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2-(Chlorodimethylstannyl)-4-methylbenzenesulfonyl Pyrrolidide

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Abstract. [Sn(C$_{13}$H$_{20}$ClNO$_2$S)], $M_r = 408.51$, orthorhombic, *Pbca*, $a = 7.596$ (5), $b = 18.654$ (13), $c = 23.447$ (19) Å, $V = 3322$ (4) Å^3, $Z = 8$, $D_x = 1.633$ Mg m^{-3}, λ(Mo Kα) = 0.71073 Å, $\mu = 1.82$ mm^{-1}, $F(000) = 1632$, $T = 291$ (1) K, final $R = 0.047$ for 1652 unique observed [$F \geq 4.0\sigma(F)$] diffractometer data. This is the first case of complexation of a triorganotin halide by an SO$_2$ group forming an intramolecular pentacoordination at the Sn atom. The five atoms bound to Sn form a distorted trigonal bipyramid with Cl and O in the apical positions [Sn—Cl 2.438 (3), Sn—O 2.529 (6) Å, Cl—Sn—O 171.4 (2)°] and the C atoms in the equatorial plane [Sn—C 2.113 (8), 2.13 (1), 2.171 (8) Å, C—Sn—C 107.4 (3), 124.1 (3), 122.2 (4)°]. The O—Sn—C and Cl—Sn—C angles are in the range from 75.3 (2) (ring angle) to 99.1 (3)° and the bond angles at S are in the range from 105.5 (4) to 118.5 (4)°. Short intermolecular contacts around the free O at S indicate weak hydrogen bonds.

Experimental. The crystals were obtained from hexane/dichloromethane, 3:1, m.p. 419 K. A well developed crystal platelet of size ∼0.06 × 0.13 × 0.35 mm was used. Its quality was checked with optical polarizing microscopy. The crystal was mounted on a glass fibre. D_m was not determined. Intensity data were collected with $\omega/2\theta$ scans, constant scan speed, scan width 0.8° + dispersion (0.02° steps, 1 s per step). A modified Hilger & Watts diffractometer (Lange & Burzlaff, 1991) with graphite-monochromated Mo Kα radiation was used. The lattice parameters were determined from a least-squares fit of 20 reflections with $2\theta_{max} = 17.7°$. Three standard reflections (200, 060, 0,0,10) were recorded every 100 reflections and an intensity loss of up to 7% was detected during data collection; 13 110
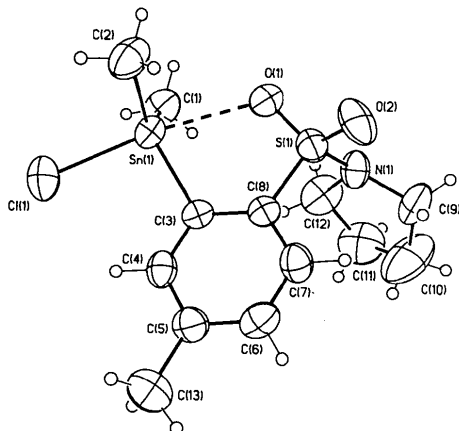

Fig. 1. General view (*SHELXTL-Plus* graphic) of the asymmetric unit, showing the atom-numbering scheme. Anisotropic ellipsoids represent 50% probability boundaries.
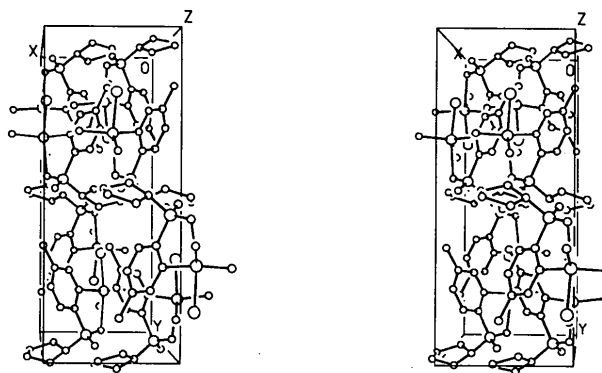

Fig. 2. Stereoscopic view (*SHELXTL-Plus* graphic) of the unit cell. H atoms are omitted for clarity.

