

C11—Sn—C12	105.4 (1)	C12—Sn—C11	91.2 (3)
C11—Sn—N1	94.4 (2)	N1—Sn—N2	73.0 (3)
C11—Sn—N2	167.4 (2)	N1—Sn—C10	90.7 (3)
C11—Sn—C10	86.9 (3)	N1—Sn—C11	88.6 (3)
C11—Sn—C11	90.1 (3)	N2—Sn—C10	93.2 (3)
C12—Sn—N1	160.2 (2)	N2—Sn—C11	89.4 (4)
C12—Sn—N2	87.2 (2)	C10—Sn—C11	176.9 (4)
C12—Sn—C10	90.5 (3)		

Table 2. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
N2—HN2A...C11 ⁱ	0.950	2.369	3.285 (9)	161.7
N2—HN2B...C12 ⁱⁱ	0.950	2.332	3.274 (8)	171.2

Symmetry codes: (i) $x, y, -1 + z$; (ii) $2 - x, 1 - y, 2 - z$.

H atoms of the aminoquinoline were kept fixed at calculated positions (C—H = 0.95 Å), those of the methyl groups were kept fixed at their observed positions. U_{iso} was set to be 20% larger than U_{eq} for the atom to which they were attached. The distances from Sn to the minimum and maximum values of $\Delta\rho$ are 0.94 and 1.22 Å, respectively.

Data collection: MAD (Allibon, 1995). Cell refinement: MAD. Data reduction: KRYSTAL (Hazell, 1995). Program(s) used to solve structure: SIR92 (Altomare *et al.*, 1994) and KRYSTAL. Program(s) used to refine structure: modified ORFLS (Busing, Martin & Levy, 1962). Molecular graphics: ORTEPIII (Burnett & Johnson, 1996) and KRYSTAL. Software used to prepare material for publication: KRYSTAL.

We are indebted to Nanyang Technological University (Grants No. RP18/96KLE and RG76/94) and to the Carlsberg Foundation and to the Danish Science Research Council for the diffractometer.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: AB1483). Services for accessing these data are described at the back of the journal.

References

- Allibon, J. (1995). *MAD* (Aarhus version). Institut Laue-Langevin, Grenoble, France.
- Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
- Bengtson, A., Goh, N. K., Hazell, A., Khoo, L. E., Ouyang, J. & Petersen, K. R. (1996). *Acta Chem. Scand.* **50**, 1020–1024.
- Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
- Busing, W. R., Martin, K. O. & Levy, H. A. (1962). *ORFLS*. Report ORNL-TM-305. Oak Ridge National Laboratory, Tennessee, USA.
- Crowe, A. J., Smith, P. J. & Atassi, G. (1984). *Inorg. Chim. Acta*, **93**, 179–184.
- Hall, V. J. & Tiekink, E. R. T. (1996). *Acta Cryst.* **C52**, 2143–2145.
- Hazell, A. (1995). *KRYSTAL. An Integrated System of Crystallographic Programs*. Aarhus University, Denmark.
- Hazell, A., Ouyang, J. & Khoo, L. E. (1997). *Acta Cryst.* **C53**, 406–408.
- Kabanos, T. A., Keramidis, A. D., Mentzafos, D., Russo, U., Tersis, A. & Tsangaris, J. M. (1992). *J. Chem. Soc. Dalton Trans.* pp. 2729–2733.
- Lockhart, T. P. & Manders, W. F. (1986). *Inorg. Chem.* **25**, 892–895.
- Petrosyan, V. S., Yashina, N. S. & Reutov, O. A. (1976). *Adv. Organomet. Chem.* **14**, 63–97.
- Saxena, A. K. & Huber, F. (1989). *Coord. Chem. Rev.* **95**, 109–123.
- Teoh, S.-H., Teo, S.-B., Lee, L.-K. & Fun, H.-K. (1994). *J. Coord. Chem.* **33**, 69–73.

