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(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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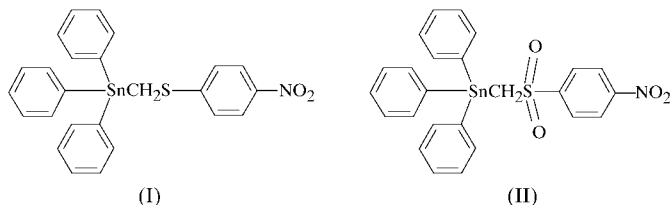
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In the crystal structures of (4-nitrophenylsulfanylmethyl)triphenylstannane, $[\text{Sn}(\text{C}_6\text{H}_5)_3(\text{C}_7\text{H}_6\text{NO}_2\text{S})]$, (I), and (4-nitrophenylsulfonylmethyl)triphenylstannane, $[\text{Sn}(\text{C}_6\text{H}_5)_3(\text{C}_7\text{H}_6\text{NO}_4\text{S})]$, (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.



Both (I) (Fig. 1) and (II) (Fig. 2) crystallize in the triclinic space group $P\bar{1}$ 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.

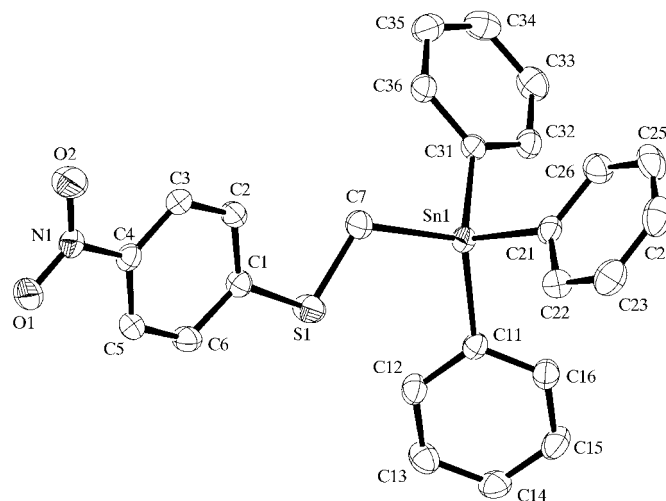


Figure 1

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.

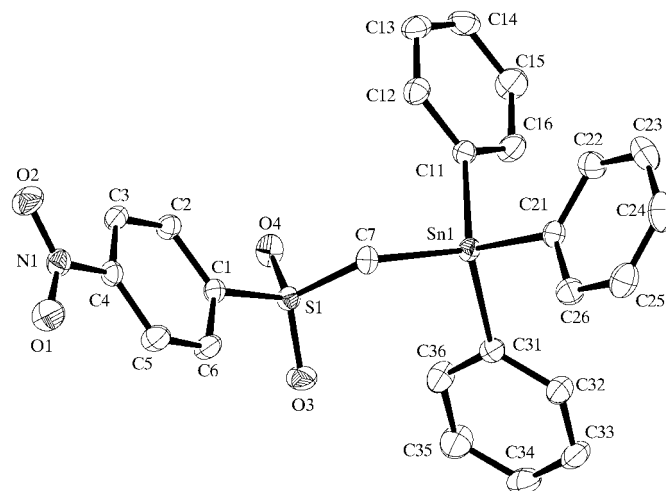


Figure 2

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.

