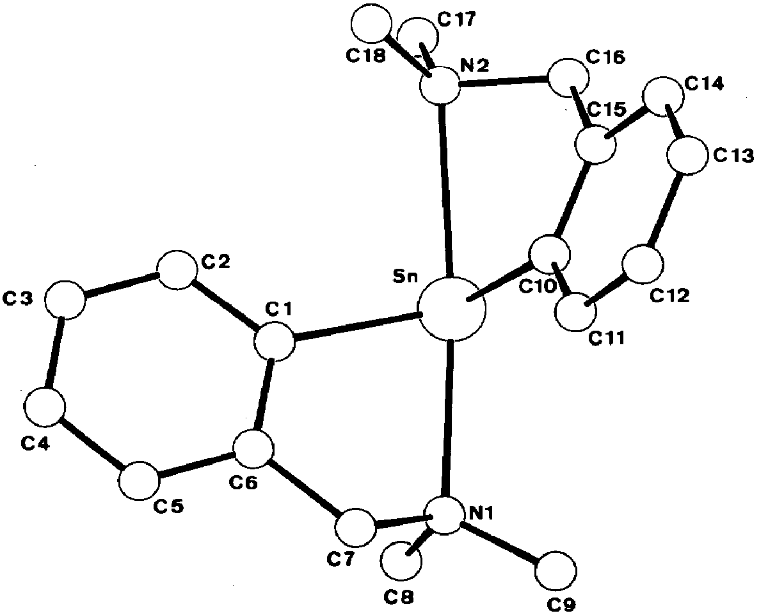

Fig. 1. Structure of $Sn(C_6H_4CH_2NMe_2$-*o*$)_2$ (**1**).

bond is significantly longer than the other: d(SnN(1)) 2.52 Å, d(SnN(2)) 2.66 Å. This is a consequence of the packing: H(2) lies between the two methyl groups on N(2), whereas the equivalent hydrogen atom on the other ring, H(11) lies between one methyl group and the methylene bonded to N(1). There is a close contact H(2)–H(18) (*d* 2.54 Å), which is minimized by movement of the second aryl ligand away resulting in a longer N–Sn bond. There are no intermolecular contacts of less than 3.0 Å. Both tin to nitrogen distances are within the range already found in other tin(II) compounds having neutral nitrogen donor atoms, 2.47–2.75 Å [12,14]. The NSnN bond angle in **1** is 165°.

We could not find any further evidence for a difference between the two amine groups in **1**; the $^{1}$H and $^{13}$C NMR spectra at room temperature show that, in solution, the chelate rings are equivalent. When the solution is cooled to −80°C, the $^{13}$C NMR spectrum showed one pair of inequivalent methyl group resonances but only one methylene resonance. The diastereotopic nature of the *N*-methyl groups probably arises from a freezing out of exchange processes about the chiral tin atom at −80°C. The equivalence of the *N*-methyl groups at higher temperatures probably results from rapid ‘polytopal rearrangement’ by processes (e.g., Berry pseudorotation) described by Muetterties for trigonal bipyramidal structures [15]. In contrast, Van Koten and co-workers reported that pentacoordinate tin(IV) compounds containing $C_6H_4CH_2NMe_2$-*o* and related ligands undergo inversion of configuration by mechanisms in which Sn–N bond forming and breakage are the main steps [16,17]. Recently, Stegmann and coworkers concluded that averaging of proton couplings in the EPR spectra of trigonal bipyramidal tin(IV) radical species containing chelate ligands was due to ‘permutational isomerization’ [18]. Further, an equivalence of the methyl groups observed at high temperatures in the trigonal bipyramidal spirophosphorane $HP(N,O\text{-}NCMe_2CH_2O)_2$, which also contains an unsymmetrical chelate ligand, was ascribed to Berry pseudorotation about the phosphorus atom [19].

