Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

(4-Nitrophenylsulfanylmethyl)triphenylstannane and (4-nitrophenylsulfonylmethyl)triphenylstannane: $R_2^2(X)$ rings (X is 10, 18 or 24) and C—H$\cdots\pi$ interactions

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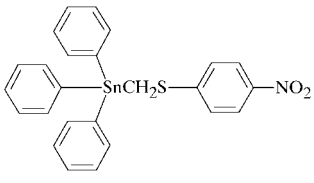
Received 27 May 2002
Accepted 10 June 2002
Online 29 June 2002

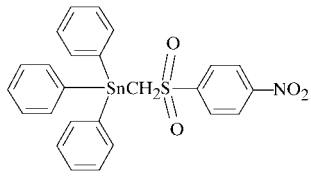
In the crystal structures of (4-nitrophenylsulfanylmethyl)triphenylstannane, $[Sn(C_6H_5)_3(C_7H_6NO_2S)]$, (I), and (4-nitrophenylsulfonylmethyl)triphenylstannane, $[Sn(C_6H_5)_3(C_7H_6NO_4S)]$, (II), the molecules are linked by paired C—H$\cdots$O hydrogen bonds into centrosymmetric dimers which combine to form sheets. In (I), two such dimers form to give $R_2^2(10)$ and $R_2^2(24)$ rings. In (II), similar dimers form, here with $R_2^2(10)$ and $R_2^2(18)$ rings, but with an additional dimer due to the presence of the sulfone group, giving $R_2^2(10)$ rings. In both structures, C—H$\cdots\pi$ interactions lead to a doubling of the width of the sheets.

Comment

The intermolecular non-bonded interactions in a number of nitro, sulfanyl and sulfonyl aromatic derivatives have been investigated in the solid state (Kelly *et al.*, 2002; Cannon *et al.*, 2000, 2001; Glidewell *et al.*, 2001; Wardell *et al.*, 2000*a*,*b*). Continuing our studies, the structures of (4-nitrophenylsulfanylmethyl)triphenylstannane, (I), and (4-nitrophenylsulfonylmethyl)triphenylstannane, (II), have now been investigated and the results are presented here.



(I)



(II)

Both (I) (Fig. 1) and (II) (Fig. 2) crystallize in the triclinic space group $P\overline{1}$ and form sheets *via* similar soft interactions. Molecules of (I) are linked by two different C—H$\cdots$O hydrogen bonds (Table 1) into centrosymmetric dimers, where nitro O atoms act as acceptors. The phenyl atom C5 at (x, y, z) acts as a hydrogen-bond donor to nitro atom O1 at ($3-x$, $2-y$, $-z$), thus forming a dimer characterized by an $R_2^2(10)$ motif with an inversion centre at ($\frac{3}{2}$,1,0). Similarly, atom C36 at (x, y, z) acts as a donor to nitro atom O2 at ($2-x$, $1-y$, $-z$), giving an $R_2^2(24)$ ring with an inversion centre at (1,$\frac{1}{2}$,0). These two dimers combine to form a sheet which propagates along [110] (Fig. 3). In addition, the phenyl rings of neighbouring molecules are weakly linked by C—H$\cdots\pi$ interactions. One of these interactions extends the sheet along the z direction; atom C15 at (x, y, z) donates to the C21–C26 phenyl ring at ($1-x$, $2-y$, $1-z$), with a distance of 3.661 (3) Å between atom C15 and the ring centroid. Such dimers are shown in Fig. 4, and they have the effect of doubling the width of the sheets shown in Fig. 3.

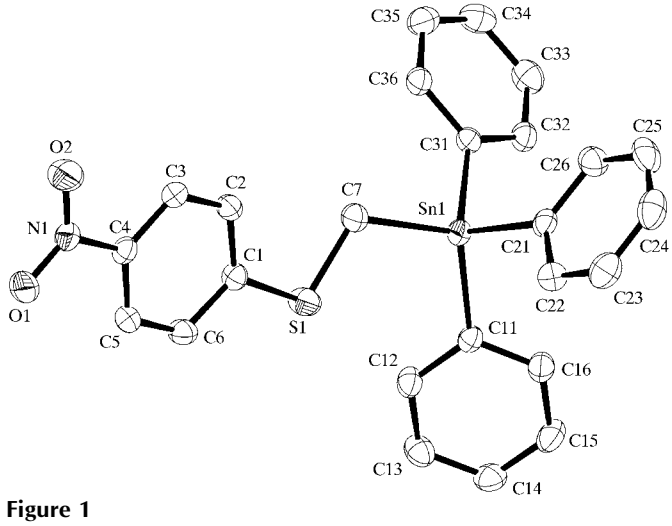


Figure 1
A view of the molecule of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted for clarity.