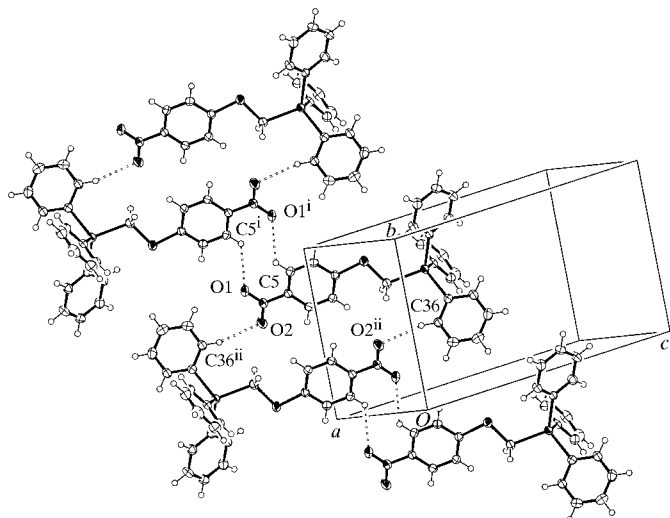


Figure 3
Part of the crystal structure of (I) showing dimers containing $R_2^2(10)$ and $R_2^2(24)$ rings [symmetry codes: (i) $3 - x, 2 - y, -z$; (ii) $2 - x, 1 - y, -z$].

Molecules of (II) are also linked *via* intermolecular C—H \cdots 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 \cdots O bonding to the second nitro O atom. In (I), a phenyl H atom from the Ph_3Sn group

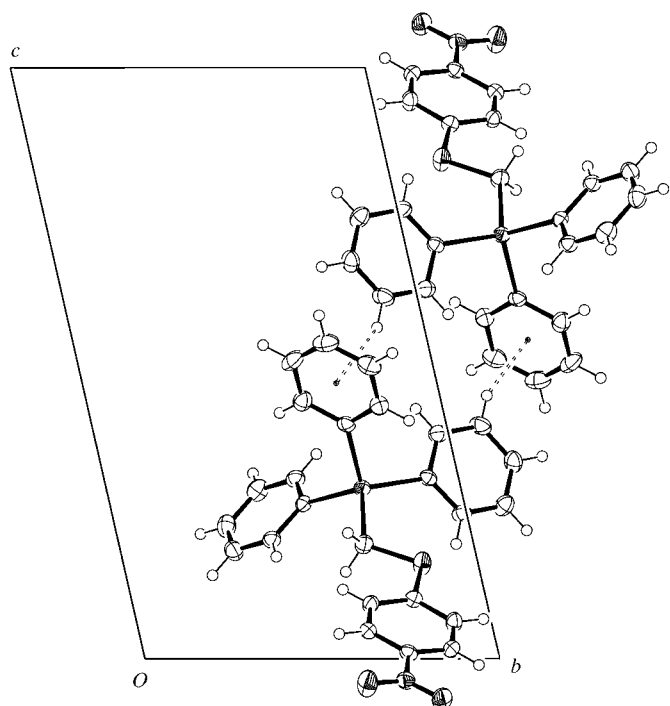


Figure 4
The dimers formed from C—H \cdots π interactions in (I) *via* the symmetry translation $(1 - x, 2 - y, 1 - z)$.

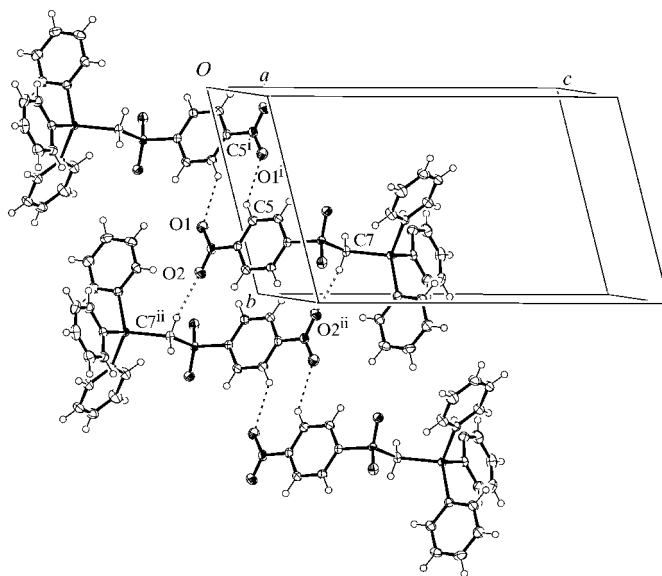


Figure 5
Part of the crystal structure of (II) showing dimers containing $R_2^2(10)$ and $R_2^2(18)$ rings [symmetry codes: (i) $-x, 1 - y, -z$; (ii) $-x, 2 - y, -z$].

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).