There has recently been increasing interest in stannylenes as ligands in transition metal complexes [20–23]. During the course of our work, a report on the structure of $W(CO)_5[Sn(C_6H_4CH_2NMe_2\text{-}o)_2]$ (**2**) and its diphenylphosphine analogue appeared [24]. The synthesis of **2**, which is the first complex containing the stannylene **1** as ligand, was achieved by a halide exchange with $(C_6H_4CH_2NMe_2\text{-}o)^-$ in $W(CO)_5(SnCl_2)$. For the preparation of the new Co–Sn complex $Co(\eta^5\text{-}C_5H_5)(\eta^2\text{-}C_2H_4)[Sn(C_6H_4CH_2NMe_2\text{-}o)_2]$ (**3**) we treated $Co(\eta^5\text{-}C_5H_5)(\eta^2\text{-}C_2H_4)_2$ [25] with the stannylene **1**. This reaction proceeds with the liberation of one mole of ethylene. The $^{1}$H NMR spectrum of **3** at −30°C shows that all four ethylene protons are inequivalent, as are the methylene protons. The peak due to the *N*-methyl groups was very broad. The $^{13}$C NMR spectrum of **3** at −30°C shows two signals for the ethylene carbons and two broad peaks for the *N*-methyl carbons. The peaks due to the *N*-methyl carbons become sharp at −50°C. The broadening of these signals at higher temperature can be ascribed to fluxional processes similar to those in **1**, which will result in the equivalency of the *N*-methyl groups.

In order to obtain more detailed information about the structure of **3**, a crystal structure study was carried out. There was disorder in the crystal involving a 70/30 exchange of the positions of the ethylene and cyclopentadienyl ligands around cobalt. The geometry around tin is distorted trigonal bipyramidal (Fig. 2). The axial NSnN angle, 151°, is almost 15° smaller than that in **1**, but is still larger than the

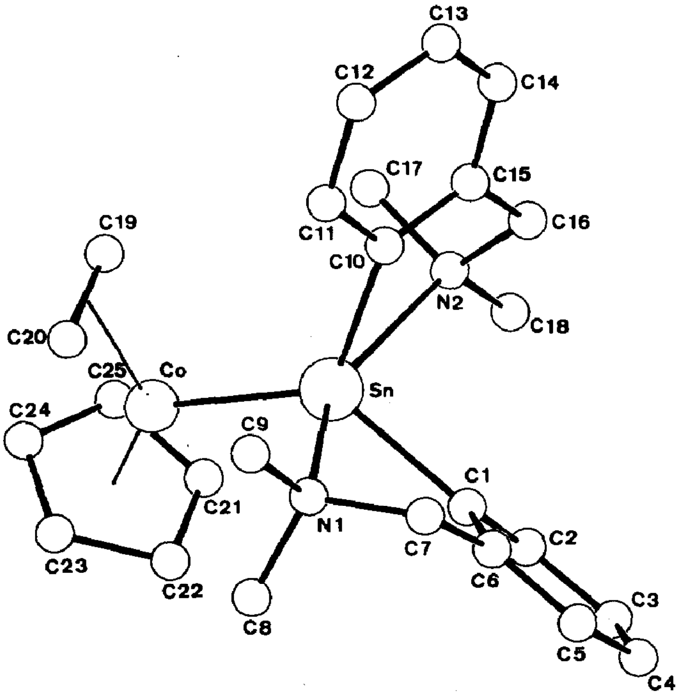

Fig. 2. Structure of $Co(\eta^5\text{-}C_5H_5)(\eta^2\text{-}C_2H_4)[Sn(C_6H_4CH_2NMe_2\text{-}o)_2]$ (3).

NSnN angle in the tungsten–tin compound (141°) [24]. The tin–nitrogen bonds in **3** are of almost equal lengths, 2.59 and 2.61 Å (compared with values for **1** of 2.52 and 2.66 Å), and are only marginally longer than those in **2** (2.56 Å). The tin–cobalt bond (2.44 Å) is relatively short when compared with other bonds between the metal and a terminal stannylene: 2.75 Å in **2**, 2.47–2.50 Å in $Pt\{Sn[N(SiMe_3)_2]_2\}_3$ [22], 2.55 Å in $Rh(\eta^6\text{-}C_6H_5Me)(C_8H_{14})\{Sn[N(SiMe_3)_2]_2Cl\}$ [26], 2.56 Å in $Cr(CO)_5\{Sn[CH(SiMe_3)_2]_2\}$ [20] and 2.65 Å in $Cr(CO)_5(SnBu^t_2py)$ [27].

