

Dipyridinium diaquatetrachlorotin(IV)
1,5-naphthalenedisulfonateShan Gao,^a Li-Huo Huo,^a Hui
Zhao^a and Seik Weng Ng^{b*}^aCollege of Chemistry and Materials Science,
Heilongjiang University, Harbin 150080,
People's Republic of China, and ^bDepartment of
Chemistry, University of Malaya, Kuala Lumpur
50603, Malaysia

Correspondence e-mail: seikweng@um.edu.my

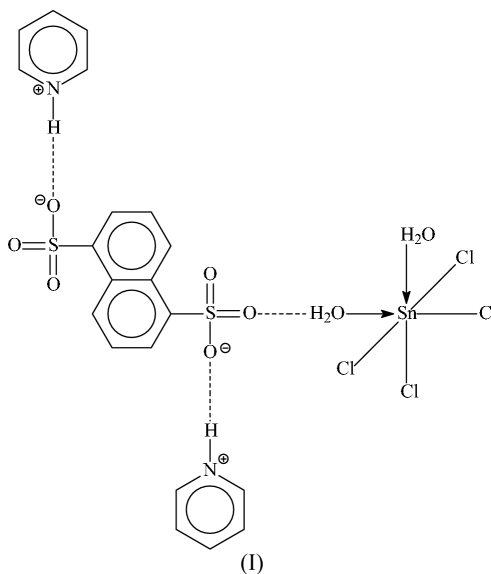
Key indicators

Single-crystal X-ray study
 $T = 295$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
Disorder in main residue
 R factor = 0.026
 wR factor = 0.074
Data-to-parameter ratio = 19.1For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

In the title compound, $(\text{C}_5\text{H}_6\text{N})_2[\text{SnCl}_4(\text{H}_2\text{O})_2](\text{C}_{10}\text{H}_6\text{O}_6\text{S}_2)$, the organic salt exists as a centrosymmetric ion triple in which each disordered cation is linked to the dianion by an $\text{N}\cdots\text{O}$ hydrogen bond. The six-coordinate neutral tin complex interacts with the salt through the coordinated water molecules, giving rise to a tightly bound three-dimensional network structure.

Comment

The arylsulfonate anion is capable of yielding crystalline metal complexes; the structural chemistry of metal arylsulfonates has been recently reviewed (Cai, 2004). For 1,5-naphthalenedisulfonate in particular, the synthesis of its metal derivative sometimes gives either an organic naphthalenedisulfonate without the metal (Gao *et al.*, 2005) or an aqua complex in which the dianion interacts indirectly with the metal atom through the coordinated water molecules (Cai *et al.*, 2001; Chen *et al.*, 2001).



Tin(IV) tetrachloride is a strong Lewis acid that affords a large number of adducts with O-atom donor ligands; among the ligands, water furnishes a small number of $\text{SnCl}_4 \cdot 2\text{H}_2\text{O}$ complexes. The ligands are aligned *cis* to each other in most of the complexes, which can be regarded as cocrystals (Azadmehr *et al.*, 2001; Cusack *et al.*, 1984; Hough *et al.*, 1986; Lorenzo *et al.*, 2001; Sobota *et al.*, 1996; Valle *et al.*, 1984); a *trans* alignment is noted for only one (Hough *et al.*, 1986), as found by searching the Cambridge Structural Database (Version 5.25; Allen, 2002). The *cis* alignment of the water donors is also noted in the cocrystal $2\text{C}_5\text{H}_6\text{N}^+ \cdots$

Received 4 January 2005

Accepted 6 January 2005

Online 15 January 2005

$C_{10}H_6O_6S_2^{2-} \cdot (H_2O)_2Cl_4Sn$, (I) (Fig. 1), which was formed in an attempt to synthesize a compound with a covalent tin–sulfonate linkage. The neutral $(H_2O)_2Cl_4Sn$ unit, which lies on a special position of site symmetry 2, interacts with the dianion, which lies on a center of inversion, *via* hydrogen bonds to form a three-dimensional network.

The pyridinium cation is disordered; it forms a relatively short hydrogen bond to one-half of the dianion. The distance is shorter than that found in the ion pair 4,4'-bipyridinium 1,5-naphthalenedisulfonate, which exists as a dihydrate. Its pyridinium N atom forms two hydrogen bonds, one to the sulfonate O atom [$O \cdots O = 2.885$ (2) Å] and the other to a water molecule [$O \cdots O = 2.810$ (3) Å] (Zhang *et al.*, 2004).

Experimental

Tin(IV) chloride pentahydrate (0.70 g, 2 mmol) was reacted with 1,5-naphthalenedisulfonic acid (0.66 g, 2 mmol) in water. Pyridine was placed over the water layer to induce the product to crystallize in the organic solvent and also to serve as a hydrogen chloride abstractor. Colorless crystals were isolated from this layer after several days. Analysis calculated for $C_{20}H_{22}Cl_4N_2O_8S_2Sn$: C 32.33, H 2.98, N 3.77%; found: C 32.01, H 3.31, N 3.71%.

Crystal data

$(C_5H_6N)_2[SnCl_4(H_2O)_2] \cdot (C_{10}H_6O_6S_2)$	$D_x = 1.821 \text{ Mg m}^{-3}$
$M_r = 743.01$	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 6001 reflections
$a = 20.517$ (3) Å	$\theta = 3.2\text{--}27.5^\circ$
$b = 11.689$ (2) Å	$\mu = 1.54 \text{ mm}^{-1}$
$c = 13.686$ (3) Å	$T = 295$ (2) K
$\beta = 124.33$ (3)°	Block, colorless
$V = 2710.3$ (9) Å ³	$0.28 \times 0.22 \times 0.16 \text{ mm}$
$Z = 4$	

Data collection

Rigaku R-AXIS RAPID IP diffractometer	3110 independent reflections
ω scans	3002 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	$R_{int} = 0.014$
$T_{min} = 0.521$, $T_{max} = 0.791$	$\theta_{max} = 27.5^\circ$
6020 measured reflections	$h = -26 \rightarrow 26$
	$k = 0 \rightarrow 15$
	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0468P)^2 + 3.8448P]$
$R[F^2 > 2\sigma(F^2)] = 0.026$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.074$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.01$	$\Delta\rho_{max} = 0.61 \text{ e \AA}^{-3}$
3110 reflections	$\Delta\rho_{min} = -0.48 \text{ e \AA}^{-3}$
163 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters (Å, °).

