Isostructural or not? Adducts of some aryltin halides with (*E*)-1,2-bis(4-pyridyl)ethene

Stanley A. Bajue,[a] Choy Lewis,[a] Karen Clarke,[a] Fitzgerald B. Bramwell,[b] Brian O. Patrick[b] and Carolyn Pratt Brock[b]*

[a]Department of Physical Sciences and Computer Science, Medgar Evers College, City University of New York, Brooklyn, NY 11225-2298, USA, and [b]Department of Chemistry, University of Kentucky, Lexington, KY 40506-0055, USA
Correspondence e-mail: cpbrock@uky.edu

The title complexes $[\mu$-(*E*)-4,4′-(ethene-1,2-diyl)dipyridine-$\kappa^2 N$:N']bis[halotris(4-methylphenyl)tin(IV)], $[Sn_2(C_7H_7)_6X_2$-$(C_{12}H_{10}N_2)]$, where halo is chloro (X = Cl) and bromo (X = Br) are isostructural. In both crystals, the molecules lie on inversion centers, and there are voids of *ca* 80 Å^3 that could, but apparently do not, accommodate water molecules. The corresponding iodo structure (X = I) is almost, but not quite, isostructural with the other two compounds; when Br is changed to I, the length of the *c* axis *decreases* by more than 1 Å and the voids are no longer large enough to accomodate any solvent molecule. The related complex $[\mu$-(*E*)-4,4′-(ethene-1,2-diyl)dipyridine-$\kappa^2 N$:N']bis[chlorotriphenyltin(IV)], $[Sn_2(C_6H_5)_6Cl_2(C_{12}H_{10}N_2)]$, crystallizes in a related structure, but the molecules lie on general rather than on special positions. The molecular structures of the four complexes are similar, but the conformation of the phenyl derivative is approximately eclipsed rather than staggered.

Comment

The structures of the four molecules shown in the *Scheme* below were determined as part of a study of Sn complexes (Bajue *et al.*, 1992). The structures are interesting crystallographically because the members of the set R = Me/X = Cl, R = Me/X = Br, and R = Me/X = I are almost, but not quite, isostructural. The packing in the R = H/X = Cl crystal is similar even though the molecular volume is *ca* 13% smaller.

The molecular structures of the four compounds (see Fig. 1) are similar. The tolyl groups are staggered in the R = Me/X = Cl, R = Me/X = Br, and R = Me/X = I molecules, all of which lie on inversion centers. In the R = H/X = Cl molecule, which has no imposed crystallographic symmetry, the phenyl groups are approximately eclipsed [torsion angles 22.0 (1), 17.1 (1), and 15.0 (1)° for angles Cn—Sn1$\cdots$Sn2—C(n + 24), with n = 7, 13, and 19]. While the R = Me/X = Cl and R = Me/X = Br mol-

ecules are nearly identical, the positions and orientations of the tolyl rings in the R = Me/X = I molecule are a little different. An important stilbene-like disorder (Bouwstra *et al.*, 1984, and references therein) of the bridging *trans*-1,2-bis(4-pyridyl)ethene ligand (hereafter, the bpe ligand) in the iodo compound is obvious (see Fig. 1 and *Experimental*). This disorder is good evidence that the structure of the iodo compound differs in significant, if small, ways from the structures of the chloro and bromo compounds.

R=Me/X=Cl
R=Me/X=Br
R=Me/X=I
R=H/X=Cl
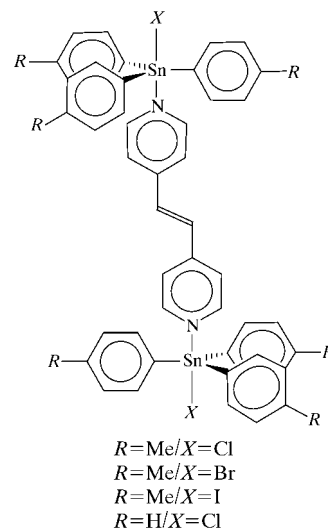
The bpe ligand is significantly non-planar in the R = Me/X = I molecule, but is essentially planar in the other three molecules. The torsion angles C2—C3—C6—C6′ and C4—C3—C6—C6′ for the major component of R = Me/X = I are −157.5 (7) and 23.2 (11)°, but are 0 (1) and 179 (1)° for R = Me/X = Cl, and are 4 (1) and 177 (1)° for R = Me/X = Br. For the two independent halves of the R = H/X = Cl molecule, the corresponding angles are 6 (1) and 174 (1)°, and 6 (1) and 172 (1)°.