## Experimental

All manipulations were carried out under argon by standard techniques. All solvents were distilled first from $NaAlEt_4$, and then from Na/K alloy prior to use. Tin(II) chloride was dried [28], and stannocene [29], $o\text{-}LiC_6H_4CH_2NMe_2$ [30] and $Co(\eta^5\text{-}C_5H_5)(\eta^2\text{-}C_2H_4)_2$ [25] were prepared by published methods. $^1H$ NMR spectra were recorded at 200.5 MHz, $^{13}C$ NMR spectra at 75.5 MHz and $^{119}Sn$ NMR spectra at 149.2 MHz (with $SnMe_4$ as reference).

### *Synthesis of $Sn(C_6H_4CH_2NMe_2\text{-}o)_2$ (1)*

*(A) From $SnCl_2$.* $o\text{-}LiC_6H_4CH_2NMe_2$ (3.42 g, 24.3 mmol) was added during 5 min to a solution of $SnCl_2$ (2.12 g, 11.2 mmol) in THF (100 ml) at −5 °C. The reaction mixture was stirred at −5 °C for 30 min, during which the slurry turned to a clear yellow solution. This solution was stirred for a further 2 h at room temperature, then evaporated to dryness under reduced pressure. The residue was extracted with toluene (ca. 100 ml) and the extract was filtered through a sintered

glass frit, then evaporated to dryness under reduced pressure. The residue was extracted with diethyl ether (ca. 200 ml) and the extract was filtered and cooled to −30 °C. The yellow crystals which formed on standing were isolated by decantation, washed twice with a small quantity of ether, and dried under vacuum. A second crop was isolated by concentrating the mother liquor. The crystals were recrystallized from ether to give pale yellow analytically pure crystals of **1** (2.52 g, 6.5 mmol, 58%). Anal. Found: C, 55.87; H, 5.98; N, 7.29; Sn, 30.78, MW 394 (cryoscopically in $C_6H_6$). $C_{18}H_{24}N_2Sn$ calcd.: C, 55.84; H, 6.26; N, 7.24; Sn 30.66%; MW 387. $^1H$ NMR (THF-$d_8$) $\delta$ 2.45 (s, N$Me_2$), 3.76 (s, NC$H_2C_6H_4$), 7–8 (m, $C_6H_4$); $^{13}C$ NMR ($C_6D_5CD_3$) $\delta$ 46.3 ($^2J$(CSn) 33 Hz, N$Me_2$), 68.7 (N$CH_2C_6H_4$), 146.8 ($^2J$(CSn) 33 Hz, *C*(6) and *C*(14)), 170.35 ($^1J$(CSn) 398 Hz ($^{117}$Sn), 415 Hz ($^{119}$Sn), *C*(1) and *C*(9)); $^{119}$Sn NMR (THF-$d_8$) $\delta$ 169 (40 °), 127 (−40 °, $w_{1/2}$ ca. 100 Hz).

*(B) From SnCp$_2$.* *o*-$LiC_6H_4CH_2NMe_2$ (4.53 g, 32.1 mmol) was added during 5 min to stannocene (3.45 g, 13.9 mmol) in THF (150 ml) at 0 °C. The solution was stirred at 0 °C for 10 min and then at room temperature overnight. Work-up was carried out as in (A), with ca. 300 ml toluene for the initial extraction. **1** was again isolated as pale yellow crystals (3.18 g, 8.2 mmol, 59%).