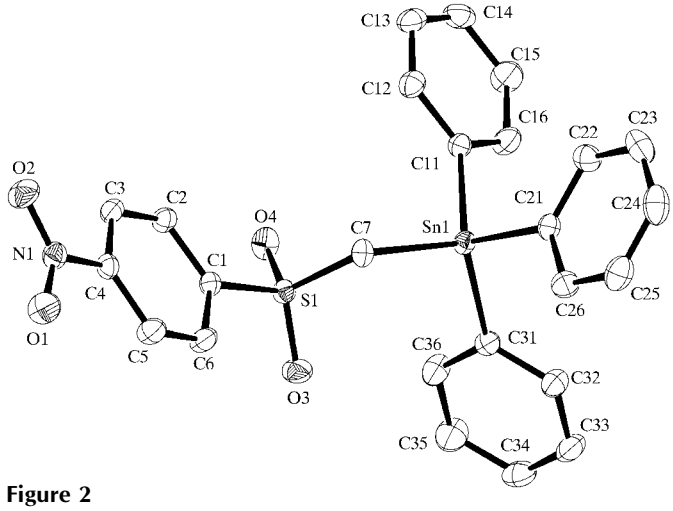


Figure 2
A view of the molecule of (II) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted for clarity.

 DOI: 10.1107/S0108270102010375

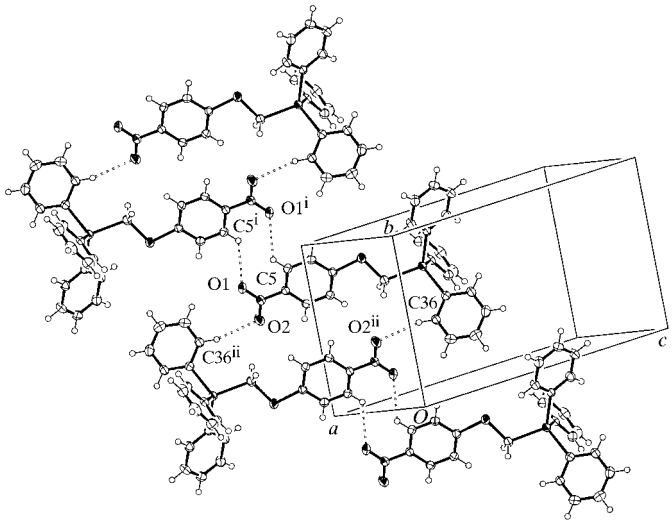


Figure 3
Part of the crystal structure of (I) showing dimers containing $R_2^2(10)$ and $R_2^2(24)$ rings [symmetry codes: (i) $3-x, 2-y, -z$; (ii) $2-x, 1-y, -z$].

Molecules of (II) are also linked *via* intermolecular C—H···O interactions. In this case, the sulfone group leads to one further bond (Table 2). Considering the nitro acceptor interactions first, phenyl atom C5 at (x, y, z) acts as a hydrogen-bond donor to nitro atom O1 at $(-x, 1-y, -z)$, to give an $R_2^2(10)$ motif centred at $(0,\frac{1}{2},0)$. Similarly, atom C7 at (x, y, z) donates to the nitro atom O2 at $(-x, 2-y, -z)$, giving an $R_2^2(18)$ ring centred at (0,1,0), forming a chain along [010] (Fig. 5). Thus, the effect of the sulfone group is to alter the local conformation and thus the C—H···O bonding to the second nitro O atom. In (I), a phenyl H atom from the Ph_3Sn group

