$\omega/2\theta$ scans
Absorption correction:
$\psi$ scans
$T_{min} = 0.59$, $T_{max} = 0.99$
2406 measured reflections
2180 independent reflections
1796 observed reflections
[$F > 4\sigma(F)$]

$h = -9 \rightarrow 9$
$k = 0 \rightarrow 18$
$l = 0 \rightarrow 11$
2 standard reflections
monitored every 400 reflections
intensity decay: none

*Refinement*

Refinement on $F^2$
$R(F) = 0.0515$
$wR(F^2) = 0.1410$
$S = 1.265$
2180 reflections
113 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0738P)^2 + 1.51P]$
where $P = [\max(F_o^2,0) + 2F_c^2]/3$

$(\Delta/\sigma)_{max} = 0.074$
$\Delta\rho_{max} = 3.1$ e Å$^{-3}$
$\Delta\rho_{min} = -3.3$ e Å$^{-3}$
Extinction correction: none
Atomic scattering factors from *SHELXTL-Plus* (Sheldrick, 1990)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å$^2$)*

$U_{eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

| | $x$ | $y$ | $z$ | $U_{eq}$ |
|---|---|---|---|---|
| Au | 0.1801 (1) | 0.1156 (1) | 0.0890 (1) | 0.024 (1) |
| Cl(1) | 0.4666 (5) | 0.1222 (3) | 0.0708 (5) | 0.047 (1) |
| Cl(2) | 0.1250 (5) | 0.1925 (2) | −0.1339 (4) | 0.041 (1) |
| Cl(3) | −0.1068 (4) | 0.1033 (3) | 0.1086 (4) | 0.045 (1) |
| N | 0.2268 (11) | 0.0457 (7) | 0.2869 (11) | 0.025 (2) |
| C(1) | 0.2083 (18) | 0.0839 (8) | 0.4264 (13) | 0.030 (3) |
| C(2) | 0.2413 (19) | 0.0301 (10) | 0.5509 (16) | 0.041 (3) |
| C(3) | 0.2945 (21) | −0.0581 (10) | 0.5444 (16) | 0.045 (4) |
| C(4) | 0.3135 (19) | −0.0957 (9) | 0.4006 (15) | 0.039 (3) |
| C(5) | 0.2810 (19) | −0.0430 (9) | 0.2788 (17) | 0.041 (3) |
| C(6) | 0.1581 (25) | 0.1813 (9) | 0.4385 (17) | 0.052 (4) |
| O | 0.2155 (15) | 0.2386 (7) | 0.3313 (11) | 0.044 (2) |

Table 2. *Selected geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| Au—N | 2.021 (9) | C(1)—C(2) | 1.35 (2) |
| Au—Cl(2) | 2.265 (3) | C(1)—C(6) | 1.48 (2) |
| Au—Cl(1) | 2.268 (4) | C(2)—C(3) | 1.36 (2) |
| Au—Cl(3) | 2.278 (4) | C(3)—C(4) | 1.41 (2) |
| N—C(1) | 1.374 (14) | C(4)—C(5) | 1.32 (2) |
| N—C(5) | 1.37 (2) | C(6)—O | 1.38 (2) |
| N—Au—Cl(2) | 179.2 (3) | N—C(1)—C(2) | 117.6 (11) |
| N—Au—Cl(1) | 90.0 (3) | N—C(1)—C(6) | 120.6 (11) |
| Cl(2)—Au—Cl(1) | 90.52 (14) | C(2)—C(1)—C(6) | 121.8 (12) |
| N—Au—Cl(3) | 88.7 (3) | C(3)—C(2)—C(1) | 123.2 (14) |
| Cl(2)—Au—Cl(3) | 90.80 (14) | C(2)—C(3)—C(4) | 118.3 (12) |
| Cl(1)—Au—Cl(3) | 177.87 (14) | C(3)—C(4)—C(5) | 118.5 (13) |
| C(1)—N—C(5) | 119.8 (11) | N—C(5)—C(4) | 122.7 (14) |
| C(1)—N—Au | 122.9 (8) | O—C(6)—C(1) | 115.0 (13) |
| C(5)—N—Au | 117.4 (9) | | |

The H atom bonded to O was located from a difference Fourier map; other H atoms were placed in calculated positions. H atoms were refined with a common isotropic displacement parameter. The highest values of $\Delta\rho$ are near the heavy atoms and are probably the result of uncorrected thermal motion or inadequacies in the scattering factors.