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Chlorodimethyl(*N*-pyrrolidinecarbo-dithioato-*S,S'*)tin(IV)

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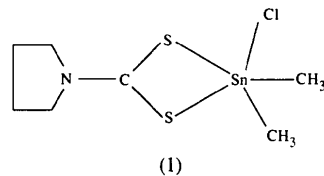
(Received 22 July 1996; accepted 27 March 1997)

Abstract

The Sn atom in [SnCl(C₅H₈NS₂)(CH₃)₂] is in a distorted trigonal bipyramidal environment, with the two methyl C atoms and an S atom in the equatorial plane, and the Cl and other S atom occupying axial positions. The carbodithioate ligand is unsymmetrically coordinated to the Sn atom with Sn—S distances of 2.7555 (10) and 2.4825 (12) Å.

Comment

Although a number of pentadentate diorganotin complexes of the type R_2ClSnS_2C have been reported, examples of structural studies are limited to Me₂SnCl(S₂CNMe₂), PhBuSnCl(S₂CNET₂), Ph₂SnCl(S₂CO'Pr) (Furue, Kimura, Yasuoka, Kasai & Masao, 1970; Wei, Kumar Das & Sinn, 1985; Dakternieks, Hoskins, Jackson, Tiekink & Winter, 1985). In these complexes, the Sn atom is asymmetrically bonded to the two S atoms with an average difference in the Sn—S bond lengths of 0.3 Å, similar to that observed in the title compound, Me₂ClSn[S₂CN(CH₂)₄], (1). However, the Sn—S distances in the related 1,2-dithio anionic complex 2-chloro-2,2-diphenyl-5-methyl-1,3,2-benzodithiastannole are similar (Sau, Day & Holmes, 1981). As in (1), the chloro group in all these complexes invariably occupies an axial position with the two organo groups in equatorial positions.



The displacement ellipsoid plot of (1) with the numbering scheme is shown in Fig. 1. The geometry about the Sn atom is a distorted trigonal bipyramid with the Cl and S1 atoms in axial positions and the two methyl C atoms and S2 in the equatorial plane. Despite

the less crowded environment about the Sn atom, the Me—Sn—Me angle in (1) [125.4(3)°] is smaller than those found in Me₂Sn[S₂CN(CH₂)₄]₂ (137.3°; Lockhart, Manders & Schlemper, 1985) and Me₂Sn(S₂CNMe₂)₂ (136°; Kimura, Yasuoka, Kasai & Kakudo, 1972), both of which are six-coordinate. The observed Me—Sn—Me and S—Sn—S angles are consistent with the presence of the more electronegative chloro group exerting its influence on the geometry. The crystal structure is further stabilized by intermolecular contacts involving centrosymmetrically related pairs of molecules [S1...S1 3.392(2) and S1...H7A 2.94(15) Å].

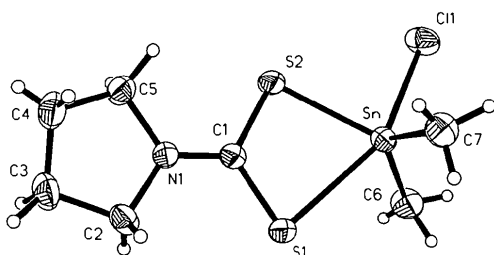


Fig. 1. A 30% probability displacement ellipsoid plot of (1) with the atomic numbering scheme.

Experimental

The title compound was prepared by reacting equimolar quantities of dimethyltin dichloride with ammonium pyrrolidinecarbodithioate in ethanol.

Crystal data

[SnCl(C₅H₈NS₂)(CH₃)₂]

M_r = 330.45

Triclinic

P $\bar{1}$

a = 6.422(1) Å

b = 10.129(3) Å

c = 10.176(1) Å

α = 85.60(1)°

β = 72.95(1)°

γ = 75.97(2)°

V = 614.0(2) Å³

Z = 2

D_x = 1.788 Mg m⁻³

D_m not measured

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 39 reflections

θ = 5.0–12.5°

μ = 2.593 mm⁻¹

T = 293(2) K

Rectangular slab

1.16 × 0.52 × 0.26 mm

Colourless

Data collection

Siemens P4 diffractometer

ω -2 θ scans

Absorption correction:

empirical ψ scans

(XSCANS; Siemens, 1994)