A quick look at the packing diagrams for the three R = Me structures (see Figs. 2 and 3) suggests that the compounds are isostructural, but a comparison of the cell constants indicates otherwise. The value of *a* increases by 0.095 (2) Å and by 0.431 (2) Å when Cl is changed to Br and then I; the corresponding pairs of changes for *b* are 0.041 (2) and 0.263 (2) Å. The changes for *c*, however, are +0.055 (3) Å when Cl is changed to Br but −1.074 (3) Å when Br is changed to I. The *c* axis is very significantly shorter in the I compound than in the Br compound. The volume per molecule increases by 19.9 (4) Å^3 when Cl is changed to Br, but remains unchanged [ΔV is −0.1 (4) Å^3] when Br is changed to I. The structure of the I derivative is more closely packed than are the structures of the Br and Cl derivatives.

The free spaces in the R = Me/X = Cl and R = Me/X = Br structures are concentrated in regions classified by *PLATON* (Spek, 2003) as 'solvent-accessible voids' (see Fig. 2). Although the empty spaces seemed large enough (81 and 83 Å^3) to accommodate small solvent molecules, a more detailed analysis (Spek, 2002) indicated that the spaces are too constricted (*i.e.* dumb-bell-shaped) to hold any molecule
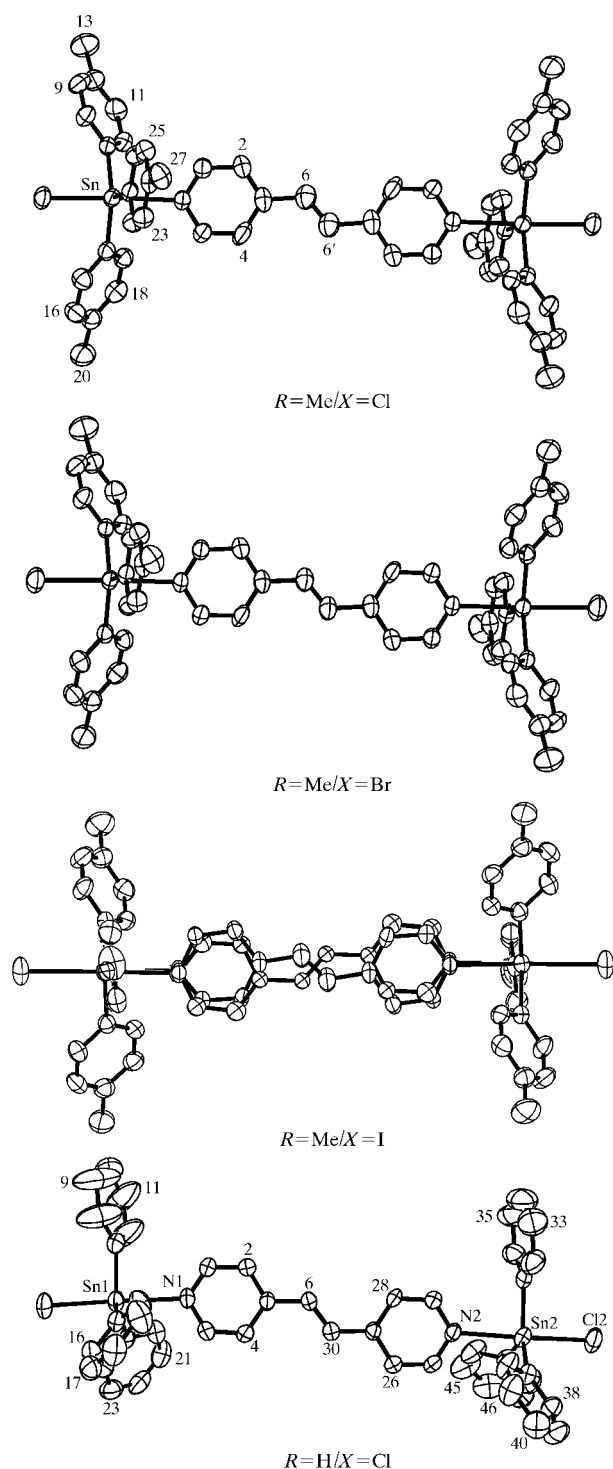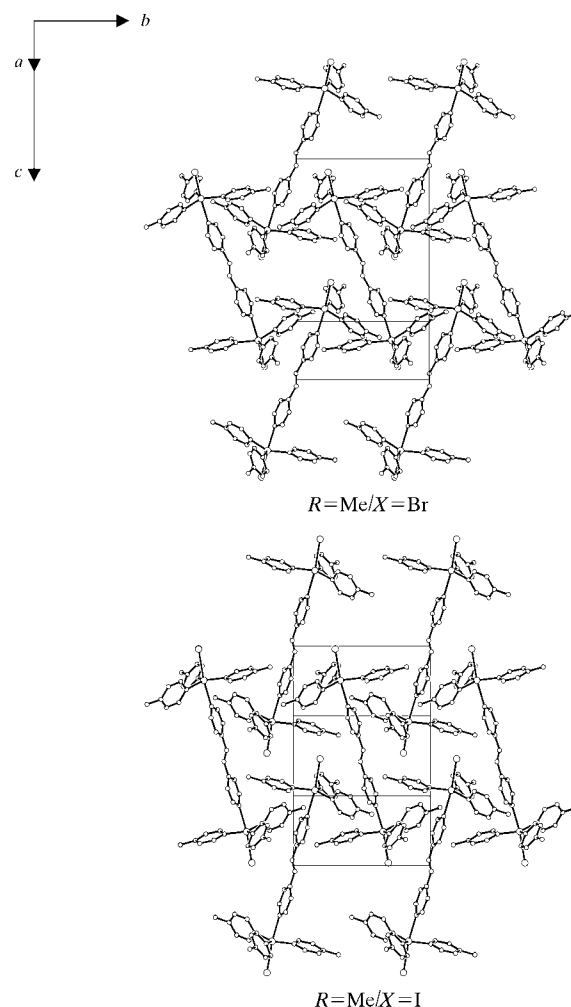
Figure 1