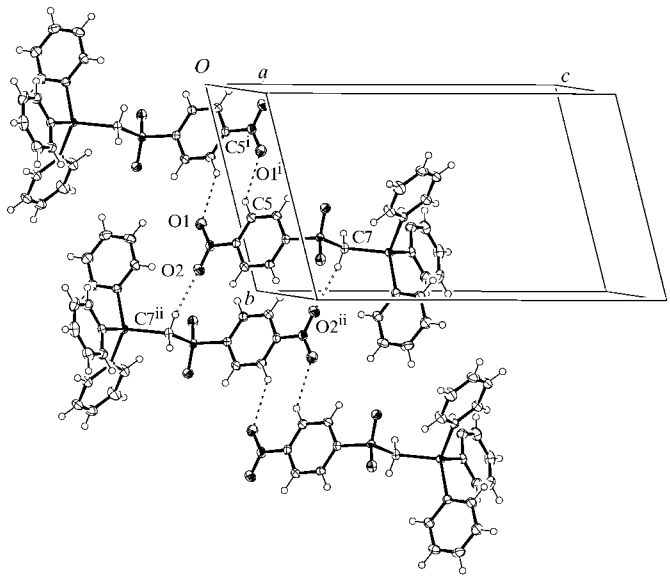


Figure 5
Part of the crystal structure of (II) showing dimers containing $R_2^2(10)$ and $R_2^2(18)$ rings [symmetry codes: (i) $-x, 1-y, -z$; (ii) $-x, 2-y, -z$].

acts as the donor, whereas in (II), the CH_2 group is the donor, effectively contracting the size of the $R_2^2(X)$ ring.

An extra hydrogen bond occurs due to the presence of the sulfone group. Phenyl atom C2 at (x, y, z) acts as a hydrogen-bond donor to sulfone atom O4 at $(1-x, 2-y, -z)$, forming an $R_2^2(10)$ motif with an inversion centre at $(\frac{1}{2},1,0)$. These three dimers thus combine to form a sheet which propagates along [110] (Fig. 6), as in the structure of (I).

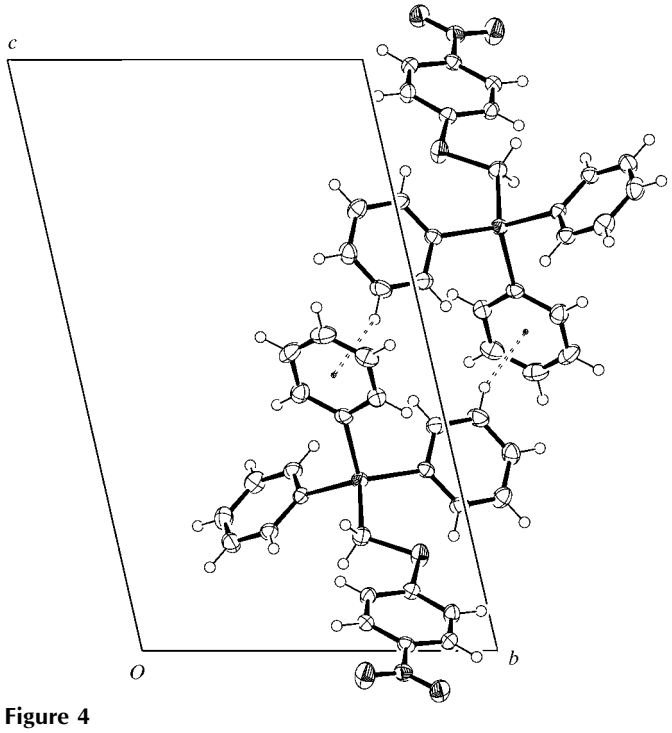


Figure 4
The dimers formed from C—H···π interactions in (I) *via* the symmetry translation $(1-x, 2-y, 1-z)$.