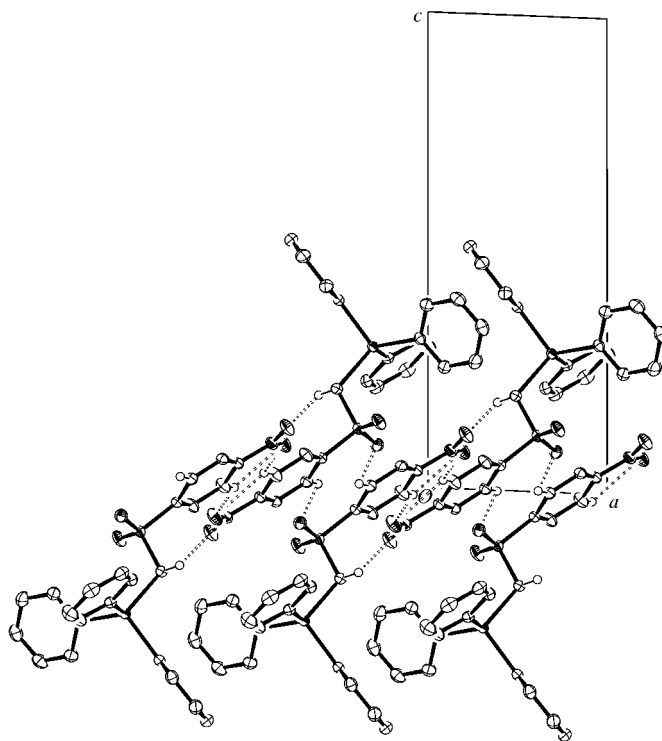


Figure 6
The chains formed by C—H \cdots O interactions in (II).

As with (I), the phenyl groups in (II) form C—H $\cdots\pi$ 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 \cdots O_{sulfone} separation is Sn \cdots 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 Ph₃GeCH₂SO₂C₆H₅ (CSD refcode GESYIM; Howie & Wardell, 1997), Ph₃Sn(CH₂)₂CH(SC₆H₄NO₂-2)CH₂Cl (ZIKHOQ; Aupers & Wardell, 1995), Ph₃Sn(CH₂)₂SO₂-C₆H₄Me-4 [(III), $n = 2$, YEZVEE; Cox & Wardell, 1994], Ph₃Sn(CH₂)₄SO₂C₆H₄Me-4 [(III), $n = 4$, YEZVII; Cox & Wardell, 1994], Ph₃Sn(CH₂)₃SO₂C₆H₄Me-4 [(III), $n = 3$, ZAVHIN; Howie & Wardell, 1994], IPh₂Sn(CH₂)₃-SO₂C₆H₄Me-4 (ZAVHOT; Howie & Wardell, 1994), Ph₃Sn-

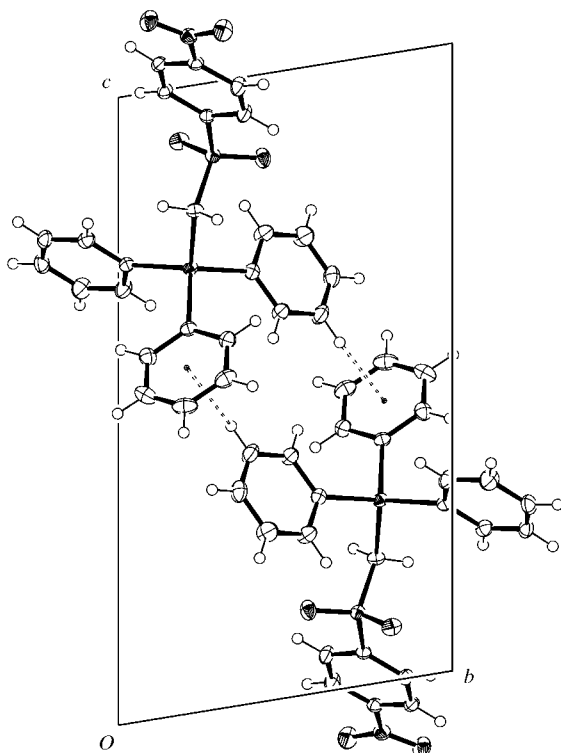


Figure 7
The dimers formed by C—H $\cdots\pi$ interactions in (II) *via* the symmetry translation ($1 - x, 2 - y, 1 - z$).