T_{min} = 0.273, *T_{max}* = 0.484

3572 measured reflections

2823 independent reflections

2689 reflections with

I > 2 σ (*I*)

R_{int} = 0.022

θ_{\max} = 27.5°

h = -1 → 8

k = -13 → 13

l = -13 → 13

3 standard reflections

every 97 reflections

intensity decay: <3%

Refinement

Refinement on *F*²

R[*F*² > 2 σ (*F*²)] = 0.032

wR(*F*²) = 0.085

S = 1.152

2823 reflections

165 parameters

All H atoms refined

w = 1/[$\sigma^2(F_o^2) + (0.0399P)^2 + 0.6691P$]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} < 0.001

$\Delta\rho_{\max}$ = 0.463 e Å⁻³

$\Delta\rho_{\min}$ = -0.901 e Å⁻³

Extinction correction: none

Scattering factors from

International Tables for Crystallography (Vol. C)

Table 1. Selected geometric parameters (Å, °)

Sn—C6	2.105(5)	N1—C1	1.309(5)
Sn—C7	2.110(5)	N1—C2	1.474(5)
Sn—Cl	2.4597(11)	N1—C5	1.476(5)
Sn—S2	2.4825(12)	C2—C3	1.513(7)
Sn—S1	2.7555(10)	C3—C4	1.492(9)
S1—C1	1.706(4)	C4—C5	1.518(7)
S2—C1	1.738(4)		
C6—Sn—C7	125.4(3)	C1—S2—Sn	90.58(12)
C6—Sn—Cl	95.7(2)	C1—N1—C2	123.5(3)
C7—Sn—Cl	99.8(2)	C1—N1—C5	124.7(3)
C6—Sn—S2	123.7(2)	C2—N1—C5	111.7(3)
C7—Sn—S2	109.0(2)	N1—C1—S1	122.3(3)
Cl—Sn—S2	87.54(4)	N1—C1—S2	119.6(3)
C6—Sn—S1	91.0(2)	S1—C1—S2	118.1(2)
C7—Sn—S1	96.1(2)	N1—C2—C3	103.7(4)
Cl—Sn—S1	154.48(4)	C4—C3—C2	105.8(4)
S2—Sn—S1	68.41(3)	C3—C4—C5	104.7(4)
C1—S1—Sn	82.47(12)	N1—C5—C4	103.1(4)

The title structure was solved by direct methods and refined by a full-matrix least-squares method on *F*². The H atoms were determined from the Fourier map and refined isotropically. PARST (Nardelli, 1983) was used for geometrical calculations.

Data collection: XSCANS (Siemens, 1994). Cell refinement: XSCANS. Data reduction: XSCANS. Program(s) used to solve structure: SHELXTL/PC (Sheldrick, 1990). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: SHELXTL/PC. Software used to prepare material for publication: SHELXL93.

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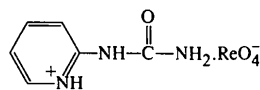
Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1288). Services for accessing these data are described at the back of the journal.

References

- Dakternieks, D., Hoskins, B. F., Jackson, P. A., Tiekink, E. R. T. & Winter, G. (1985). *Inorg. Chim. Acta*, **101**, 203–206.
- Furue, K., Kimura, T., Yasuoka, N., Kasai, N. & Masao, K. (1970). *Bull. Chem. Soc. Jpn.*, **43**, 1661–1667.
- Kimura, T., Yasuoka, N., Kasai, N. & Kakudo, M. (1972). *Bull. Chem. Soc. Jpn.*, **45**, 1649–1654.
- Lockhart, T. P., Manders, W. F. & Schlemper, E. O. (1985). *J. Am. Chem. Soc.*, **107**, 7451–7453.
- Nardelli, M. (1983). *Comput. Chem.*, **7**, 95–98.
- Sau, A. C., Day, R. O. & Holmes, R. R. (1981). *Inorg. Chem.*, **20**, 3076–3081.