Data collection: Nonius CAD-4 software. Cell refinement: Nonius CAD-4 software. Data reduction: Nonius CAD-4 software. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: NA1179). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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# Diphenyltin Diiodide

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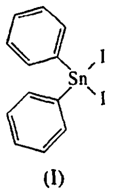
## Abstract

The title compound, diiododiphenyltin, [$SnI_2(C_6H_5)_2$], has essentially tetrahedral coordination geometry with intermolecular I···I and Sn···I separations close to the sum of the appropriate van der Waals radii.

## Comment

The title compound, (I), consists of essentially individual $Ph_2SnI_2$ molecules separated by I···I and Sn···I distances close to the sum of the appropriate van der Waals radii; the van der Waals radii for Sn and I were taken to be 2.20 and 1.95–2.12 Å, respectively (Huheey,



(I)

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Keiter & Keiter, 1993). Molecules of (I) form layers coincident with the ($\bar{1}01$) plane, with Sn and I atoms in the plane.

The four primary bonds to tin in (I) are arranged in a slightly distorted tetrahedral array, with the bond angles ranging between 105.17 (3) (I2—Sn—I1) and 116.7 (4) Å (C7—Sn—C1) (Fig. 1). The corresponding bond angles in Et$_2$SnI$_2$ were reported to be 104.0 (2) and 130.2 (11) Å (Alcock & Sawyer, 1977). The Sn—C [2.133 (9) and 2.140 (10) Å] and Sn—I [2.6749 (10) and 2.7097 (10) Å] bond lengths are comparable to those found in both Ph$_2$SnCl$_2$ [2.104 (5)–2.119 (5) Å; Greene & Bryan, 1971] and Et$_2$SnI$_2$ [2.719 (4) Å; Alcock & Sawyer, 1977].

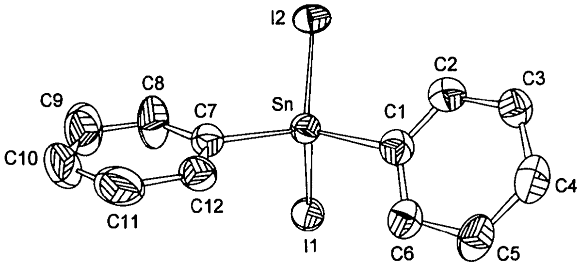

Fig. 1. View of the title compound showing the atom-numbering scheme. Ellipsoids are plotted at the 40% probability level and H atoms have been omitted for clarity.

Significant intermolecular contacts in compound (I) are I1···I2$^{\rm ii}$ and I2···I1$^{\rm iii}$ [4.0745 (12) Å], I1···I2$^{\rm i}$ and I2···I1$^{\rm iv}$ [4.2490 (11) Å], and possibly also Sn$^{\rm i}$···I1 [4.3885 (10) Å] (Fig. 2; symmetry operations are as given in Table 2). The Sn$^{\rm i}$···I1 separation is just outside the limits of the van der Waals radii sum (4.15–4.32 Å), but the facts that the I1$^{\rm i}$—Sn$^{\rm i}$···I1 angle is close to linearity [174.21 (3)°] and the Sn—I1 bond is slightly longer than Sn—I2 suggest that this interaction is also significant. Alcock & Sawyer (1977) similarly interpreted intermolecular Sn···I separations of 4.284 (5) Å as indicating secondary bonding in Et$_2$SnI$_2$, with the consequent formation of chains of molecules. Secondary bonding and chain formation was also suggested for other Et$_2$Sn$X_2$ compounds ($X$ = Cl, Br) (Alcock & Sawyer, 1977). In the case of Et$_2$SnI$_2$, both I atoms of one molecule form bridging secondary bonds to the same neighbouring molecule; the somewhat different situation for (I) is shown in Fig. 2. While Greene & Bryan (1971) interpreted their structural data for Ph$_2$SnCl$_2$ in terms of molecular species in which distortion at the Sn atom from tetrahedral is induced by electronic effects rather than by intermolecular interactions, a subsequent reinterpretation by Bokii, Struchkov & Prokofiev (1972) utilized a weakly aggregated model, which seems to be appropriate also for the present compound.