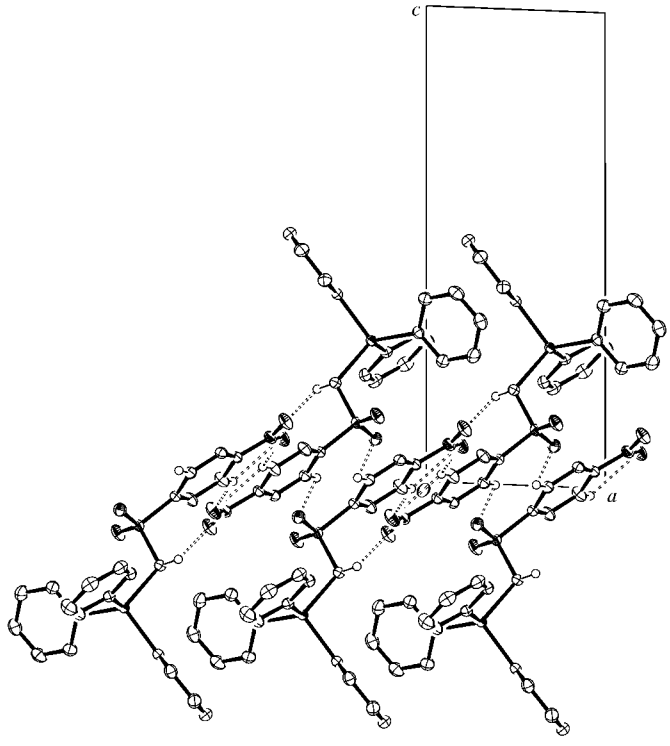


Figure 6
The chains formed by C—H···O interactions in (II).

As with (I), the phenyl groups in (II) form C—H···π interactions with neighbouring molecules within the sheet. In addition, atom C33 at (x, y, z) donates a hydrogen bond to the C21–C26 phenyl ring at ($2-x$, $1-y$, $1-z$), with a distance of 3.361 Å between the H atom and the centroid (Fig. 7), which, as in (I), has the effect of doubling the width of the sheet.

The Sn centre in (I) is four-coordinate; the bond angles, in the range 106.27 (10)–114.64 (8)°, indicate a slightly distorted tetrahedral geometry. The Sn—C bond lengths are in the expected region and fall in a narrow range, 2.138 (2)–2.172 (2) Å. The bond lengths in (II) show a greater range, with those involving the phenyl groups being between 2.1275 (15) and 2.1371 (15) Å, and the Sn—C_{alkyl} length being longer than these, at 2.1815 (14) Å. The bond angles subtended at Sn in (II) range from 101.15 (5) to 111.76 (6)°, again indicative of a slightly distorted tetrahedral geometry. The closest Sn···$O_{sulfone}$ separation is Sn···O4 3.5906 (12) Å, a little within the sum of the van der Waals radii of 3.70 Å.

A number of related triphenyl–Sn and –Ge structures have been reported, along with one related iododiphenyltin compound (CSD database, Release 5.23; Allen & Kennard, 1993). These are $Ph_3GeCH_2SO_2C_6H_5$ (CSD refcode GESYIM; Howie & Wardell, 1997), $Ph_3Sn(CH_2)_2CH(SC_6H_4NO_2$-2)$CH_2$-Cl (ZIKHOQ; Aupers & Wardell, 1995), $Ph_3Sn(CH_2)_2SO_2$-C_6H_4Me-4 [(III), n = 2, YEZVEE; Cox & Wardell, 1994], $Ph_3Sn(CH_2)_4SO_2C_6H_4$Me-4 [(III), n = 4, YEZVII; Cox & Wardell, 1994], $Ph_3Sn(CH_2)_3SO_2C_6H_4$Me-4 [(III), n = 3, ZAVHIN; Howie & Wardell, 1994], $IPh_2Sn(CH_2)_3$-$SO_2C_6H_4$Me-4 (ZAVHOT; Howie & Wardell, 1994), Ph_3Sn-$CHClCH_2SC_6H_3NO_2$-2-Me-4 (PAGHEK; Howie *et al.*, 1992), $Ph_3SnCH(SCN)CH_2SC_6H_4NO_2$-2 (PAGHIO; Howie *et al.*, 1992), (cyclohexyl)$_3SnCH_2SC_6H_4{}^t$Bu-4 (JERMIC; Cox *et al.*, 1990), $Ph_3Ge(CH_2)SO_2C_6H_5$ (NOJXEP; Wardell & Cox, 1996), $Ph_3SnC(SMe)$═CHC_6H_5 (GODLOA and GODLUG; Bruhn *et al.*, 1999), $Ph_3SnC(SCH_2Ph)CHCHC(SCH_2Ph)SnPh_3$ (POMXUK; Block *et al.*, 1994) and $Ph_3SnCH_2CH_2SC_6H_4$Me-4 (ZUWQIR; Cox *et al.*, 1995).