Perspective drawings of the molecules of the four title compounds; enough information is given about the atom-numbering schemes that the numbers of all atoms can be worked out. The schemes for the first three molecules, all of which lie on inversion centers, are the same except for the label of the halogen atom and the labels of the two independent N atoms (N1 and N1A) in the R = Me/X = I structure. The C atoms in the phenyl rings are numbered sequentially, with the lowest number assigned to the C atom attached to the Sn atom (see Table 1). The N atom (label not shown) of the bpe ligand is attached to atoms Sn, C1, and C5. The numbering scheme for the fourth molecule, which lies on a general position, is similar. The shapes of the ellipsoids correspond to 50% probability contours of atomic displacement. H atoms have been omitted for clarity.

larger than water. Since no water was present during the synthesis or recrystallization, and since little electron density is found in the void regions (see *Experimental*), we conclude that the voids are empty. *PLATON* (Spek, 2003) identifies no void in the R = Me/X = I or R = H/X = Cl structure.

The crystal packing can be described in terms of planes that contain the real-space directions [101] and [010] (R = Me; see Fig. 3) or [001] and [010] (R = H; see bottom half of Fig. 4). Within these layers (Fig. 2 and the top half of Fig. 4) there are short $X \cdots H$—C interactions (see Table 2) that involve atoms C4 and C6 and either their symmetry equivalents in the other half of the molecule (R = Me) or atoms C26 and C30 (R = H). These interactions are substantially weaker in the X = I structure than in the other three structures.

