

C12	0.3513 (7)	0.1936 (6)	0.4050 (3)	0.0439 (14)
C13	0.4287 (8)	0.1981 (7)	0.4715 (4)	0.051 (2)
C14	0.3956 (9)	0.3137 (8)	0.5001 (4)	0.065 (2)
C15	0.2822 (13)	0.4258 (9)	0.4638 (6)	0.114 (4)
C16	0.2043 (11)	0.4252 (8)	0.3967 (5)	0.097 (3)
C21	-0.0475 (7)	0.4309 (6)	0.2784 (3)	0.0426 (14)
C22	-0.1515 (8)	0.3792 (7)	0.3299 (4)	0.055 (2)
C23	-0.2945 (9)	0.4642 (10)	0.3431 (5)	0.072 (2)
C24	-0.3362 (10)	0.6010 (11)	0.3042 (5)	0.083 (3)
C25	-0.2366 (11)	0.6560 (9)	0.2535 (5)	0.083 (3)
C26	-0.0917 (9)	0.5720 (7)	0.2404 (4)	0.064 (2)
C31	0.2563 (7)	0.4090 (5)	0.1990 (3)	0.0401 (14)
C32	0.4101 (7)	0.4017 (6)	0.2141 (4)	0.0475 (15)
C33	0.4989 (9)	0.4675 (7)	0.1572 (4)	0.060 (2)
C34	0.4255 (9)	0.5452 (7)	0.0858 (4)	0.063 (2)
C35	0.2723 (9)	0.5530 (7)	0.0688 (4)	0.057 (2)
C36	0.1883 (8)	0.4829 (6)	0.1242 (3)	0.048 (2)
C40	0.1368 (9)	-0.0887 (7)	0.1891 (5)	0.067 (2)
C41	0.0192 (13)	-0.0651 (9)	0.1258 (6)	0.110 (4)
C42	0.2441 (18)	-0.2119 (10)	0.2171 (10)	0.203 (9)

Table 2. Selected geometric parameters (\AA , $^\circ$)

Rh—N2	1.963 (5)	P1—C11	1.832 (6)
Rh—N4	1.972 (5)	N1—O1	1.315 (6)
Rh—N3	1.974 (5)	N2—O2	1.374 (7)
Rh—N1	1.990 (5)	N3—O3	1.316 (6)
Rh—C40	2.146 (6)	N4—O4	1.365 (6)
Rh—P1	2.489 (2)	C40—C42	1.346 (11)
P1—C31	1.830 (6)	C40—C41	1.468 (10)
P1—C21	1.833 (6)		
N2—Rh—N4	174.3 (2)	N3—Rh—P1	88.19 (14)
N2—Rh—N3	101.1 (2)	N1—Rh—P1	97.31 (14)
N4—Rh—N3	78.7 (2)	C40—Rh—P1	176.1 (2)
N2—Rh—N1	78.7 (2)	C31—P1—C21	107.0 (3)
N4—Rh—N1	100.9 (2)	C31—P1—C11	101.7 (3)
N3—Rh—N1	174.5 (2)	C21—P1—C11	100.2 (3)
N2—Rh—C40	85.9 (3)	C31—P1—Rh	108.8 (2)
N4—Rh—C40	88.4 (3)	C21—P1—Rh	115.3 (2)
N3—Rh—C40	88.0 (2)	C11—P1—Rh	122.3 (2)
N1—Rh—C40	86.6 (2)	C42—C40—C41	124.3 (7)
N2—Rh—P1	94.33 (15)	C42—C40—Rh	119.3 (6)
N4—Rh—P1	91.33 (14)	C41—C40—Rh	116.2 (5)