Of these, the first four (GESYIM, ZIKHOQ, YEZVEE and YEZVII) have C—H···O bonds which can be considered in relation to (I) and (II). The latter four compounds (GODLOA, GODLUG, POMXUK and ZUWQIR) display C—H···S interactions, while the rest do not form hydrogen bonds, as detected by *PLATON* (Spek, 2002).

$Ph_3GeCH_2SO_2C_6H_5$ (GESYIM), a Ge analogue to (II), forms hydrogen bonds *via* the CH_2 group as donor to a sulfone O atom, thus forming simple $C(4)$ chains along [001]. No rings are formed. $Ph_3Sn(CH_2)_2CH(SC_6H_4NO_2$-2)$CH_2Cl$ (ZIKHOQ), in which the nitro group is *ortho* to S, again has CH_2 as the donor to one nitro O atom as acceptor, forming $R_2^2(16)$ dimers. The other H atom of the CH_2 group donates to the other nitro O atom, forming a chain of dimers along [001]. $Ph_3Sn(CH_2)_2SO_2C_6H_4$Me-4 (YEZVEE) again donates a hydrogen bond *via* the CH_2 group adjacent to S to a sulfone O atom; in addition, a phenyl H atom acts as a donor to the same O atom, forming $R_3^2(13)$ groups which link to give $C(8)$ chains along [100]. In the related compound $Ph_3Sn(CH_2)_4$-$SO_2C_6H_4$Me-4 (YEZVII), similar hydrogen bonding occurs, although here *via* the third CH_2 group from S, to give an $R_3^2(16)$ motif, linking to give $C(12)$ chains along [010]. Both motifs are enlarged by the extra CH_2 groups in the latter compound.

The products of the reactions of ω-sulfanylalkylstannanes, $R_3Sn(CH_2)_nSR'$, with oxidants depend greatly on n and on the oxidant. For example, (II) was obtained by H_2O_2 oxidation of (I), whereas the reaction of the related compound $Ph_3Sn(CH_2)_nSC_6H_4$Me-4 [(IV), n = 1] with $NaIO_4$ led to cleavage of the molecule with formation of Ph_3SnCH_2I and 4-$MeC_6H_4SO_3H$ (Taylor & Wardell, 1976; see also Peterson, 1971). The use of 3-$ClC_6H_4CO_3H$ with (IV), with n = 1, also resulted in cleavage (Wardell, unpublished observation). In contrast, oxidations of (IV) with n = 3 or 4 proceeded readily to the sulfones (III) with n = 3 or 4, or the corresponding sulfoxides, depending on the molar ratios of the reagents (Wardell & Wigzell, 1983). Particularly sensitive to oxidants is (IV) with n = 2. The reaction of (IV) with n = 2 with either $NaIO_4$ or 3-$ClC_6H_4CO_3H$ led to loss of ethylene (Wardell & Wigzell, 1983). Compound (IV) with n = 2 was, however, obtained by the addition of Ph_3SnH to H_2C═CH-$SO_2C_6H_4$Me-4 (Wardell & Wigzell, 1983). The contrast between the Ge and Sn compounds is clear from the reactions of Ph_3MCH_2SR' (M is Ge or Sn) with 3-$ClC_6H_4CO_3H$ or Br_2. The Br_2–R_3SnCH_2SR' reactions invariably result in Sn—C bond cleavage, while the Ge compounds can be oxidized to $Ph_3GeCH_2SO_mR$ (m = 1 or 2) by either Br_2 in MeOH or 3-$ClC_6H_4CO_3H$ (Taylor & Wardell, 1976; Wardell & Cox, 1996).