Drawings of these layers for the R = Me/X = Cl and R = Me/X = Br structures are nearly superimposable. There are very

Figure 2
Slices of the R = Me/X = Br (upper drawing) and R = Me/X = I (lower drawing) structures viewed along $-a^*+c^*$. These slices are the layers described in the text. The vectors $a+c$ are in the planes of the drawings and point vertically downwards; the centers of the drawings are at $\frac{1}{2},\frac{1}{2},\frac{1}{2}$. The possible voids are located at, for example, $0,\frac{1}{2},0$ and at $\frac{1}{2},0,\frac{1}{2}$. The differences between the two structures are most easily seen in the regions of the drawing in which the phenyl rings appear most densely packed. In the R = Me/X = I structure, a single circle represents the disordered atoms C6 and C6A.

subtle differences, however, between the layers for the X = Br and X = I structures (see Fig. 2). The long molecular axes for the R = H/X = Cl compound make a smaller angle (see top half of Fig. 4) with the b axis, but the basic arrangement is the same.

No significant difference between the stacking of these layers in the R = Me/X = Cl and R = Me/X = Br compounds can be identified, but the distance across $x = z = \frac{1}{2}$, $y = 0$ between the I atoms in molecules related by the translation $a+c$ is substantially shorter (6.46 Å) than the corresponding distances between the Br or Cl atoms (8.77 and 9.09 Å; all s.u. values < 0.002 Å). The shorter of the two $X \cdots X$ contacts across the inversion center at $x = y = z = \frac{1}{2}$ are similar in the three structures (8.68, 8.42 and 8.27 Å for X = I, Br and Cl, respectively). All other $X \cdots X$ contacts are longer than 12 Å.

There is no question that the R = Me/X = Cl and R = Me/X = Br compounds should be considered isostructural. These crystals are so similar that the formation of mixed crystals (or solid solutions) seems likely and may even have been observed (see *Experimental*). The R = Me/X = I structure is very similar, but important differences in the molecular conformation, the intermolecular interactions, and the ratios of the cell constants can be identified. The choice of whether or not to describe the Br and I structures as isostructural would probably depend on the purpose of the comparison.

For the R = Me/X = Cl and R = H/X = Cl structures it is the similarities, rather than the structural differences, that are easily missed. The doubling of the asymmetric unit is a surprise, because most molecules and ions that can conform to inversion symmetry do (Yao *et al.*, 2002). It looks as if (see Fig. 3) the methyl groups in the tolyl series prevent tighter packing of the layers. When the Me groups are replaced by H atoms, the structure collapses (see bottom half of Fig. 4) so that the 'voids' are much smaller. The change in imposed symmetry and conformation means that the contacts on the two sides of the layer are different (see Fig. 4). The tighter packing is reflected in the melting point; T_{fus} rises by *ca* 50 K when R = Me/X = Cl is changed to R = H/X = Cl, even though the molar mass drops by 84 Da (see *Experimental*).
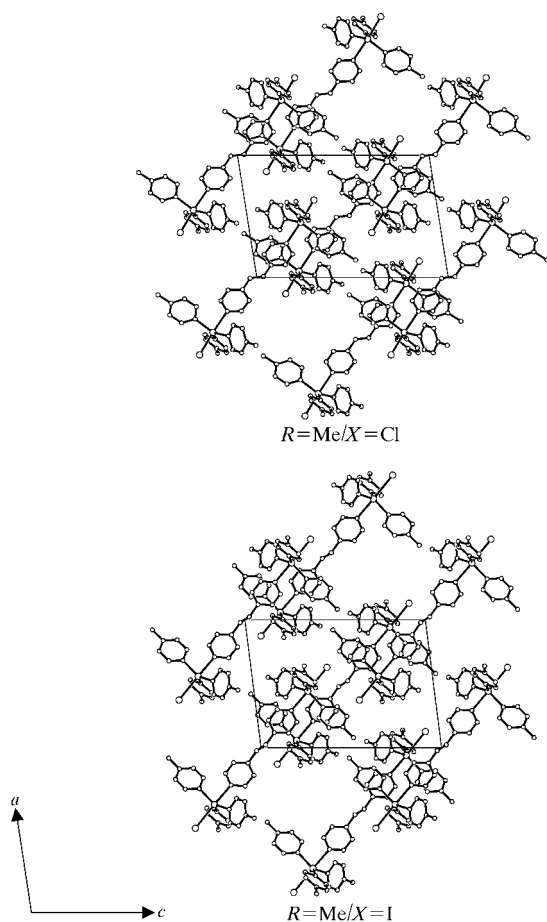
R=Me/X=Cl