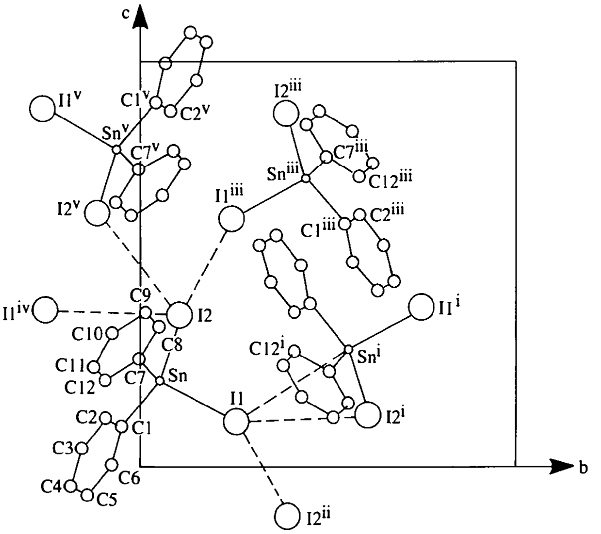

Fig. 2. The unit cell of the title compound viewed down **a**, showing intermolecular contacts. Atoms are represented by circles of arbitrary size and H atoms have been omitted for clarity. Symmetry operations are as given in Table 2.

## Experimental

The title compound ($^{119}$Sn NMR: $\delta$ −243.8 p.p.m. in CDCl$_3$) was prepared from the halogen-exchange reaction of Ph$_2$SnCl$_2$ and excess NaI in dry acetone and subsequent recrystallization from EtOH [m.p. 347.5–348 K, literature value 344–345 K; Ingham, Rosenberg & Gilman, 1960].

*Crystal data*

[SnI$_2$(C$_6$H$_5$)$_2$]
$M_r$ = 526.71
Monoclinic
$P2_1/c$
$a$ = 9.548 (7) Å
$b$ = 11.924 (11) Å
$c$ = 12.714 (10) Å
$\beta$ = 94.68 (6)°
$V$ = 1443 (2) Å$^3$
$Z$ = 4
$D_x$ = 2.424 Mg m$^{-3}$
$D_m$ not measured

Mo $K\alpha$ radiation
$\lambda$ = 0.71073 Å
Cell parameters from 14 reflections
$\theta$ = 10.7–11.4°
$\mu$ = 5.98 mm$^{-1}$
$T$ = 298 K
Block
0.6 × 0.5 × 0.4 mm
Colourless

*Data collection*

Nicolet $P3$ diffractometer
$\theta/2\theta$ scans
Absorption correction: $\psi$ scans (North, Phillips & Mathews, 1968)
$T_{\rm min}$ = 0.049, $T_{\rm max}$ = 0.112
4665 measured reflections
3758 independent reflections
2779 observed reflections [$F > 4\sigma(F)$]

$R_{\rm int}$ = 0.025
$\theta_{\rm max}$ = 30°
$h = -13 \rightarrow 13$
$k = 0 \rightarrow 16$
$l = 0 \rightarrow 17$
2 standard reflections monitored every 50 reflections intensity variation: 2%

*Refinement*

Refinement on $F$
$R$ = 0.048
$wR$ = 0.066

$\Delta\rho_{\rm max}$ = 0.72 e Å$^{-3}$
$\Delta\rho_{\rm min}$ = −1.41 e Å$^{-3}$ (0.20 Å from I1)

$S = 0.75$
2779 reflections
137 parameters
$w = 1/[\sigma^2(F) + 0.012134F^2]$
$(\Delta/\sigma)_{max} = 0.050$
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å²)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