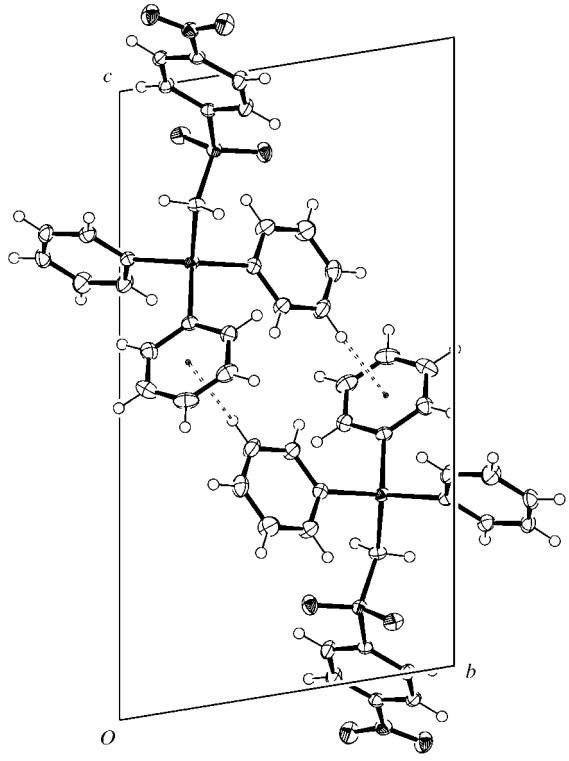


Figure 7
The dimers formed by C—H···π interactions in (II) *via* the symmetry translation ($1-x$, $2-y$, $1-z$).

Experimental

Compound (I) was prepared from Ph_3SnCH_2I, $HSC_6H_4NO_2$ and NaOEt (2 mmol scale) in EtOH (20 ml). After refluxing for 2 h, the mixture was cooled, all volatiles removed and the residue recrystallized from EtOH (m.p. 416–418 K). Analysis found: C 58.4, H 5.5, S 6.3, N 3.1%; calculated for $C_{24}H_{21}NO_2SSn$: C 58.0, H 4.1, S 6.2, N 2.7%. Spectroscopic analysis, ^{1}H NMR (250 Hz, $CDCl_3$, δ, p.p.m.): 2.88 [*s*, 2H, $J(^{119,117}Sn-^1H)$ = 48.8 Hz, CH_2], 7.41 (*m*, 11H, *m*-H + *p*-H of Ph_3Sn + 2H from C_6H_4), 7.60 [*m*, 6H, $J(^{119,117}Sn-^1H) \sim 57$ Hz, *o*-H of Ph_3Sn], 8.10 (*d*, 2H, *J* = 8.8 Hz, C_6H_4); ^{13}C NMR (63.3 Hz, $CDCl_3$, δ, p.p.m.): 5.2 (CH_2), 123.8 (C_3), 124.6 (C_2), 128.9 (*m*-C of Ph_3Sn), 129.7 (*p*-C of Ph_3Sn), 136.7 (*i*-C of Ph_3Sn), 137.0 (*o*-C of Ph_3Sn), 144.0 (C_1), 152.7 (C_4); ^{119}Sn NMR (93.3 Hz, CD_2Cl_2, δ, p.p.m.): −118; IR (KBr, cm^{-1}): 1578 and 1339 (NO_2); Raman (cm^{-1}): 1587, 1331.

Compound (II) was obtained by H_2O_2 oxidation (30% solution in water) of (I) in a mixed H_2O–CH_2Cl_2 medium. After stirring the reaction mixture overnight at room temperature, all volatiles were removed and the residue was recrystallized from EtOH (m.p. 472–475 K). Spectroscopic analysis, ^{1}H NMR (250 Hz, $CDCl_3$, δ, p.p.m.): 3.44 [*s*, 2H, $J(^{119,117}Sn-^1H)$ = 41.2 Hz, CH_2], 7.43 (*m*, 9H, *p*-H + *m*-H of Ph_3Sn), 7.63 [*m*, 6H, $J(^{119,117}Sn-^1H) \sim 60$ Hz, *o*-H of Ph_3Sn], 8.06 (*d*, 2H, *J* = 8.6 Hz, C_6H_4), 8.28 (*m*, 2H, *J* = 8.6 Hz, C_6H_4); ^{13}C NMR (63.3 Hz, $CDCl_3$, δ, p.p.m.): 44.5 (CH_2), 124.4 (C_3), 128.2 (C_2), 129.0 (*m*-C of Ph_3Sn), 129.9 (*p*-C of Ph_3Sn), 135.8 (*i*-C of Ph_3Sn), 137.0 (*o*-C of Ph_3Sn), C_4 and C_1 not observed; IR (Nujol mull, cm^{-1}): 1527 and 1304 (NO_2), 1377 and 1145 (SO_2).