Intensities were corrected for Lorentz and polarization factors and absorption using XP21 (with a modified version of EMPABS) (Pavelčík, 1993). The structure was solved by heavy-atom methods with XFPS (Pavelčík, Rizzoli & Andreetti, 1990) and subsequent Fourier syntheses using SHELXL93 (Sheldrick, 1993). Anisotropic displacement parameters were refined for all non-H atoms. Highly anisotropic displacement parameters of atoms C41 and C42 indicate possible disorder of the isopropyl group orientation around the Rh—C40 bond, but splitting of C41 and C42 into two or more corresponding pairs did not yield better results. Both (O)H atoms were located from the difference Fourier map and refined with isotropic displacement parameters fixed at 0.06 \AA^2 . The (C)H atoms were apparent in the difference map, but they were included in the refinement as riding atoms in calculated positions with isotropic displacement parameters. The only exception were the isopropyl H atoms which were not included because of high anisotropic displacement parameters of C41 and C42 atoms. $\Delta\rho_{\text{max}} = 2.4 \text{ e \AA}^{-3}$ near Rh (1.57 \AA) allows no reasonable crystallochemical explanation and is considered to be false. Geometrical analysis was performed using PARST (Nardelli, 1983) and SHELXL93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1124). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tetraethylammonium Bis(isothiocyanato)-triphenylstannate

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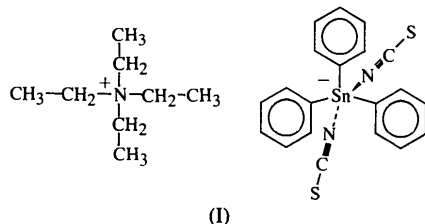
Abstract

The $[\text{Sn}(\text{C}_6\text{H}_5)_3(\text{NCS})_2]^-$ organotin anion of the title compound, $(\text{C}_8\text{H}_{20}\text{N})[\text{Sn}(\text{C}_6\text{H}_5)_3(\text{NCS})_2]$, lies on a crystallographic twofold axis and the trigonal bipyramidal

structure has NCS groups in axial and phenyl groups in equatorial positions. The tetraethylammonium cation is disordered over two orientations.

Comment

The title compound, $[N(C_2H_5)_4][Sn(C_6H_5)_3(NCS)_2]$, (I), was isolated on work-up of a reaction mixture derived from the sequential addition to 5,5'-(2-thioxo-1,3-dithide-4,5-diyl) bis(benzenecarbothiate), $[(PhCO)_2-C_3S_5]$, of (i) NaOMe and (ii) $Ph_2Sn(NCS)_2$ and NEt_4Br . The product was recrystallized from ethanol.



Atoms Sn, C2, C5 and H5 in the five-coordinate organotin anion lie on a twofold symmetry axis with the phenyl groups arranged equatorially and the thioisocyanato groups in axial positions. The angles about Sn in this trigonal bipyramidal structure are almost ideal; the C—Sn—C angles are 119.4 (3) and 120.28 (13)° [and 120.28 (13)°] and the N—Sn—N' angle is 179.6 (2)°. The NCS group is linear, with an N—C—S angle of 179.3 (5)° and an S··Sn··S' pseudo-angle of 173.72 (5)° (Fig. 1), indicating a slight bowing of the axial groups. Bond lengths are similar to those found in an identical anion complexed with 1-(salicylideneimino)-2-methoxybenzene (Charland, Gabe, Khoo & Smith, 1989). The structure of $[N(C_2H_5)_4][Sn(C_6H_5)_3Br_2]$ (Wharf & Simard, 1991) shows similar features, but has a slightly greater distortion from ideal geometry about the metal atom. The Sn—C distances of 2.135 (4)–2.142 (7) Å and the C—C phenyl bond lengths of 1.376 (8)–1.410 (7) Å are normal.

The tetraethylammonium cation is unusual as the N atom occupies a crystallographic inversion centre (as the cation does not possess inversion symmetry each of the atoms of the ethyl groups appears as two images related by inversion). Selecting one of these, with atom-occupancy factors of 0.5, enables the second orientation to be generated by the symmetry operation $(-\frac{1}{2}-x, \frac{3}{2}-y, 1-z)$. In $Na(Et_4N)[\{(CN)_2C_2S_2\}_3Sn]$ (Day, Holmes, Shafieezad, Chandrasekhar & Holmes, 1988), which also crystallizes in space group $C2/c$, the N atom lies on a crystallographic twofold axis and the atoms of the ethyl substituents are ordered since the molecular symmetry of the Et_4N^+ cation is compatible with the crystallographic site symmetry. As expected, uncertainties in molecular parameters and atomic displacement parameters are higher for the

disordered cation (especially for the methyl groups) compared to the anion. Refinement in the alternative space group Cc was not successful. The largest residual electron density was at the Sn position.