| | $x$ | $y$ | $z$ | $U_{eq}$ |
|---|---|---|---|---|
| Sn | 0.21397 (7) | 0.05703 (5) | 0.21018 (5) | 0.0452 (2) |
| I1 | 0.15287 (10) | 0.25475 (6) | 0.11184 (7) | 0.0689 (3) |
| I2 | 0.39800 (9) | 0.10823 (7) | 0.37307 (6) | 0.0652 (3) |
| C1 | 0.3067 (10) | −0.0480 (8) | 0.0984 (8) | 0.048 (2) |
| C2 | 0.4418 (11) | −0.0857 (9) | 0.1186 (8) | 0.052 (2) |
| C3 | 0.4997 (11) | −0.1533 (10) | 0.0453 (9) | 0.057 (3) |
| C4 | 0.4238 (14) | −0.1785 (10) | −0.0473 (9) | 0.065 (3) |
| C5 | 0.2888 (15) | −0.1368 (12) | −0.0700 (10) | 0.071 (4) |
| C6 | 0.2286 (13) | −0.0709 (11) | 0.0033 (9) | 0.066 (3) |
| C7 | 0.0211 (10) | 0.0007 (8) | 0.2654 (8) | 0.048 (2) |
| C8 | −0.0365 (17) | 0.0561 (14) | 0.3463 (14) | 0.095 (6) |
| C9 | −0.1633 (18) | 0.0181 (17) | 0.3787 (14) | 0.093 (6) |
| C10 | −0.2352 (14) | −0.0676 (15) | 0.3287 (14) | 0.086 (5) |
| C11 | −0.1762 (13) | −0.1205 (12) | 0.2485 (12) | 0.074 (4) |
| C12 | −0.0478 (12) | −0.0887 (9) | 0.2163 (9) | 0.055 (3) |

Table 2. *Selected geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| I1—Sn | 2.7097 (10) | I2—Sn | 2.6749 (10) |
| C1—Sn | 2.140 (10) | C7—Sn | 2.133 (9) |
| I2—Sn—I1 | 105.17 (3) | C1—Sn—I1 | 106.7 (3) |
| C1—Sn—I2 | 111.5 (3) | C7—Sn—I1 | 105.5 (3) |
| C7—Sn—I2 | 110.3 (3) | C7—Sn—C1 | 116.7 (4) |
| C2—C1—Sn | 120.1 (7) | C6—C1—Sn | 118.0 (8) |
| C8—C7—Sn | 120.3 (9) | C12—C7—Sn | 119.2 (8) |
| I1···Sn$^{i}$ | 4.3885 (10) | I2···I1$^{iii}$ | 4.0745 (12) |
| I1···I2$^{ii}$ | 4.0745 (12) | I2···I1$^{iv}$ | 4.2490 (11) |
| I1···I2$^{i}$ | 4.2490 (11) | I2···I2$^{v}$ | 4.4550 (11) |

Symmetry codes: (i) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$; (ii) $x - \frac{1}{2}, \frac{1}{2} - y, z - \frac{1}{2}$; (iii) $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$; (iv) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (v) $1 - x, -y, 1 - z$.

The structure was solved by heavy-atom methods and refinement was by full-matrix least-squares methods. All non-H atoms were refined anisotropically. H atoms were placed in calculated positions (C—H = 0.95 Å) and refined riding upon the C atom to which they are attached with a common group $U_{iso}$. All computations were performed on the SUN SPARCserver (UNIX operating system) of the University of Aberdeen.

Data collection: *Nicolet P*3 *Software* (Nicolet XRD Corporation, 1980). Cell refinement: *Nicolet P*3 *Software*. Data reduction: *RDNIC* (Howie, 1980). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *PLOTAID* (Cradwick, 1970) and *ORTEX* (McArdle, 1994). Software used to prepare material for publication: *XPUB* (Low, 1980).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1048). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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# *catena*-Poly[silver-$\mu$-(di-2-pyridyl ketone)-*N*,*O*:*N*′,*O*] Tetrafluoroborate

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## Abstract

The title complex, $[Ag(C_{11}H_8N_2O)]_n^+.n(BF_4)^-$, contains polymeric [silver(di-2-pyridyl ketone)]$_n^+$ zigzag chains, counterbalanced by $BF_4^-$ anions. The Ag atoms reside on inversion centers, the di-2-pyridyl ketone ligands lie with their carbonyl groups along crystallographic twofold axes, and the $BF_4^-$ ions also reside on inversion centers (and are disordered). The $AgN_2O_2$ moiety is precisely planar, with Ag—N 2.180 (3) and Ag—O 2.552 (1) Å, and N—Ag—O 69.35 (9)°.

## Comment

We have described previously *catena*-poly[(nitrato-*O*,*O*′)silver(I)-$\mu$-(di-2-pyridyl ketone-*N*:*N*′)] (Sommerer, Westcott & Abboud, 1994), a metal-containing ex-