Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

trans-(2-Acetylpyridine-κN 2-furylhydrazonato-κ^2N^1,O)dichlorophenyltin(IV) dichloromethane solvate

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Key indicators

Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.009 Å
Disorder in main residue
R factor = 0.037
wR factor = 0.079
Data-to-parameter ratio = 11.2

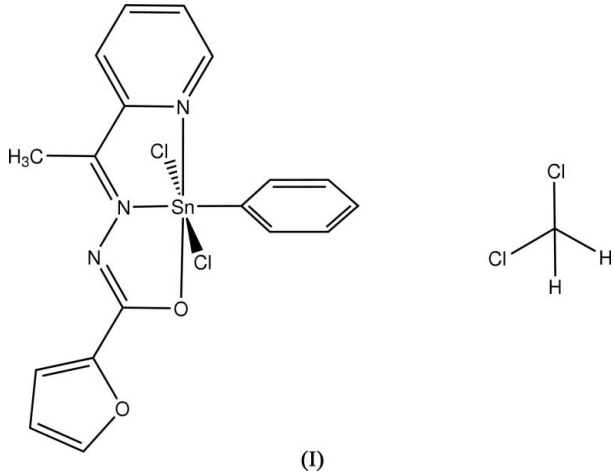
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 14 February 2006
Accepted 14 March 2006

In the structure of the title compound, $[Sn(C_{12}H_{10}N_3O_2)Cl_2(C_6H_5)]\cdot CH_2Cl_2$, the Sn^{IV} ion is in a distorted octahedral coordination geometry, with the phenyl and the N,N',O-donor 2-acetylpyridine 2-furylhydrazonate ligands in equatorial positions and the Cl atoms in axial positions. The crystal structure shows π–π interactions between the pyridine and the coordinated phenyl rings. In the crystal structure, the molecules pack forming channels filled with disordered solvent molecules.

Comment

The title complex, (I), was prepared as part of a research programme devoted to the investigation of the coordination modes of N,N',N'',S,S'- and N,N',S-donor thiosemicarbazones with organotin(IV) compounds (Sousa *et al.*, 1999, 2000, 2001; Francisco *et al.*, 2004). In the context of our continuing interest in the structures and biological activities of these kinds of compounds, we have started to study the coordination behaviour of N,N',N'',O,O'- and N,N',O-donor aroylhydrazones towards organotin(IV) derivatives. In this paper we report the structure of the title compound, (I), obtained from the reaction of the ligand 2-acetylpyridine 2-furylhydrazone with the monoorganotin(IV) $Sn(C_6H_5)Cl_3$ acid.



(I)

In the structure of (I), the Sn^{IV} atom has a distorted octahedral coordination, with the phenyl and hydrazone ligands in the equatorial plane and the chlorides in the axial positions (Fig. 1). The 2-acetylpyridine 2-furylhydrazonate ligand is N,N',O-coordinated through the pyridine and imine N atoms and the hydrazone O atom. The coordinated pyridine and hydrazone groups are approximately planar and the mean plane of the furyl ring deviates by 6.7 (5)° from that through

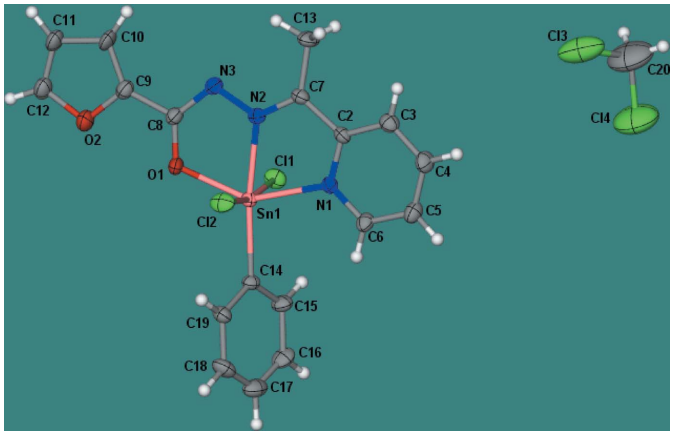


Figure 1
A drawing of the title molecular structure, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Only the major component of disordered atoms is shown.