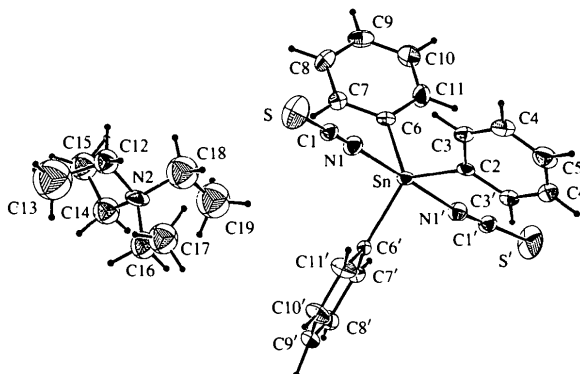


Fig. 1. The atomic arrangement in the title molecule. Displacement ellipsoids are shown at the 50% probability level. Primed atoms are related to their unprimed equivalents by the operation of the crystallographic twofold axis. Only one of the components of the disorder in the Et_4N^+ cation is shown.

Experimental

Crystal data

$(C_8H_{20}N)[Sn(C_6H_5)_3(NCS)_2]$
 $M_r = 596.40$
 Monoclinic
 $C2/c$
 $a = 15.273 (5) \text{ \AA}$
 $b = 9.931 (7) \text{ \AA}$
 $c = 19.1975 (15) \text{ \AA}$
 $\beta = 97.933 (12)^\circ$
 $V = 2884 (2) \text{ \AA}^3$
 $Z = 4$
 $D_x = 1.374 \text{ Mg m}^{-3}$
 D_m not measured

Mo $K\alpha$ radiation
 $\lambda = 0.71069 \text{ \AA}$
 Cell parameters from 250 reflections
 $\theta = 2.14\text{--}24.88^\circ$
 $\mu = 1.051 \text{ mm}^{-1}$
 $T = 150 (2) \text{ K}$
 Cube
 $0.22 \times 0.20 \times 0.20 \text{ mm}$
 Colourless

Data collection

Delft Instruments FAST diffractometer
 Area detector scans
 Absorption correction: none
 5976 measured reflections
 2152 independent reflections

1923 observed reflections
 $[I > 2\sigma(I)]$
 $R_{int} = 0.0804$
 $\theta_{max} = 24.88^\circ$
 $h = -17 \rightarrow 17$
 $k = -10 \rightarrow 9$
 $l = -21 \rightarrow 19$

Refinement

Refinement on F^2
 $R(F) = 0.0466$
 $wR(F^2) = 0.1109$
 $S = 1.032$
 2152 reflections
 160 parameters
 H atoms refined as riding model
 $w = 1/[\sigma^2(F_o^2) + (0.0657P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.05$
 $\Delta\rho_{max} = 1.49 \text{ e \AA}^{-3}$
 $\Delta\rho_{min} = -0.74 \text{ e \AA}^{-3}$
 Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

U_{iso} for C12–C19; $U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$ for all others.