R=Me/X=I

Figure 3
Slices of the R = Me/X = Cl (upper drawing) and R = Me/X = I (lower drawing) structures viewed along b; the centers of the drawings are at $\frac{1}{2},\frac{1}{2},\frac{1}{2}$. The differences between the two structures are most easily seen around the central bond (C6—C6′) of the bpe ligand. The layers described in the text are parallel to $a+c$ and b, and so are viewed edge-on; these layers extend from the lower left to the upper right of the drawings. In the X = I structure, a single circle represents the disordered atoms C6 and C6A.
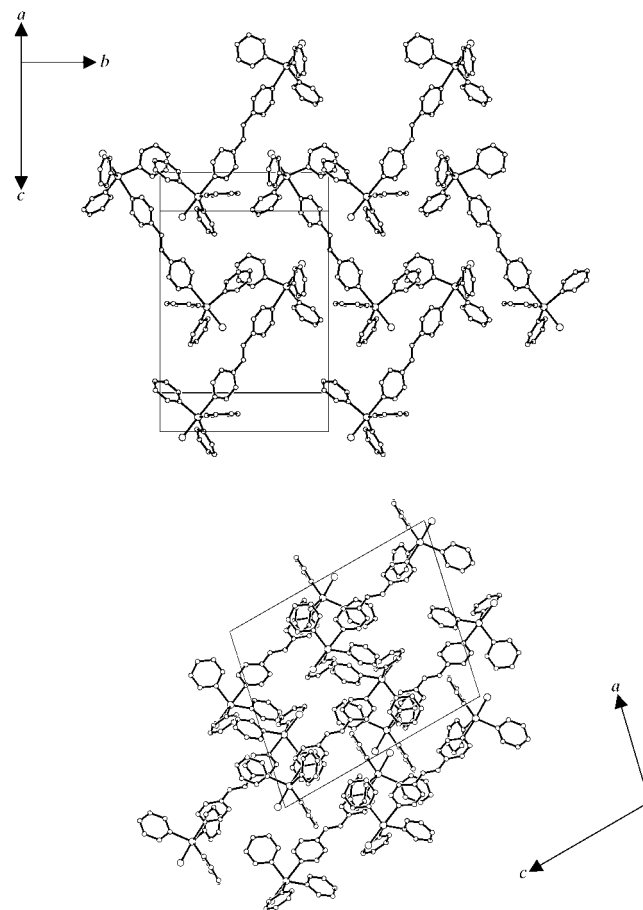
Figure 4
Slices of the R = H/X = Cl structure drawn to maximize the similarities to the drawings shown in Figs. 2 and 3. The upper drawing shows a slice of the structure viewed along a^*; the center of the drawing is at $\frac{1}{4},1,\frac{1}{4}$. The lower drawing shows a slice viewed along $-b$; the center of the drawing is at $\frac{1}{2},\frac{1}{2},\frac{1}{2}$.

metal-organic compounds

Experimental

The title adducts were prepared by refluxing the triorganotin halides with (E)-4,4′-(ethene-1,2-diyl)bis(pyridine) (bpe) in ethanol for about 1 h. Typically, a 0.001 mol sample of the tin compound was used. The reactions were carried out using various mole ratios of Sn-to-ligand, but only 2:1 adducts were obtained. The melting points are 409–410 (R = Me/X = Cl), 395–397 (R = Me/X = Br), 369–371 (R = Me/X = I), and 460–462 K (R = H/X = Cl).