### *Synthesis of Co($\eta^5$-$C_5H_5$)($\eta^2$-$C_2H_4$)[Sn($C_6H_4CH_2NMe_2$-o)$_2$] (3)*

**1** (1.61 g, 4.2 mmol) was added to a solution of Co($\eta^5$-$C_5H_5$)($\eta^2$-$C_2H_4$)$_2$ (0.76 g, 4.2 mmol) in diethyl ether (20 ml) at 0 °C. The reaction mixture was stirred at 0 °C for 30 min and then at room temperature for 1 h. The red precipitate formed was dissolved in warm ether, and the solution was filtered then cooled to −30 °C. The solid formed was isolated by decantantation, washed with a small quantity of ether, and dried in a vacuum to yield glistening red crystals of **3** (0.62 g, 1.15 mmol, 28%). (Note: Solutions of **3** decompose on prolonged standing (more than one day) even at −30 °C). Anal. Found: C, 55.66; H, 6.00; Co, 10.90; N, 5.11; Sn, 22.15. $C_{25}H_{33}CoN_2Sn$ calcd.: C, 55.69; H, 6.17; Co, 10.93; N, 5.20; Sn 22.01%. $^1H$ NMR (THF-$d_8$ at −30 °C) $\delta$ 1.07, 1.67, 1.85, 1.94 (br m, $C_2H_4$), 2.48 (br s, N$Me_2$), 3.25, 3.62 (br d, $^2J_{AB}$ ca. 13 Hz, NC$H_AH_BC_6H_4$), 4.39 (s, $C_5H_5$), 7–8 (m, $C_6H_4$); $^{13}C$ NMR ($C_6D_5CD_3$ at −30 °C) $\delta$ 11.5, 16.9 ($C_2H_4$), 45.7, 47.4 (br, N$Me_2$), 66.3 (N$CH_2C_6H_4$), 77.1 ($C_5H_5$); $^{119}$Sn NMR (THF-$d_8$) $\delta$ 121 (−40 °, $w_{1/2}$ ca. 1100 Hz).

### *Structural determination of 1 and 3*

A single crystal was mounted in a capillary under argon. Unit cell parameters were determined and intensity data collected on an Enraf–Nonius CAD 4 diffractometer using Mo-$K_\alpha$ radiation. Crystallographic data are summarized in Table 3, selected bond distances and angles in Tables 1 (**1**) and 2 (**3**), atomic coordinates and thermal parameters in Tables 4 (**1**) and 5 (**3**).

### *Structure solution and refinement of 1*

The structure of **1** was solved by the heavy atom method (SHELX-86). All hydrogen atoms were located by difference Fourier synthesis and were included in the refinement at fixed positions with isotropic thermal parameters. Refinement was by full matrix least-squares methods.

### *Structure solution and refinement of 3*

The tin atom in **3** was located by Patterson methods and the lighter atoms by Fourier synthesis. Of the hydrogen atoms, only 12 were located, and the remaining

Table 1

Selected bond distances (Å) and angles (°) for 1

| | | | |
|---|---|---|---|
| Sn–N(1) | 2.516(3) | C(10)–Sn–C(1) | 100.5(1) |
| Sn–N(2) | 2.660(3) | C(10)–Sn–N(2) | 72.1(1) |
| Sn–C(1) | 2.216(3) | C(10)–Sn–N(1) | 98.0(1) |
| Sn–C(10) | 2.227(3) | C(1)–Sn–N(2) | 96.7(1) |
| N(1)–C(7) | 1.469(4) | C(1)–Sn–N(1) | 73.6(1) |
| N(2)–C(16) | 1.486(4) | N(2)–Sn–N(1) | 164.9(1) |
| C(1)–C(2) | 1.401(5) | C(7)–N(1)–Sn | 105.9(2) |
| C(1)–C(6) | 1.398(4) | C(16)–N(2)–Sn | 91.7(2) |
| C(2)–C(3) | 1.369(6) | C(6)–C(1)–C(2) | 116.5(3) |
| C(3)–C(4) | 1.388(6) | C(6)–C(1)–Sn | 117.6(2) |
| C(4)–C(5) | 1.389(6) | C(2)–C(1)–Sn | 125.3(2) |
| C(5)–C(6) | 1.398(5) | C(7)–C(6)–C(5) | 119.9(3) |
| C(6)–C(7) | 1.511(5) | C(7)–C(6)–C(1) | 119.2(3) |
| C(10)–C(11) | 1.400(5) | C(5)–C(6)–C(1) | 120.9(3) |
| C(10)–C(15) | 1.409(5) | C(6)–C(7)–N(1) | 111.4(3) |
| C(11)–C(12) | 1.390(5) | C(15)–C(10)–C(11) | 116.4(3) |
| C(12)–C(13) | 1.396(6) | C(15)–C(10)–Sn | 112.7(2) |
| C(13)–C(14) | 1.361(6) | C(11)–C(10)–Sn | 130.8(2) |
| C(14)–C(15) | 1.404(5) | C(16)–C(15)–C(14) | 120.1(3) |
| C(15)–C(16) | 1.507(5) | C(16)–C(15)–C(10) | 118.8(3) |
| | | C(14)–C(15)–C(10) | 121.2(3) |
| | | C(15)–C(16)–N(2) | 110.4(3) |