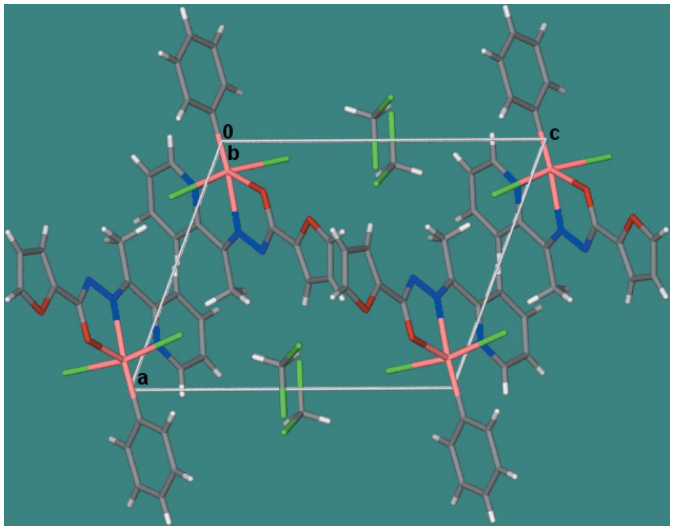


Figure 2
A packing diagram of (I), viewed down the *b* axis. Only the major component of disordered atoms is shown.

the remaining non-H atoms of the ligand. The mean plane of the phenyl ring is rotated by 67.4 (4) and 25.7 (4)°, respectively, with respect to the 2-acetylpyridine 2-furylhydrazonate ligand and Sn1/Cl1/Cl2/N2 unit.

The *trans*-chlorides are bent towards the 2-acetylpyridine 2-furylhydrazonate ligand. A similar coordination mode was observed in $[Sn(C_8H_9N_4O)(C_6H_5)Cl_2]$, (II), where $C_8H_{10}N_4O$ is 2-acetylpyridine semicarbazone (Carcelli *et al.*, 2001). The bond distances and angles in (II) [Sn—Cl1 = 2.473 (1) Å, Sn—Cl2 = 2.469 (1) Å, Sn—N1(pyridine) = 2.227 (2) Å, Sn—N2(imine) = 2.181 (2) Å and Sn—O = 2.099 (2) Å; C9—Sn—N1 = 100.76 (7)°, N1—Sn—N2 = 73.17 (6)°, N2—Sn—O = 73.72 (6)°, O—Sn—C9 = 112.30 (7)°, and Cl1—Sn—Cl2 = 169.47 (2)°] are in good agreement with the corresponding values in (I) (Table 1).

The C14–C19 phenyl (ph) ring of each molecule is sandwiched between the pyridine (py) and furyl (fur) rings of the molecules generated by the symmetry codes $(-x, -\frac{1}{2}+y, -z)$ and $(-x, \frac{1}{2}+y, -z)$, repectively. The ph and py rings are almost parallel, with a centroid–centroid distance of 3.710 (4) Å, a value within the expected distances for π–π interactions (Janiak, 2000). The disordered fur ring is parallel to the ph ring, but the centroid–centroid distance is longer than the accepted distances for π–π interactions [C19···O2 = 4.130 (5) Å and C19*A*···O2*A* = 4.38 (1) Å], indicating that there are no important interactions between these rings.

In the crystal structure, the molecules are packed to form channels, which are filled with the disordered solvent molecules (Fig. 2).