Compound with R = Me/X = Cl

Crystal data

$[Sn_2(C_7H_7)_6Cl_2(C_{12}H_{10}N_2)]$	$D_x = 1.385$ Mg m^{-3}
$M_r = 1037.26$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 50 324
$a = 11.641$ (1) Å	reflections
$b = 12.019$ (1) Å	$\theta = 2.0$–25.0°
$c = 17.999$ (2) Å	$\mu = 1.15$ mm^{-1}
$\beta = 98.94$ (1)°	$T = 293$ (1) K
$V = 2487.7$ (4) Å^3	Block, colorless
$Z = 2$	$0.32 \times 0.32 \times 0.24$ mm

Data collection

Nonius KappaCCD diffractometer	4372 independent reflections
φ and ω scans with 1.0° steps	3781 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan	$R_{int} = 0.017$
($DENZO$–SMN; Otwinowski &	$\theta_{max} = 25.0°$
Minor, 1997)	$h = 0 \rightarrow 13$
$T_{min} = 0.65$, $T_{max} = 0.74$	$k = -14 \rightarrow 14$
8292 measured reflections	$l = -21 \rightarrow 21$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.031P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.028$	$+ 1.8P]$
$wR(F^2) = 0.070$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} = 0.001$
4372 reflections	$\Delta\rho_{max} = 0.72$ e Å^{-3}
271 parameters	$\Delta\rho_{min} = -0.45$ e Å^{-3}
H-atom parameters constrained	

Compound with R = Me/X = Br

Crystal data

$[Sn_2Br_2(C_7H_7)_6(C_{12}H_{10}N_2)]$	$D_x = 1.481$ Mg m^{-3}
$M_r = 1126.18$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 20 962
$a = 11.736$ (1) Å	reflections
$b = 12.060$ (1) Å	$\theta = 3.6$–25.0°
$c = 18.054$ (2) Å	$\mu = 2.62$ mm^{-1}
$\beta = 98.76$ (1)°	$T = 295$ (1) K
$V = 2525.5$ (4) Å^3	Block, colourless
$Z = 2$	$0.37 \times 0.21 \times 0.21$ mm

Table 1
Selected distances (Å) in the four title compounds.

When two distances are given on the same line for the same compound the distance to the lower numbered atom is given first. The Sn—N distance given for the X = I compound is for the major site.

		R = Me/ X = Cl	R = Me/ X = Br	R = Me/ X = I	R = H/ X = Cl
Sn	X	2.515 (1)	2.663 (1)	2.910 (1)	2.475 (1), 2.516 (1)
Sn	N	2.492 (2)	2.477 (3)	2.482 (4)	2.478 (2), 2.479 (2)
Sn	C7(C7,C31)	2.136 (3)	2.135 (3)	2.136 (4)	2.130 (3), 2.124 (3)
Sn	C14(C13,C37)	2.141 (3)	2.147 (3)	2.146 (4)	2.139 (3), 2.132 (3)
Sn	C21(C19,C43)	2.130 (3)	2.127 (3)	2.127 (4)	2.143 (3), 2.145 (3)

Table 2
Calculated H$\cdots X$ contact distances (Å) in the four title compounds.

For the R = H/X = Cl compound, the first two distances are H6$\cdots$Cl2 and H30$\cdots$Cl1, and the second two are H28$\cdots$Cl2 and H4$\cdots$Cl1.

	H6$\cdots X$	H4$\cdots X$	van der Waals radii sum (Bondi, 1964)
R = Me/X = Cl	2.95	2.81	2.95
R = Me/X = Br	3.01	2.88	3.05
R = Me/X = I	3.21	3.33	3.18
R = H/X = Cl	2.85, 2.88	2.86, 3.29	2.95

Symmetry information: in the three R = Me structures, H4 and H6($1 - x$, $1 - y$, $1 - z$) interact with $X(\frac{3}{2} - x, -\frac{1}{2} + y, \frac{3}{2} - z)$; in the R = H structure, H4 and H30 interact with Cl1(x, $\frac{3}{2} - y$, $\frac{1}{2} + z$), while H6 and H28 interact with Cl2(x, $\frac{1}{2} - y$, $\frac{1}{2} + z$). The distances for atoms H4A and H6A of the minor component in the X = I structure are substantially longer than those shown for atoms H4 and H6 of the major component.