Table 2

Selected bond distances (Å) and angles (°) for 3

| | | | |
|---|---|---|---|
| Sn–Co | 2.438(1) | C(10)–Sn–C(1) | 106.9(2) |
| Sn–N(1) | 2.593(4) | C(10)–Sn–N(2) | 72.6(1) |
| Sn–N(2) | 2.608(4) | C(10)–Sn–N(1) | 90.4(2) |
| Sn–C(1) | 2.197(5) | C(10)–Sn–Co | 123.9(1) |
| Sn–C(10) | 2.191(5) | C(1)–Sn–N(2) | 89.4(1) |
| Co–C(19) | 2.09(1) | C(1)–Sn–N(1) | 72.6(1) |
| Co–C(20) | 2.036(9) | C(1)–Sn–Co | 129.2(1) |
| N(1)–C(7) | 1.470(7) | N(2)–Sn–N(1) | 150.7(1) |
| N(2)–C(16) | 1.482(7) | N(2)–Sn–Co | 104.5(1) |
| C(1)–C(2) | 1.396(7) | N(1)–Sn–Co | 104.7(1) |
| C(1)–C(6) | 1.403(7) | C(7)–N(1)–Sn | 101.3(3) |
| C(2)–C(3) | 1.384(8) | C(16)–N(2)–Sn | 100.3(3) |
| C(3)–C(4) | 1.36(1) | C(6)–C(1)–C(2) | 117.4(4) |
| C(4)–C(5) | 1.397(9) | C(6)–C(1)–Sn | 116.8(3) |
| C(5)–C(6) | 1.391(8) | C(2)–C(1)–Sn | 125.8(4) |
| C(6)–C(7) | 1.515(7) | C(7)–C(6)–C(5) | 119.1(5) |
| C(10)–C(11) | 1.408(8) | C(7)–C(6)–C(1) | 120.2(4) |
| C(10)–C(15) | 1.404(7) | C(5)–C(6)–C(1) | 120.7(5) |
| C(11)–C(12) | 1.391(9) | C(6)–C(7)–N(1) | 111.2(4) |
| C(12)–C(13) | 1.35(1) | C(15)–C(10)–C(11) | 117.2(5) |
| C(13)–C(14) | 1.39(1) | C(15)–C(10)–Sn | 118.0(4) |
| C(14)–C(15) | 1.376(9) | C(11)–C(10)–Sn | 123.9(3) |
| C(15)–C(16) | 1.491(8) | C(16)–C(15)–C(14) | 120.3(5) |
| C(19)–C(20) | 1.45(1) | C(16)–C(15)–C(10) | 119.1(5) |
| | | C(14)–C(15)–C(10) | 120.6(5) |
| | | C(15)–C(16)–N(2) | 111.7(5) |