Experimental

The ligand 2-acetylpyridine 2-furylhydrazone was prepared by refluxing an EtOH solution (30 ml) of 2-acetylpyridine (2.3 ml, 20 mmol) and 2-furoic hydrazide (2.52 g, 20 mmol) for 2 h. The solution was kept at 258 K overnight and the colorless microcrystalline compound was then filtered, washed with a small amount of *n*-hexane, and dried in air. A second fraction of the product was obtained in the same way after concentrating the filtered solution to half of its volume (yield 2.91 g, 64%, m.p. 397 K). Analysis for $C_{12}H_{11}N_3O_2$: C 62.06 (calculated 62.87), H 4.88 (4.84), N 18.50% (18.33%). To prepare the title complex, the ligand (0.046 g, 0.20 mmol) was dissolved in methanol (10 ml) and refluxed for 5 min. $Sn(C_6H_6)Cl_3$ (0.080 g, 0.21 mmol) dissolved in CH_2Cl_2 (10 ml) was then added and refluxed for 1 h. After cooling the solution and slow evaporation of the solvent, orange crystals were obtained (yield 0.076 g, 65%, m.p. > 473 K). Analysis for $C_{19}H_{18}Cl_4N_3O_2Sn$: C 39.29 (calculated 39.11), H 3.12 (2.98), N 7.23% (7.17%).

Crystal data

$[Sn(C_{12}H_{10}N_3O_2)Cl_2(C_6H_5)]\cdot CH_2Cl_2$
M_r = 579.85
Monoclinic, $P2_1$
a = 9.116 (2) Å
b = 12.050 (3) Å
c = 11.113 (3) Å
β = 110.71 (3)°
V = 1141.9 (5) Å^3
Z = 2
D_x = 1.686 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5481 reflections
θ = 2.4–30.8°
μ = 1.61 mm^{-1}
T = 291 (2) K
Prism, orange
0.38 × 0.12 × 0.05 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.705, T_{max} = 0.927
9381 measured reflections
3998 independent reflections
3737 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 25.0°
$h = -10 \rightarrow 10$
$k = -14 \rightarrow 14$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.079
S = 1.05
3998 reflections
357 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0412P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.75 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}
Absolute structure: Flack (1983), 1888 Friedel Pairs
Flack parameter: 0.01 (3)

Table 1
Selected geometric parameters (Å, °).

Sn1—O1	2.101 (3)	Sn1—Cl2	2.4611 (16)
Sn1—C14	2.135 (5)	O1—C8	1.285 (6)
Sn1—N2	2.173 (4)	N2—C7	1.282 (6)
Sn1—N1	2.229 (4)	N2—N3	1.369 (6)
Sn1—Cl1	2.4451 (16)	N3—C8	1.324 (7)
O1—Sn1—C14	113.7 (3)	N2—Sn1—Cl1	85.76 (12)
O1—Sn1—N2	73.67 (15)	N1—Sn1—Cl1	91.52 (12)
C14—Sn1—N2	172.5 (3)	O1—Sn1—Cl2	87.84 (12)
O1—Sn1—N1	145.67 (15)	C14—Sn1—Cl2	95.15 (17)
C14—Sn1—N1	100.5 (3)	N2—Sn1—Cl2	83.74 (12)
N2—Sn1—N1	72.06 (15)	N1—Sn1—Cl2	86.38 (14)
O1—Sn1—Cl1	88.08 (12)	Cl1—Sn1—Cl2	169.44 (5)
C14—Sn1—Cl1	95.42 (17)		

H atoms were positioned geometrically, with C—H = 0.93, 0.96 and 0.97 Å for aromatic, methyl and methylene H, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = xU_{eq}(C)$, where $x = 1.5$ for methyl H and $x = 1.2$ for all other H atoms. The dichloromethane solvent molecule was refined with statistical disorder over three positions, with site occupancies of 0.54 (1) (for C20, Cl3 and Cl4), 0.16 (1) (for C20*A*, Cl3*A* and Cl4*A*) and 0.30 (1) (for C20*B*, Cl3*B* and Cl4*B*). The atoms of the furyl ring (except C9) were disordered over two positions, with site occupancies of 0.74 (2) and 0.26 (2), due to a 175° rotation around the C8—C9 bond.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *SHELXL97*, *enCIFer* (Allen *et al.*, 2004) and *PLATON* (Spek, 2003).

The authors are gratefull to UnB, UNAM and CONACYT (40332-Q) for financial support.

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