Table 1. *Atomic coordinates and equivalent isotropic displacement parameters* ($\text{Å}^2 \times 10^4$)

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

	x	y	z	U_{eq}/U
Sn(1)	-0.00826 (9)	0.18764 (3)	0.06606 (2)	455
Cl(1)	-0.0311 (4)	0.3098 (1)	0.0298 (1)	759
S(1)	-0.1580 (3)	0.0521 (1)	0.15309 (9)	478
O(1)	-0.0341 (7)	0.0623 (3)	0.1063 (2)	589
O(2)	-0.0941 (9)	0.0224 (3)	0.2054 (3)	682
N(1)	-0.310 (1)	-0.0010 (3)	0.1302 (3)	506
C(1)	-0.035 (1)	0.1326 (5)	-0.0122 (3)	634
C(2)	0.238 (1)	0.1961 (6)	0.1091 (4)	703
C(3)	-0.214 (1)	0.1951 (6)	0.1301 (3)	371
C(4)	-0.292 (1)	0.2603 (4)	0.1425 (4)	457
C(5)	-0.406 (1)	0.2693 (4)	0.1887 (4)	524
C(6)	-0.435 (1)	0.2107 (5)	0.2246 (4)	530
C(7)	-0.359 (1)	0.1454 (4)	0.2137 (3)	527
C(8)	-0.249 (1)	0.1380 (4)	0.1666 (3)	392
C(9)	-0.435 (1)	-0.0352 (5)	0.1697 (4)	724
C(10)	-0.612 (2)	-0.0183 (8)	0.1473 (6)	1237
C(11)	-0.592 (2)	-0.0035 (7)	0.0853 (6)	927
C(12)	-0.404 (1)	0.0200 (6)	0.0767 (4)	694
C(13)	-0.494 (2)	0.3403 (5)	0.2003 (4)	907

Table 2. *Bond distances* (Å), *bond angles* ($°$), *torsion angles* ($°$), *least-squares planes* (Å) *and dihedral angles* ($°$)

Sn(1)—Cl(1)	2.438 (3)	C(3)—C(4)	1.38 (1)	
Sn(1)—O(1)	2.529 (6)	C(3)—C(8)	1.39 (1)	
Sn(1)—C(1)	2.113 (8)	C(4)—C(5)	1.39 (1)	
Sn(1)—C(2)	2.13 (1)	C(5)—C(6)	1.40 (1)	
Sn(1)—C(3)	2.171 (8)	C(5)—C(13)	1.51 (1)	
S(1)—O(1)	1.459 (6)	C(6)—C(7)	1.37 (1)	
S(1)—O(2)	1.431 (6)	C(7)—C(8)	1.39 (1)	
S(1)—N(1)	1.614 (7)	C(9)—C(10)	1.48 (2)	
S(1)—C(8)	1.773 (8)	C(10)—C(11)	1.49 (2)	
N(1)—C(9)	1.47 (1)	C(11)—C(12)	1.51 (2)	
N(1)—C(12)	1.49 (1)			

C(2)—Sn(1)—C(3)	107.4 (3)	S(1)—N(1)—C(9)	121.3 (6)
C(1)—Sn(1)—C(3)	124.1 (3)	C(9)—N(1)—C(12)	109.7 (7)
C(1)—Sn(1)—C(2)	122.2 (4)	Sn(1)—C(3)—C(8)	120.8 (6)
O(1)—Sn(1)—C(3)	75.3 (2)	Sn(1)—C(3)—C(4)	120.6 (6)
O(1)—Sn(1)—C(2)	87.7 (3)	C(4)—C(3)—C(8)	117.5 (7)
O(1)—Sn(1)—C(1)	82.4 (3)	C(3)—C(4)—C(5)	122.3 (7)
Cl(1)—Sn(1)—C(3)	97.5 (2)	C(4)—C(5)—C(13)	121.4 (8)
Cl(1)—Sn(1)—C(2)	99.1 (3)	C(4)—C(5)—C(6)	118.2 (8)
Cl(1)—Sn(1)—C(1)	98.3 (2)	C(6)—C(5)—C(13)	120.4 (8)
Cl(1)—Sn(1)—O(1)	171.4 (2)	C(5)—C(6)—C(7)	120.9 (8)
N(1)—S(1)—C(8)	109.6 (4)	C(6)—C(7)—C(8)	119.3 (8)
O(2)—S(1)—C(8)	109.2 (4)	C(3)—C(8)—C(7)	121.7 (7)
O(2)—S(1)—N(1)	106.9 (4)	S(1)—C(8)—C(7)	117.7 (6)
O(1)—S(1)—C(8)	105.5 (4)	S(1)—C(8)—C(3)	120.6 (6)
O(1)—S(1)—N(1)	107.0 (4)	N(1)—C(9)—C(10)	105.7 (9)
O(1)—S(1)—O(2)	118.5 (4)	C(9)—C(10)—C(11)	107 (1)
Sn(1)—O(1)—S(1)	116.8 (3)	C(10)—C(11)—C(12)	106 (1)
S(1)—N(1)—C(12)	117.3 (6)	N(1)—C(12)—C(11)	105.1 (8)