Table 3

Crystal data for the X-ray structure analyses [a]

| Formula | $C_{18}H_{24}N_2Sn$ | $C_{25}H_{33}CoN_2Sn$ |
|---|---|---|
| $M_r$ | 387.1 | 539.2 |
| $a$ (Å) | 10.096(1) | 9.735(2) |
| $b$ | 10.056(1) | 19.917(4) |
| $c$ | 17.665(1) | 12.429(1) |
| $\beta$ (°) | 95.37(1) | 90.36(1) |
| $V$ (Å$^3$) | 1785.6 | 2410.0 |
| $Z$ | 4 | 4 |
| Crystal class | monoclinic | monoclinic |
| Space group | $P2_1/c$ | $P2_1/a$ |
| $D_c$ (gcm$^{-3}$) | 1.44 | 1.49 |
| $\mu$(Mo-$K_\alpha$) (cm$^{-1}$) | 14.31 | 17.40 |
| Absorption correction: | – | empirical<br>min: 0.745 max: 1.326 |
| Total no. of reflns. | 4372 ($\pm h$, $+k$, $+l$) | 5846 ($\pm h$, $+k$, $+l$) |
| Independent reflns. | 4020 | 5442 |
| Obs. refl. ($I \geqslant 2\sigma(I)$) | 3402 | 4171 |
| No. of variables | 190 | 237 |
| $R$ | 0.031 | 0.046 |
| $R_w$ | 0.039 | 0.044 |
| Final diff. Fourier (eÅ$^{-3}$) | 1.2 | 0.69 |

[a] Lists of structure factors, anisotropic thermal parameters, H-atom parameters and further information on the data collection have been deposited with the Fachinformationszentrum Energie Physik Mathematik, D-7514 Eggenstein-Leopoldshafen, and may be obtained on request by submission of the deposition number CSD 53030, the name of the authors, and the full literature citation for this paper.

Table 4

Atomic fractional coordinates and equivalent isotropic thermal parameters (Å$^2$) for **1**

$$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij} a_i^\star a_j^\star \bar{a}_i \cdot \bar{a}_j$$

| Atom | $x$ | $y$ | $z$ | $U_{eq}$ |
|---|---|---|---|---|
| Sn | 0.2615(1) | 0.1790(1) | 0.1507(1) | 0.039 |
| N(1) | 0.1232(3) | −0.0261(2) | 0.1646(1) | 0.042 |
| N(2) | 0.3630(2) | 0.4232(3) | 0.1596(2) | 0.043 |
| C(1) | 0.0515(3) | 0.2391(3) | 0.1229(2) | 0.041 |
| C(2) | 0.0093(4) | 0.3550(3) | 0.0838(2) | 0.053 |
| C(3) | −0.1208(4) | 0.3793(4) | 0.0588(2) | 0.062 |
| C(4) | −0.2176(4) | 0.2856(4) | 0.0707(2) | 0.063 |
| C(5) | −0.1798(3) | 0.1685(4) | 0.1084(2) | 0.054 |
| C(6) | −0.0470(3) | 0.1461(3) | 0.1352(2) | 0.043 |
| C(7) | −0.0092(3) | 0.0216(3) | 0.1799(2) | 0.047 |
| C(8) | 0.1190(4) | −0.0853(4) | 0.0889(2) | 0.061 |
| C(9) | 0.1716(4) | −0.1253(4) | 0.2221(2) | 0.061 |
| C(10) | 0.2906(3) | 0.2355(3) | 0.2730(2) | 0.042 |
| C(11) | 0.2284(4) | 0.1896(3) | 0.3356(2) | 0.051 |
| C(12) | 0.2648(4) | 0.2341(4) | 0.4091(2) | 0.060 |
| C(13) | 0.3672(5) | 0.3268(4) | 0.4224(2) | 0.063 |
| C(14) | 0.4293(4) | 0.3741(4) | 0.3628(2) | 0.057 |
| C(15) | 0.3931(3) | 0.3295(3) | 0.2884(2) | 0.045 |
| C(16) | 0.4621(3) | 0.3842(3) | 0.2231(2) | 0.049 |
| C(17) | 0.4308(4) | 0.4546(4) | 0.0909(2) | 0.062 |
| C(18) | 0.2903(4) | 0.5405(4) | 0.1818(2) | 0.060 |