CHClCH₂SC₆H₃NO₂-2-Me-4 (PAGHEK; Howie *et al.*, 1992), Ph₃SnCH(SCN)CH₂SC₆H₄NO₂-2 (PAGHIO; Howie *et al.*, 1992), (cyclohexyl)₃SnCH₂SC₆H₄Me-4 (JERMIC; Cox *et al.*, 1990), Ph₃Ge(CH₂)SO₂C₆H₅ (NOJXEP; Wardell & Cox, 1996), Ph₃SnC(SMe)=CHC₆H₅ (GODLOA and GODLUG; Bruhn *et al.*, 1999), Ph₃SnC(SCH₂Ph)CHCHC(SCH₂Ph)SnPh₃ (POMXUK; Block *et al.*, 1994) and Ph₃SnCH₂CH₂SC₆H₄Me-4 (ZUWQIR; Cox *et al.*, 1995).

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

Ph₃GeCH₂SO₂C₆H₅ (GESYIM), a Ge analogue to (II), forms hydrogen bonds *via* the CH₂ group as donor to a sulfone O atom, thus forming simple C(4) chains along [001]. No rings are formed. Ph₃Sn(CH₂)₂CH(SC₆H₄NO₂-2)CH₂Cl (ZIKHOQ), in which the nitro group is *ortho* to S, again has CH₂ as the donor to one nitro O atom as acceptor, forming R₂²(16) dimers. The other H atom of the CH₂ group donates to the other nitro O atom, forming a chain of dimers along [001]. Ph₃Sn(CH₂)₂SO₂C₆H₄Me-4 (YEZVEE) again donates a hydrogen bond *via* the CH₂ 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 R₂²(13) groups which link to give C(8) chains along [100]. In the related compound Ph₃Sn(CH₂)₄-SO₂C₆H₄Me-4 (YEZVII), similar hydrogen bonding occurs, although here *via* the third CH₂ group from S, to give an R₂²(16) motif, linking to give C(12) chains along [010]. Both motifs are enlarged by the extra CH₂ groups in the latter compound.

The products of the reactions of ω -sulfanylalkylstannanes, R₃Sn(CH₂)_nSR', with oxidants depend greatly on n and on the oxidant. For example, (II) was obtained by H₂O₂ oxidation of (I), whereas the reaction of the related compound Ph₃Sn(CH₂)_nSC₆H₄Me-4 [(IV), $n = 1$] with NaIO₄ led to cleavage of the molecule with formation of Ph₃SnCH₂I and 4-MeC₆H₄SO₃H (Taylor & Wardell, 1976; see also Peterson, 1971). The use of 3-ClC₆H₄CO₃H 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 NaIO₄ or 3-ClC₆H₄CO₃H led to loss of ethylene (Wardell & Wigzell, 1983). Compound (IV) with $n = 2$ was, however, obtained by the addition of Ph₃SnH to H₂C=CH-SO₂C₆H₄Me-4 (Wardell & Wigzell, 1983). The contrast between the Ge and Sn compounds is clear from the reactions of Ph₃MCH₂SR' (M is Ge or Sn) with 3-ClC₆H₄CO₃H or Br₂. The Br₂-R₃SnCH₂SR' reactions invariably result in Sn—C bond cleavage, while the Ge compounds can be oxidized to Ph₃GeCH₂SO_mR ($m = 1$ or 2) by either Br₂ in MeOH or 3-ClC₆H₄CO₃H (Taylor & Wardell, 1976; Wardell & Cox, 1996).