Data collection

Nonius KappaCCD diffractometer	3932 reflections with $I > 2\sigma(I)$
φ and ω scans with 1.0° steps	$R_{int} = 0.027$
Absorption correction: multi-scan	$\theta_{max} = 25.0°$
($SORTAV$; Blessing, 1995, 1997)	$h = -13 \rightarrow 13$
$T_{min} = 0.50$, $T_{max} = 0.58$	$k = -14 \rightarrow 14$
13 805 measured reflections	$l = -19 \rightarrow 21$
4416 independent reflections	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.051P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.036$	$+ 4.2P]$
$wR(F^2) = 0.105$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} < 0.001$
4416 reflections	$\Delta\rho_{max} = 0.52$ e Å^{-3}
271 parameters	$\Delta\rho_{min} = -1.37$ e Å^{-3}
H-atom parameters constrained	

Compound with R = Me/X = I

Crystal data

$[Sn_2(C_7H_7)_6I_2(C_{12}H_{10}N_2)]$	$D_x = 1.605$ Mg m^{-3}
$M_r = 1220.16$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 20 894
$a = 12.167$ (1) Å	reflections
$b = 12.323$ (1) Å	$\theta = 3.2$–25.0°
$c = 16.980$ (2) Å	$\mu = 2.25$ mm^{-1}
$\beta = 97.28$ (1)°	$T = 295$ (1) K
$V = 2525.4$ (4) Å^3	Block, pale yellow
$Z = 2$	$0.45 \times 0.40 \times 0.30$ mm

Data collection

Nonius KappaCCD diffractometer	3862 reflections with $I > 2\sigma(I)$
φ and ω scans with 1.0° steps	$R_{int} = 0.035$
Absorption correction: multi-scan	$\theta_{max} = 25.0°$
($SORTAV$; Blessing, 1995, 1997)	$h = -14 \rightarrow 14$
$T_{min} = 0.36$, $T_{max} = 0.48$	$k = -14 \rightarrow 14$
13 846 measured reflections	$l = -20 \rightarrow 20$
4428 independent reflections	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.01P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.030$	$+ 1.8P]$
$wR(F^2) = 0.062$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{max} = 0.001$
4428 reflections	$\Delta\rho_{max} = 0.38$ e Å^{-3}
303 parameters	$\Delta\rho_{min} = -0.72$ e Å^{-3}
H atoms treated by a mixture of	Extinction correction: $SHELXL$97
independent and constrained	Extinction coefficient: 0.0009 (2)
refinement	

Compound with R = H/X = Cl

Crystal data

$[Sn_2(C_6H_5)_6Cl_2(C_{12}H_{10}N_2)]$	$D_x = 1.466$ Mg m^{-3}
$M_r = 953.10$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 49 334
$a = 15.767$ (2) Å	reflections
$b = 14.640$ (2) Å	$\theta = 1.0$–$25.0°$
$c = 19.148$ (3) Å	$\mu = 1.31$ mm^{-1}
$\beta = 102.34$ (1)$°$	$T = 293$ (1) K
$V = 4317.8$ (11) Å^3	Plate, colourless
$Z = 4$	$0.32 \times 0.21 \times 0.16$ mm

Data collection

Nonius KappaCCD diffractometer	5778 reflections with $I > 2\sigma(I)$
φ and ω scans with 1.0$°$ steps	$R_{int} = 0.038$
Absorption correction: multi-scan	$\theta_{max} = 25.0°$
($SORTAV$; Blessing, 1995, 1997)	$h = 0 \rightarrow 18$
$T_{min} = 0.66$, $T_{max} = 0.81$	$k = 0 \rightarrow 17$
10 037 measured reflections	$l = -22 \rightarrow 21$
7585 independent reflections	

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.027$	$w = 1/[\sigma^2(F_o^2)]$
$wR(F^2) = 0.064$	$(\Delta/\sigma)_{max} = 0.003$
$S = 0.81$	$\Delta\rho_{max} = 0.41$ e Å^{-3}
7585 reflections	$\Delta\rho_{min} = -0.53$ e Å^{-3}
487 parameters	

For the R = Me/X = Cl compound, the position of the largest peak in the final difference Fourier map (0.72 e Å^{-3}) suggests the possibility of minor disorder of atom C6 of the type known for stilbene (Bouwstra *et al.*, 1984, and references therein). The second largest peak (0.38 e Å^3) lies 1.83 Å from the inversion center at $0,\frac{1}{2},0$ in a region that *PLATON* (Spek, 2003) identifies as a solvent-accessible void with a volume of 81 Å^3. Crystals had been grown from benzene, which has an approximate volume (Hofmann, 2002) of 114 Å^3, but ethanol (approximate volume 70 Å^3) may also have been present. In any event, no pattern of peaks corresponding to a localized solvent molecule could be identified. Other peaks in the difference map (0.36 e Å^{-3}) are near the mid-points of bonds or suggest a set of alternate positions for the methyl H atoms. The deepest trough (-0.45 e Å^{-3}) is 0.75 Å from the Sn atom.