Table 5

Atomic fractional coordinates and equivalent isotropic thermal parameters (Å$^2$) for **3**

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij} a_i^* a_j^* \bar{a}_i \cdot \bar{a}_j$

| Atom | $x$ | $y$ | $z$ | $U_{eq}$ |
|---|---|---|---|---|
| Sn | 0.2592(1) | 0.0706(1) | 0.2451(1) | 0.047 |
| Co | 0.1571(1) | −0.0407(1) | 0.2607(1) | 0.056 |
| N(1) | 0.5209(4) | 0.0488(2) | 0.2306(3) | 0.053 |
| N(2) | 0.0512(4) | 0.1529(2) | 0.2540(3) | 0.062 |
| C(1) | 0.3321(5) | 0.1232(2) | 0.1009(4) | 0.050 |
| C(2) | 0.2514(6) | 0.1417(3) | 0.0126(4) | 0.067 |
| C(3) | 0.3063(7) | 0.1762(3) | −0.0736(5) | 0.080 |
| C(4) | 0.4426(7) | 0.1899(3) | −0.0771(5) | 0.080 |
| C(5) | 0.5283(6) | 0.1697(3) | 0.0074(5) | 0.073 |
| C(6) | 0.4735(5) | 0.1361(2) | 0.0952(4) | 0.057 |
| C(7) | 0.5688(5) | 0.1124(3) | 0.1841(5) | 0.067 |
| C(8) | 0.5302(5) | −0.0073(3) | 0.1533(4) | 0.067 |
| C(9) | 0.6094(5) | 0.0322(3) | 0.3233(5) | 0.079 |
| C(10) | 0.2947(5) | 0.1380(2) | 0.3817(4) | 0.054 |
| C(11) | 0.3751(6) | 0.1211(3) | 0.4723(5) | 0.074 |
| C(12) | 0.3792(7) | 0.1622(4) | 0.5626(5) | 0.099 |
| C(13) | 0.3039(9) | 0.2193(4) | 0.5659(7) | 0.114 |
| C(14) | 0.2198(7) | 0.2362(3) | 0.4789(6) | 0.094 |
| C(15) | 0.2150(5) | 0.1965(3) | 0.3883(5) | 0.067 |
| C(16) | 0.1198(6) | 0.2135(3) | 0.2979(5) | 0.077 |
| C(17) | −0.0385(5) | 0.1226(3) | 0.3374(5) | 0.077 |
| C(18) | −0.0343(6) | 0.1714(3) | 0.1619(6) | 0.088 |
| C(19) | 0.1433(8) | −0.0309(5) | 0.4275(8) | 0.081 |
| C(19a) | −0.009(2) | −0.0293(9) | 0.148(2) | 0.069 |
| C(20) | 0.2675(8) | −0.0650(5) | 0.3948(8) | 0.073 |
| C(20a) | 0.112(2) | −0.0453(9) | 0.094(2) | 0.078 |
| C(21) | 0.0389(8) | −0.0366(3) | 0.1315(6) | 0.093 |
| C(21a) | 0.153(2) | −0.0522(7) | 0.412(1) | 0.059 |
| C(22) | 0.1558(8) | −0.0682(3) | 0.0985(6) | 0.085 |
| C(22a) | 0.270(2) | −0.0843(7) | 0.368(1) | 0.087 |
| C(23) | 0.1592(8) | −0.1260(3) | 0.1695(6) | 0.087 |
| C(23a) | 0.223(2) | −0.1283(7) | 0.289(1) | 0.095 |
| C(24) | 0.0522(8) | −0.1274(3) | 0.2395(6) | 0.090 |
| C(24a) | 0.079(2) | −0.1331(7) | 0.283(1) | 0.074 |
| C(25) | −0.0382(8) | −0.0725(3) | 0.2182(6) | 0.092 |
| C(25a) | 0.039(2) | −0.0816(7) | 0.366(1) | 0.103 |

21 were placed at calculated positions. The cyclopentadienyl and ethylene ligands were disordered, and were refined as rigid bodies with site occupancies of 70/30.

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