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Sn	0	0.34721 (5)	1/4	0.0192 (2)
S	–0.08497 (14)	0.3195 (2)	0.49334 (9)	0.0596 (5)
N1	–0.0316 (3)	0.3479 (4)	0.3620 (2)	0.0276 (10)
C1	–0.0538 (3)	0.3369 (5)	0.4164 (3)	0.0253 (12)
C2	0	0.1315 (7)	1/4	0.021 (2)
C3	0.0138 (3)	0.0571 (5)	0.3125 (3)	0.0267 (12)
C4	0.0150 (3)	–0.0819 (6)	0.3130 (3)	0.0346 (13)
C5	0	–0.1512 (9)	1/4	0.043 (2)
C6	0.1190 (3)	0.4556 (5)	0.2838 (2)	0.0196 (10)
C7	0.1208 (3)	0.5559 (5)	0.3343 (2)	0.0251 (11)
C8	0.1997 (3)	0.6266 (5)	0.3569 (3)	0.0319 (13)
C9	0.2754 (3)	0.5959 (6)	0.3289 (3)	0.0337 (13)
C10	0.2740 (3)	0.4967 (6)	0.2784 (3)	0.0364 (14)
C11	0.1962 (3)	0.4284 (6)	0.2556 (3)	0.0344 (13)
N2	–1/4	3/4	1/2	0.038 (2)
C12†	–0.2432 (7)	0.7474 (10)	0.5806 (3)	0.045 (3)
C13†	–0.2986 (15)	0.860 (2)	0.6051 (8)	0.116 (9)
C14†	–0.2461 (7)	0.8920 (7)	0.4796 (6)	0.051 (3)
C15†	–0.1710 (9)	0.9723 (11)	0.5179 (7)	0.056 (4)
C16†	–0.3392 (5)	0.7006 (10)	0.4731 (6)	0.050 (3)
C17†	–0.3657 (9)	0.5682 (13)	0.5027 (8)	0.065 (4)
C18†	–0.1801 (7)	0.6679 (13)	0.4816 (6)	0.080 (5)
C19†	–0.1677 (13)	0.682 (2)	0.4028 (7)	0.086 (6)

† Partial occupancy (see *Comment*).

Table 2. Selected geometric parameters (\AA , $^\circ$)

Sn–C6	2.135 (4)	S–C1	1.622 (6)
Sn–C2	2.142 (7)	N1–C1	1.147 (7)
Sn–N1	2.268 (5)		
C6–Sn–C6 ⁱ	119.4 (3)	C2–Sn–N1	90.19 (11)
C6–Sn–C2	120.28 (13)	N1 ⁱ –Sn–N1	179.6 (2)
C6–Sn–N1 ⁱ	90.2 (2)	C1–N1–Sn	172.5 (4)
C6–Sn–N1	89.6 (2)	N1–C1–S	179.3 (5)

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

Data were corrected for Lorentz and polarization effects but not for absorption. The diffractometer was fitted with an Oxford Cryosystems low-temperature device (Cosier & Glazer, 1986). Absence of crystal decay in the X-ray beam was confirmed by checking equivalent reflections at the beginning and end of the data collection period of *ca* 8 h. All non-H atoms were refined with anisotropic displacement parameters, except the C atoms of the disordered ethyl substituents in the Et_2^+ cation which were refined isotropically with C–N and C–C bond lengths restrained to 1.47 (1) and 1.52 (1) \AA , respectively.

Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai, 1994).

The use of the EPSRC X-ray Crystallographic Service at the University of Wales, Cardiff, is gratefully acknowledged.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BM1025). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Chloro(ethylenediamine)(6-phenylimidazo[2,1-*b*]thiazole-*N*⁷)platinum(II) Nitrate

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Abstract

Platination of 6-phenylimidazo[2,1-*b*]thiazole at the imidazole N atom to give chloro(ethylenediamine)(6-phenylimidazo[2,1-*b*]thiazole)platinum(II) nitrate, $[\text{PtCl}(\text{C}_2\text{H}_8\text{N}_2)(\text{C}_{11}\text{H}_8\text{N}_2\text{S})]\text{NO}_3$, is accompanied by a rotation of 49.3 (8) $^\circ$ of the phenyl ring and a loss of extended conjugation in the normally planar 6-phenylimidazo[2,1-*b*]thiazole molecule.

Comment

Cisplatin, *cis*- $[\text{Pt}(\text{NH}_3)_2\text{Cl}_2]$, has long been used in the treatment of various forms of cancer (Loehrer & Einhorn, 1984). The reported activities of both platinum triamine complexes (Hollis, Amundsen & Stern, 1989) and platinum imidazole and thiazole compounds (van Beusichem & Farrell, 1992) in preliminary antitumor screens imply that additional structure–activity relationships for platinum-based therapeutic agents should be established. Therefore, we prepared a series of platinum triamine imidazothiazole complexes as part of a program to map out the relationships between structure and antitumor activity for this new class of potential chemotherapeutic agents (Arvanitis, Berardini, Parkinson & Schneider, 1993). The title compound, (I), is a member of that series.