For the R = Me/X = Br compound, the largest peak in the final difference Fourier map (0.52 e Å^{-3}) is 0.68 Å from the Sn atom. The second largest peak (0.51 e Å^{-3}) suggests a stilbene-like disorder of atom C6. The third largest peak (0.37 e Å^{-3}) is part of a cluster around the Br atom. The fourth largest peak (0.37 e Å^{-3}) lies 1.76 Å from the inversion center at $0,\frac{1}{2},0$ in a solvent-accessible region of volume 83 Å^3. Crystals had been grown from ethanol. The most important feature of the final difference map, however, is a set of three large troughs (-1.14 to -1.37 e Å^{-3}) near the Br atom. It seems possible that some of the supposed Br atoms were actually Cl atoms.

For the R = Me/X = I compound, when the standard anisotropic refinement had converged the atomic ellipsoids showed clearly that the bpe ligand is disordered in the same way as stilbene (Bouwstra *et al.*, 1984, and references therein). The final disorder model included two sites (see Fig. 1); the occupancy factor for the major site (N1/C1–C6) is 0.739 (7). The refinement included restraints to keep the pyridyl rings planar (FLAT 0.10) and to keep the distances and angles in the two BPE ligands similar (SAME and SADI 0.002). The two N atoms were required to have the same anisotropic displacement

parameters; restraints (SIMU 0.012) kept the displacement parameters of atoms C1–C6 and C1A–C6A similar.

For the R = H/X = Cl compound, the atomic ellipsoids for the phenyl ring containing atoms C7–C12 suggest disorder between rings displaced by *ca* 0.6 Å. A number of disorder models were explored but we eventually decided they offered no advantage. The molecular geometry of this ring, which has C—C bonds that are a little shorter than average, is not accurate. The top five peaks in the final difference map (0.41 to 0.25 e Å^{-3}) are associated with this ring. The deepest trough (-0.58 e Å^{-3}) is 0.74 Å from the Sn atom.

The H atoms in all four structures were placed at calculated positions. The C—H distances in the methyl groups were fixed at 0.96 Å; all other C—H distances were fixed at 0.93 Å. The U_{iso} values for the methyl H atoms were set to 1.5 times the U_{eq} value for the attached C atom; the multiplicative factor was 1.2 for all other H atoms. The use of calculated positions for the methyl groups of the tolyl rings (HFIX/AFIX 33) in the X = Cl and X = Br structures is almost certainly a serious approximation; in the X = I structure there were contacts so short that the methyl groups were allowed to rotate (AFIX 137).

For all compounds, data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997) in *maXus* (Mackay *et al.*, 1998); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1990).

References

Bajue, S. A., Bramwell, F. B., Charles, M., Cervantes-Lee, F. & Pannell, K. (1992). *Inorg. Chim. Acta*, **197**, 83–97.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–37.
Blessing, R. H. (1997). *J. Appl. Cryst.* **30**, 421–426.
Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.
Bouwstra, J. A., Schouten, A. & Kroon, J. (1984). *Acta Cryst.* C**40**, 428–431.
Hofmann, D. W. M. (2002). *Acta Cryst.* B**58**, 489–493.
Mackay, S., Gilmore, C. J., Edwards, C., Tremayne, M., Stewart, N. & Shankland, K. (1998). *maXus*. University of Glasgow, Scotland, Nonius BV, Delft, The Netherlands, and MacScience Co. Ltd, Yokohama, Japan.
Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
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.
Sheldrick, G. M. (1990). *SHELXTL/PC*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2002). Personal communication.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Yao, J. W., Cole, J. C., Pidcock, E., Allen, F. H., Howard, J. A. K. & Motherwell, W. D. S. (2002). *Acta Cryst.* B**58**, 640–646.