O(1)—Sn(1)—C(3)—C(8)	8.6 (6)	C(12)—N(1)—C(9)—C(10)	-14 (1)
C(3)—Sn(1)—O(1)—S(1)	-3.2 (4)	Sn(1)—C(3)—C(8)—S(1)	-12.8 (9)
C(8)—S(1)—O(1)—Sn(1)	-1.3 (4)	N(1)—C(9)—C(10)—C(11)	24 (1)
O(1)—S(1)—C(8)—C(3)	8.5 (8)	C(9)—C(10)—C(11)—C(12)	-23 (1)
C(9)—N(1)—C(12)—C(11)	0 (1)	C(10)—C(11)—C(12)—N(1)	15 (1)

No.	Plane through atoms	Equation of the plane (x along a, y in the plane ab, z along c^*)	χ^2
1	Sn(1), O(1), S(1), C(3), C(8)	$-0.712x - 0.218y - 0.667z = -1.751$	215.7
2	C(3), C(4), C(5), C(6), C(7), C(8)	$-0.784x - 0.258y - 0.564z = -1.39$	4.2
3	N(1), C(9), C(10), C(11), C(12)	$0.147x - 0.917y - 0.372z = -1.45$	337.1

Dihedral angles: 1,2 7.6 (2); 1,3 69.9 (3); 2,3 70.6 (4)

reflections with $4.5 \le 2\theta \le 50.0°$, $0 \le h \le 10$, $-23 \le k \le 23$, $-28 \le l \le 28$ were measured. The data were corrected for Lorentz–polarization and decay but not for absorption effects and averaged ($R_{int} = 0.017$) to 2938 unique reflections, 1652 of which had $F \ge 4.0\sigma(F)$. The systematic absences $(0kl)$ $k = 2n + 1$, $(h0l)$ $l = 2n + 1$, $(hk0)$ $h = 2n + 1$ conform to space group *Pbca*. The structure was solved *via* direct methods and $\Delta\rho$ maps. It was refined (on F) using full-matrix least squares with anisotropic displacement parameters for all non-H atoms and a common isotropic displacement parameter for the H atoms, which were placed in geometrically calculated positions (C—H 0.96 Å). 173 parameters were refined. Weights $w = 1.0/[\sigma^2(F) + 0.000245F^2)]$ led to a featureless analysis of variance in terms of $\sin\theta$ and F_o. The refinement converged to $S = 1.32$, $R = 0.047$, $wR = 0.040$, $(\Delta/\sigma)_{max} = 0.006$ (no extinction correction). The correctness of the space-group choice was checked by using *MISSYM* (Le Page, 1987). The largest peaks in the final $\Delta\rho$ map were ± 0.8 (2) e Å^{-3}. Atomic scattering factors for neutral atoms and real and imaginary dispersion terms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The programs used were *PARST* (Nardelli, 1983), *SHELXTL-Plus* (Sheldrick, 1987), *PCK83* (Williams, 1984), *PLATON* (Spek, 1982), *SADIAN* (Baur & Wenninger, 1969) and *MISSYM* (Le Page, 1987). The molecule and the numbering scheme are shown in Fig. 1 and a stereoscopic view of the unit-cell contents is in Fig. 2. Positional parameters and the equivalent values of the anisotropic displacement parameters for the non-H atoms are given in Table 1.* Bond lengths, bond angles, torsion angles, least-squares planes and dihedral angles are given in Table 2.