Sn1—Cl1	2.394 (1)	Sn1—O1w	2.135 (2)
Sn1—Cl2	2.375 (1)		
Cl1—Sn1—Cl1 ⁱ	170.10 (3)	Cl2—Sn1—Cl2 ⁱ	98.91 (4)
Cl1—Sn1—Cl2	90.53 (3)	Cl2—Sn1—O1w	169.79 (5)
Cl1—Sn1—Cl2 ⁱ	95.91 (3)	Cl2—Sn1—O1w ⁱ	90.59 (5)
Cl1—Sn1—O1w	84.76 (5)	O1w—Sn1—O1w ⁱ	80.19 (9)
Cl1—Sn1—O1w ⁱ	87.67 (5)		

Symmetry code: (i) $-x + 1, y, -z + \frac{1}{2}$.

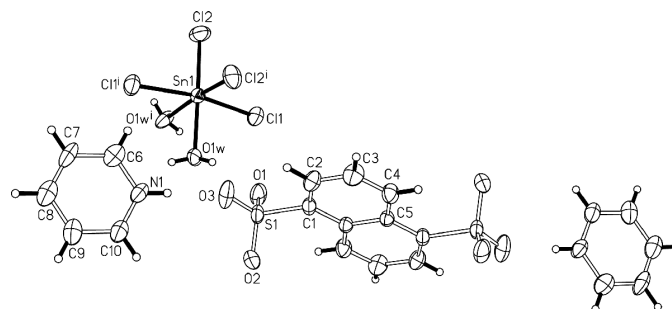


Figure 1

ORTEP (Johnson, 1976) plot of the formula unit of (I). Displacement ellipsoids are drawn at the 50% probability level, and H atoms are drawn as spheres of arbitrary radii. Only one component of each disordered pyridinium cation is shown. The symmetry code is that used in Table 1.

Table 2

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1w—H1w1 ⁱ ···O1	0.82	1.91	2.643 (2)	149
O1w—H1w2 ⁱ ···O2 ⁱⁱ	0.82	1.91	2.636 (2)	148
N1—H1 ⁱ ···O3	0.86	1.84	2.695 (3)	175
N1 ⁱ —H1 ⁱ ···O3	0.86	2.06	2.736 (4)	135

Symmetry code: (ii) $-x + 1, -y + 1, -z + 1$.

The pyridinium cation is disordered over two sites, and it was refined as two regular hexagons with 1.39 Å sides. The displacement parameters of the primed atoms were set equal to those of the corresponding unprimed atoms. As the disorder refined to nearly 0.5:0.5, the occupancies were subsequently fixed at 0.5 each. The two H atoms of atom O1w were rotated around the Sn1—O1w bond to fit the electron density. The aromatic and pyridinium H atoms were placed at calculated positions (C—H = 0.93 Å and N—H = 0.85 Å); all H atoms were included in the refinement in the riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}(\text{parent atom})$.

Data collection: *RAPID-AUTO* (Rigaku Corporation, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

The authors thank the National Natural Science Foundation of China (grant No. 20101003), the Scientific Fund for Remarkable Teachers of Heilongjiang Province (grant No. 1054 G036) and the University of Malaya for supporting this study.

References

- Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.
 Azadmehr, A., Amini, M. M., Tadjarodi, A., Taeb, A. & Ng, S. W. (2001). *Main Group Met. Chem.* **24**, 459–460.
 Cai, J. (2004). *Coord. Chem. Rev.* **248**, 1061–1083.
 Cai, J., Chen, C.-H., Liao, C.-Z., Yao, J.-H., Hu, X.-P. & Chen, X.-M. (2001). *J. Chem. Soc. Dalton Trans.* pp. 1137–1142.
 Chen, C.-H., Cai, J., Feng, X.-L. & Chen, X.-M. (2001). *J. Chem. Cryst.* **31**, 271–280.
 Cusack, P. A., Patel, B. N., Smith, P. J., Allen, D. W. & Nowell, I. W. (1984). *J. Chem. Soc. Dalton Trans.* pp. 1239–1243.
 Gao, S., Huo, L.-H. & Ng, S. W. (2005). *Acta Cryst.* **E61**, o98–o99.

- Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
- Hough, E., Nicholson, D. G. & Vasudevan, A. K. (1986). *J. Chem. Soc. Dalton Trans.* pp. 2335–2337.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Lorenzo, S., Day, A., Craig, D., Blanch, R., Arnold, A. & Dance, I. (2001). *CrystEngComm*, Paper No. 49.
- Rigaku Corporation (1998). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
- Rigaku/MSC (2002). *CrystalStructure*. Rigaku/MSI Inc., 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.
- Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
- Sobota, P., Szafert, S. & Glowiak, T. (1996). *J. Chem. Soc. Dalton Trans.* pp. 439–442.
- Valle, G., Cassol, A. & Russo, U. (1984). *Inorg. Chim. Acta*, **82**, 81–84.
- Zhang, X. L., Chen, X. M. & Ng, S. W. (2004). *Acta Cryst.* **E60**, o455–o456.