Experimental

Compound (I) was prepared from Ph₃SnCH₂I, HSC₆H₄NO₂ 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 C₂₄H₂₁NO₂SSn: C 58.0, H 4.1, S 6.2, N 2.7%. Spectroscopic analysis, ¹H NMR (250 Hz, CDCl₃, δ, p.p.m.): 2.88 [s, 2H, J(^{119,117}Sn–¹H) = 48.8 Hz, CH₂], 7.41 (m, 11H, *m*-H + *p*-H of Ph₃Sn + 2H from C₆H₄), 7.60 [m, 6H, J(^{119,117}Sn–¹H) ~ 57 Hz, *o*-H of Ph₃Sn], 8.10 (d, 2H, J = 8.8 Hz, C₆H₄); ¹³C NMR (63.3 Hz, CDCl₃, δ, p.p.m.): 5.2 (CH₂), 123.8 (C₃), 124.6 (C₂), 128.9 (*m*-C of Ph₃Sn), 129.7 (*p*-C of Ph₃Sn), 136.7 (*i*-C of Ph₃Sn), 137.0 (*o*-C of Ph₃Sn), 144.0 (C₁), 152.7 (C₄); ¹¹⁹Sn NMR (93.3 Hz, CD₂Cl₂, δ, p.p.m.): –118; IR (KBr, cm^{–1}): 1578 and 1339 (NO₂); Raman (cm^{–1}): 1587, 1331.

Compound (II) was obtained by H₂O₂ oxidation (30% solution in water) of (I) in a mixed H₂O–CH₂Cl₂ 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, ¹H NMR (250 Hz, CDCl₃, δ, p.p.m.): 3.44 [s, 2H, J(^{119,117}Sn–¹H) = 41.2 Hz, CH₂], 7.43 (m, 9H, *p*-H + *m*-H of Ph₃Sn), 7.63 [m, 6H, J(^{119,117}Sn–¹H) ~ 60 Hz, *o*-H of Ph₃Sn], 8.06 (d, 2H, J = 8.6 Hz, C₆H₄), 8.28 (m, 2H, J = 8.6 Hz, C₆H₄); ¹³C NMR (63.3 Hz, CDCl₃, δ, p.p.m.): 44.5 (CH₂), 124.4 (C₃), 128.2 (C₂), 129.0 (*m*-C of Ph₃Sn), 129.9 (*p*-C of Ph₃Sn), 135.8 (*i*-C of Ph₃Sn), 137.0 (*o*-C of Ph₃Sn), C₄ and C₁ not observed; IR (Nujol mull, cm^{–1}): 1527 and 1304 (NO₂), 1377 and 1145 (SO₂).

Compound (I)

Crystal data

[Sn(C₆H₅)₃(C₇H₆NO₂S)]
M_r = 518.18
 Triclinic, *P* $\bar{1}$
a = 6.6442 (1) Å
b = 10.3070 (2) Å
c = 17.2597 (4) Å
 α = 100.3385 (8)°
 β = 98.6403 (9)°
 γ = 103.6969 (16)°
V = 1106.51 (4) Å³
Z = 2
D_x = 1.555 Mg m^{–3}
 Mo *K*α radiation
 Cell parameters from 12 524 reflections
 θ = 2.9–27.5°
 μ = 1.27 mm^{–1}
T = 150 (2) K
 Needle, yellow
 0.40 × 0.10 × 0.03 mm

Data collection

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

Refinement

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

Table 1

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

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
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(C₆H₅)₃(C₇H₆NO₄S)]
M_r = 550.18
 Triclinic, *P* $\bar{1}$
a = 6.8715 (4) Å
b = 9.9680 (5) Å
c = 17.6403 (10) Å
 α = 81.0264 (19)°
 β = 89.4798 (19)°
 γ = 72.5146 (17)°
V = 1137.42 (11) Å³
Z = 2
D_x = 1.606 Mg m^{–3}
 Mo *K*α radiation
 Cell parameters from 10 598 reflections
 θ = 2.3–28.9°
 μ = 1.25 mm^{–1}
T = 120 (2) K
 Block, colourless
 0.5 × 0.3 × 0.3 mm

Data collection

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

Refinement

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

Table 2

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

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
C2–H2···O4 ⁱ	0.95	2.58	3.2753 (19)	131
C5–H5···O1 ⁱⁱⁱ	0.95	2.48	3.204 (2)	133
C7–H7B···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 (SHELXS86; Sheldrick, 1990) in *P* $\bar{1}$, and then the coordinates were converted to *P* $\bar{1}$. All H atoms were placed in geometrically calculated positions, with C–H distances of 0.95 (phenyl) and 0.99 Å (CH₂), 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: SHELXS86. For compound (II), data collection: SMART (Bruker, 1999); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1990). For both compounds, program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEX in OSCAIL (McArdle, 1994, 2000) and ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

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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.