Related literature. The title compound was obtained from 3-(trimethylstannyl)toluene and *N*-pyrrolidyl-sulfonyl chloride with $AlCl_3$ as a catalyst at 263 K. The reaction mixture was hydrolyzed and the white precipitate washed with cold methanol. Instead of the normal reaction course a tin–methyl bond was split and no *ipso* substitution occurred, which can normally be observed (Al-Allaf, Kobs & Neumann, 1989; Preut, Wicenec & Neumann, 1991; Neumann & Wicenec, 1991).

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Bis(4-methylbenzenecyanamidosulfonato)dipyridinecopper(II)

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Abstract. $[Cu(C_8H_7N_2O_2S)_2(C_5H_5N)_2]$, $M_r = 612.2$, triclinic, $P\bar{1}$, $a = 7.150\,(4)$, $b = 9.314\,(5)$, $c = 10.523\,(7)$ Å, $\alpha = 93.55\,(5)$, $\beta = 97.15\,(5)$, $\gamma = 109.06\,(4)°$, $V = 653.3\,(7)$ Å^3, $Z = 1$, $D_x = 1.556$ Mg m^{-3}, Mo $K\bar{\alpha}$ radiation, $\lambda = 0.71069$ Å, $\mu = 1.04$ mm^{-1}, $F(000) = 315$, $T = 293\,(2)$ K, $R = 0.032$ for 2307 observed reflections. The Cu atom lies on a centre of symmetry and is octahedrally coordinated by four N and two O atoms. The Cu—N1, Cu—N2 and Cu—O1 distances are 2.010 (2), 1.936 (2) and 2.616 (4) Å, respectively. Twelve-membered rings are formed by pairs of bidentate cyanamidosulfonato groups which link adjacent Cu atoms through Cu–O–S(O)–N–C–N–Cu bridges into infinite chains running parallel to the *ac* plane.

Experimental. The title compound was prepared from the reaction of an aqueous solution of $Cu(NO_3)_2$ containing pyridine with an aqueous solution of $Na[4-CH_3C_6H_4SO_2NCN]$ (Köhler & Freude, 1991). Crystals were obtained immediately; m.p. 440 K. Well shaped blue crystal (0.5 × 0.3 × 0.1 mm) was mounted on a Syntex $P2_1$ diffractometer using graphite-monochromated Mo $K\bar{\alpha}$ radiation; θ–2θ-scan technique. Cell parameters by least squares on 15 reflections ($8 \le \theta \le 21°$). Empirical absorption correction based on reflection intensity measurements at different azimuthal angles, trans-mission range 0.828–1.002. Total 3271 reflections ($1.5 \le \theta \le 27.5°$) measured in the range $0 \le h \le 9$, $-12 \le k \le 11$, $-13 \le l \le 11$. No significant variation in the net intensities of two reference reflections ($2\bar{1}\bar{1}$, $11\bar{3}$) measured every 98 reflections. 3020 unique reflections ($R_{int} = 0.017$ for 336 reflections) and 2307 satisfied $F_o \ge 4\sigma(F)$. Structure was solved by the Patterson method (Sheldrick, 1990), full-matrix least-squares refinement of 227 parameters based on F (Sheldrick, 1976). Anisotropic thermal parameters for non-H atoms and isotropic for H atoms. At convergence $R = 0.0324$, $wR = 0.0366$, $w = 0.32/[\sigma^2(F) + 0.00060F^2]$, $(\Delta/\sigma)_{max} \le 0.033$, $(\Delta\rho)_{max} = 0.29$, $(\Delta\rho)_{min} = -0.25$ e Å^{-3}; isotropic extinction correction $F_c^* = F_c[1 - xF_c^2/\sin(\theta)]$, $x = 2.26\,(1) \times 10^{-6}$. Scattering factors for C, H, N, O and S given in *SHELX*76 (Sheldrick, 1976) and those for neutral Cu corrected for f' and f'' from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99, 149). All calculations on an EC-1045 computer system. Atomic parameters are given in Table 1,‡ selected interatomic parameters in Table 2 and

† Deceased.