- Sheldrick, G. M. (1990). *SHELXTLPC Users Manual*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (1993). *SHELXL93, Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
- Siemens (1994). *XSCANS, X-ray Single Crystal Analysis System*. Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Wei, C., Kumar Das, V. G. & Sinn, E. (1985). *Inorg. Chim. Acta*, **100**, 245–249.

protonation of the *endo*-N atom in the pyridyl ring favours the formation of an intramolecular N—H···O hydrogen bond which is crucial for the planarity and conformation of the molecule. The *N*-(pyridinium)urea cation is planar, with the largest deviation of 0.085 (9) Å at the amino N3 atom. The overall conformation of the molecule can be described as *s-cis-s-cis* with respect to the C1—N2—C2 imino bonds, in contrast to the *s-trans-s-cis* conformation found in *N*-(2-pyridyl)urea (Velikova, Angelova & Kossev, 1997).



(I)

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N-(2-Pyridinium)urea Perrhenate†

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Abstract

The *N*-(2-pyridinium)urea cation of the title compound, C₆H₈N₃O⁺·ReO₄⁻, is planar to within 0.085 (9) Å and adopts an *s-cis-s-cis* conformation with respect to the imino C—N bonds. The structure consists of columns of centrosymmetric dimeric plane-to-plane stacked cations and hydrogen-bonded anions. The distance between the anti-parallel carbonyl groups in the dimer is 3.16 (5) Å. An extensive network of hydrogen bonds stabilizes the ordering of the ions within the columns, which are held together by weaker hydrogen bonds and van der Waals interactions.

Comment

This work is part of an investigation of adducts of unsymmetrically substituted urea derivatives and inorganic salts with potential as non-linear optical materials. We combined *N*-(2-pyridyl)urea and perrhenic acid, assuming that the anionic host sub-network would affect the molecular geometry and arrangement of the cations.

Bond lengths and angles in the title compound, (I), compare well with the values found for other *N*-(2-pyridyl)urea derivatives (Velikova, Angelova & Kossev, 1997; Le Magueres, Ouahab, Hocquet & Fournier, 1994) and for ReO₄ tetrahedra (Lock & Turner, 1975; Macíček & Todorov, 1992; Macíček & Angelova, 1995). The

The structure contains centrosymmetric dimers of plane-to-plane stacked cations and hydrogen-bonded anions. The stacking of the cations embraces carbonyl groups which are only 3.16 (5) Å apart. In addition, the two coupled cations are captured by two ReO₄⁻ anions, forming a six-membered hydrogen-bonded ring through O12 and a relatively short, though bifurcated, hydrogen bond through O14 (Table 2). These dimers are ordered in columns along the *a* axis and interlinked by centrosymmetric eight-membered hydrogen-bonded rings formed by pairs of cations, similar to those in the structure of *N*-(2-pyridyl)urea, and by the HN31···O11 branches of the bifurcated hydrogen bond to HN31. The columns are held together by weak bifurcated hydrogen bonds [HN2···O12(1 - *x*, -*y*, 1 - *z*)] along the *b* axis, as well as by van der Waals interactions along the *c* axis.

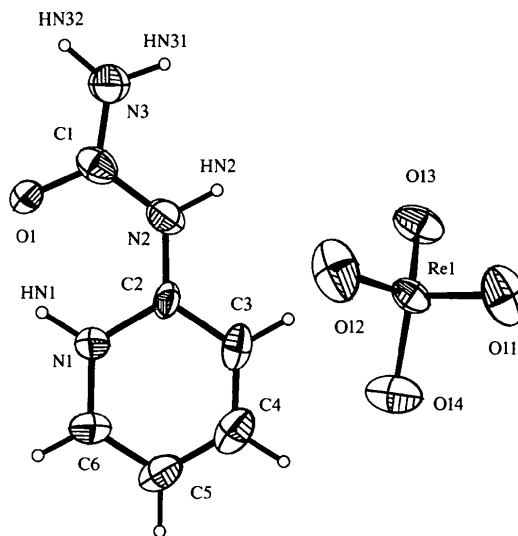


Fig. 1. The ions of the title compound, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are arbitrarily reduced.

† Alternative name: 2-ureidopyridinium tetraoxorhenate.