Compound (I)

Crystal data

$[Sn(C_6H_5)_3(C_7H_6NO_2S)]$	Z = 2
M_r = 518.18	D_x = 1.555 Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
a = 6.6442 (1) Å	Cell parameters from 12 524 reflections
b = 10.3070 (2) Å	θ = 2.9–27.5°
c = 17.2597 (4) Å	μ = 1.27 mm^{-1}
α = 100.3385 (8)°	T = 150 (2) K
β = 98.6403 (9)°	Needle, yellow
γ = 103.6969 (16)°	0.40 × 0.10 × 0.03 mm
V = 1106.51 (4) Å^3	

Data collection

Nonius KappaCCD area-detector diffractometer	5030 independent reflections
φ and ω scans	4460 reflections with $I > 2\sigma(I)$
Absorption correction: empirical (*SORTAV*; Blessing, 1995, 1997)	R_{int} = 0.052
T_{min} = 0.817, T_{max} = 0.992	θ_{max} = 27.5°
16 551 measured reflections	$h = -8 \rightarrow 8$
	$k = -13 \rightarrow 13$
	$l = -22 \rightarrow 22$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.032	$w = 1/[\sigma^2(F_o^2) + (0.0434P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.079	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.06	$\Delta\rho_{max}$ = 0.82 e Å^{-3}
5030 reflections	$\Delta\rho_{min}$ = −1.39 e Å^{-3}
271 parameters	

Table 1
Hydrogen-bonding geometry (Å, °) for (I).

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···O1^{i}	0.95	2.56	3.151 (3)	121
C36—H36···O2^{ii}	0.95	2.57	3.439 (3)	152

Symmetry codes: (i) $3-x, 2-y, -z$; (ii) $2-x, 1-y, -z$.

Compound (II)

Crystal data

$[Sn(C_6H_5)_3(C_7H_6NO_4S)]$	Z = 2
M_r = 550.18	D_x = 1.606 Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
a = 6.8715 (4) Å	Cell parameters from 10 598 reflections
b = 9.9680 (5) Å	θ = 2.3–28.9°
c = 17.6403 (10) Å	μ = 1.25 mm^{-1}
α = 81.0264 (19)°	T = 120 (2) K
β = 89.4798 (19)°	Block, colourless
γ = 72.5146 (17)°	0.5 × 0.3 × 0.3 mm
V = 1137.42 (11) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	5261 independent reflections
φ and ω scans	5061 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)	R_{int} = 0.011
T_{min} = 0.674, T_{max} = 0.688	θ_{max} = 28.9°
10 598 measured reflections	$h = -9 \rightarrow 8$
	$k = -13 \rightarrow 13$
	$l = -23 \rightarrow 23$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0246P)^2 + 0.5422P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.018	$(\Delta/\sigma)_{max}$ = 0.002
$wR(F^2)$ = 0.046	$\Delta\rho_{max}$ = 0.38 e Å^{-3}
S = 1.10	$\Delta\rho_{min}$ = −0.61 e Å^{-3}
5261 reflections	
289 parameters	
H-atom parameters constrained	

Table 2
Hydrogen-bonding geometry (Å, °) for (II).

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O4^{i}	0.95	2.58	3.2753 (19)	131
C5—H5···O1^{ii}	0.95	2.48	3.204 (2)	133
C7—H7*B*···O2^{iii}	0.99	2.36	3.3163 (19)	161

Symmetry codes: (i) $1-x, 2-y, -z$; (ii) $-x, 1-y, -z$; (iii) $-x, 2-y, -z$.

Structure (I) was solved using Patterson methods (*SHELXS*86; Sheldrick, 1990) in *P*1, and then the coordinates were converted to $P\overline{1}$. All H atoms were placed in geometrically calculated positions, with C—H distances of 0.95 (phenyl) and 0.99 Å (CH_2), and refined using a riding model.

For compound (I), data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS*86. For compound (II), data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrich, 1990). For both compounds, program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEX* in *OSCAIL* (McArdle, 1994, 2000) and *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

The authors thank the EPSRC X-ray Crystallographic Service, University of Southampton, England, for the collection of data, and acknowledge the use of the EPSRC Chemical Database Service at Daresbury (Fletcher *et al.*, 1996). JLW and SMSVW thank CNPq for support.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1559). Services for accessing these data are described at the back of the journal.

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