References

- Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 1, 31–37.
- Aupers, J. H. & Wardell, J. L. (1995). *Acta Cryst.* **C51**, 2559–2561.
- Blessing, R. H. (1995). *Acta Cryst.* **A51**, 33–38.
- Blessing, R. H. (1997). *J. Appl. Cryst.* **30**, 421–426.
- Block, E., Guo, C.-X., Thiruvazhi, M. & Toscano, P. J. (1994). *J. Am. Chem. Soc.* **116**, 9403–9404.
- Bruhn, C., Steinborn, D., Lébl, T. & Holeček, J. (1999). *Acta Cryst.* **C55**, 363–365.
- Bruker (1999). *SMART* and *SAINT*. Versions 6.02a. Bruker AXS Inc., Madison, Wisconsin, USA.
- Cannon, D., Glidewell, C., Low, J. N. & Wardell, J. L. (2000). *Acta Cryst.* **C56**, 1267–1268.
- Cannon, D., Low, J. N., McWilliam, S. A., Skakle, J. M. S., Wardell, J. L. & Glidewell, C. (2001). *Acta Cryst.* **C57**, 600–603.
- Cox, P. J., Doidge-Harrison, S. M. S. V., Nowell, I. W., Howie, R. A., Randall, A. P. & Wardell, J. L. (1990). *Inorg. Chim. Acta*, **172**, 225–232.
- Cox, P. J. & Wardell, J. L. (1994). *J. Organomet. Chem.* **482**, 221–226.
- Cox, P. J., Wardell, J. L., Adam, D. & Muir, K. W. (1995). *J. Chem. Crystallgr.* **25**, 487–491.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Fletcher, D. A., McMeeking, R. F. & Parkin, D. (1996). *J. Chem. Inf. Comput. Sci.* **36**, 746–749.
- Glidewell, C., Harrison, W. T. A., Low, J. N., Sime, J. G. & Wardell, J. L. (2001). *Acta Cryst.* **B57**, 190–200.
- Hooft, R. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
- Howie, R. A. & Wardell, J. L. (1994). *Main Group Met. Chem.* **17**, 571–582.
- Howie, R. A. & Wardell, J. L. (1997). *Z. Kristallogr. New Cryst. Struct.* **212**, 379–380.
- Howie, R. A., Wardell, J. L., Zanetti, E., Cox, P. J. & Doidge-Harrison, S. M. S. V. (1992). *J. Organomet. Chem.* **431**, 27–40.
- Kelly, C. J., Skakle, J. M. S., Wardell, J. L., Wardell, S. M. S. V., Low, J. N. & Glidewell, C. (2002). *Acta Cryst.* **B58**, 94–108.
- McArdle, P. (1994). *J. Appl. Cryst.* **27**, 438–439.
- McArdle, P. (2000). *OSCAIL for Windows*. National University of Ireland, Galway, Ireland.
- Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
- Peterson, D. J. (1971). *J. Organomet. Chem.* **26**, 215–223.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1997). *SHELXL97* and *SADABS*. University of Göttingen, Germany.
- Spek, A. L. (2002). *PLATON*. Version of 2002. University of Utrecht, The Netherlands.
- Taylor, R. D. & Wardell, J. L. (1976). *J. Chem. Soc. Dalton Trans.* pp. 1345–1351.
- Wardell, J. L. & Cox, P. J. (1996). *J. Organomet. Chem.* **515**, 253–258.
- Wardell, J. L., Low, J. N. & Glidewell, C. (2000a). *Acta Cryst.* **C56**, 679–681.
- Wardell, J. L., Low, J. N. & Glidewell, C. (2000b). *Acta Cryst.* **C56**, 862–864.
- Wardell, J. L. & Wigzell, J. McM. (1983). *J. Organomet. Chem.* **244**, 225–233.