

All non-H atoms were refined with anisotropic displacement parameters. H-atom positions were taken from difference maps. For all H atoms,  $U_{\text{iso}}(\text{H}) = 1.3U_{\text{eq}}(\text{C})$  of the parent atom and a riding model was used.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1993). Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SIMPEL* in *MolEN*. Program(s) used to refine structure: *LSFM* in *MolEN*. Molecular graphics: *MolEN* version of *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *MolEN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1131). Services for accessing these data are described at the back of the journal.

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*Acta Cryst.* (1998). **C54**, 1790–1791

## Di-*n*-butylbis(2-phenylquinazoline-4-thiolato-*N*<sup>3</sup>,*S*)tin(IV)

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## Abstract

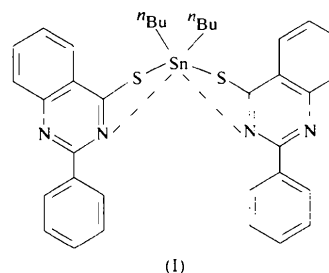
The central Sn atom in the title complex, [Sn(C<sub>4</sub>H<sub>9</sub>)<sub>2</sub>(C<sub>14</sub>H<sub>9</sub>N<sub>2</sub>S)<sub>2</sub>], is located on a twofold axis. The coordination polyhedron about the Sn atom is a skew-

trapezoidal bipyramid. The trapezoidal plane is formed by the two 2-phenylquinazoline-4-thiolate residues, which act as bidentate ligands with *cis*-Sn—S and *cis*-Sn—N bonds. The axial C atoms are shifted towards the half-sphere occupied by two N atoms, resulting in a C—Sn—C angle of 131.1(2)°. Each phenyl substituent is twisted with respect to the mean plane of the quinazoline system.

## Comment

For diorganotin(IV) bis-chelates, the coordination polyhedron around the Sn atom is either a regular *trans* or *cis* octahedron, or a distorted skew-trapezoidal bipyramid (STB), depending on the nature of the C- and heteroatom-donor ligands (Kumar Das *et al.*, 1987). Distortion from an octahedron to an STB is especially favoured if the chelate bite angle is small (Kepert, 1976, 1977).

The structure of the title compound, (I), consists of discrete molecules. The shortest intermolecular contact [3.504(7) Å] is N1...C16(−*x*, −*y*, 1 − *z*).



The coordination about Sn is skew-trapezoidal bipyramidal, with two S [Sn—S1 2.469(2) Å] and two N atoms of the chelating groups occupying the trapezoidal plane, and the butyl groups [Sn—C 2.107(4) Å] occupying the axial positions. The bond distance Sn—N3 [2.724(4) Å] is appreciably shorter than the sum of the appropriate van der Waals radii (3.75 Å; Bondi, 1964) and indicates the bidentate nature of the ligand. These bonds draw the quinazoline groups closer together, but result in severe steric repulsion between these groups (Kepert, 1976). Similar distances have been reported for Me<sub>2</sub>Sn(NO<sub>3</sub>)<sub>2</sub> (2.70 Å; Hilton *et al.*, 1973), Bu<sub>2</sub>Sn(2-SPy-5-NO<sub>2</sub>)<sub>2</sub> (2.77 Å; SPy is pyridinethiolate; Domazetis *et al.*, 1979), Me<sub>2</sub>Sn(2-SPy)<sub>2</sub> (2.70 Å; Castano *et al.*, 1990), Cyhex<sub>2</sub>Sn(2-SPy)<sub>2</sub> (2.72 Å; Cyhex is cyclohexyl; Boualam *et al.*, 1992) and Ph<sub>2</sub>Sn(2-SPy)<sub>2</sub> [2.634(4) and 2.698(4) Å; Schmiedgen *et al.*, 1993].

Each quinazoline ring adopts an almost planar conformation with maximum deviations from the mean planes through each half of the ring system exhibited by C5 [−0.005(6) Å] and C4 [0.017(5) Å]; the dihedral angle between the planes is 2.8(2)°. The phenyl ring is not

coplanar with the quinazoline moiety; the dihedral angle between the quinazoline system and the phenyl ring is 22.4 (2)°.

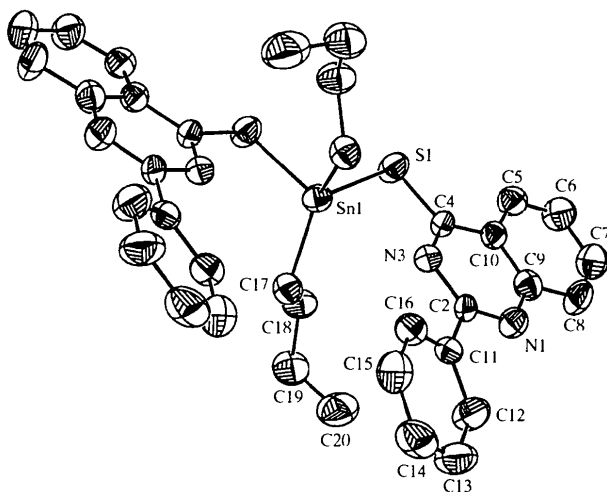


Fig. 1. View of the title molecule, showing the labelling of the non-H atoms. Displacement ellipsoids are shown at 50% probability levels and H atoms have been omitted for clarity.

## Experimental

The synthesis of (I) was carried out by reaction of dibutyltin dichloride and the sodium salt of 2-phenylquinazoline-4-thiol (molar ratio 1:2), which were stirred in methanol for 8 h at room temperature. Sodium chloride precipitate was removed and the solvent evaporated to afford a solid, which was recrystallized from acetone.

### Crystal data

[Sn(C<sub>4</sub>H<sub>9</sub>)<sub>2</sub>(C<sub>14</sub>H<sub>9</sub>N<sub>2</sub>S)<sub>2</sub>]

*M<sub>r</sub>* = 707.50

Monoclinic

*C*2/*c*

*a* = 22.112 (12) Å

*b* = 11.091 (8) Å

*c* = 15.208 (10) Å

β = 120.88 (5)°

*V* = 3201 (4) Å<sup>3</sup>

*Z* = 4

*D<sub>x</sub>* = 1.468 Mg m<sup>-3</sup>

*D<sub>m</sub>* = 1.45 (2) Mg m<sup>-3</sup>

*D<sub>m</sub>* measured by flotation in bromoform–hexane

### Data collection

Syntex P<sub>2</sub> diffractometer

θ/2θ scans

Absorption correction: none

3090 measured reflections

3090 independent reflections

1654 reflections with

*I* > 2σ(*I*)

Mo Kα radiation

λ = 0.71069 Å

Cell parameters from 15 reflections

θ = 6.1–13.4°

μ = 0.961 mm<sup>-1</sup>

*T* = 293 (2) K

Block

0.25 × 0.20 × 0.15 mm

Colourless

θ<sub>max</sub> = 26.04°

*h* = 0 → 24

*k* = 0 → 13

*l* = -17 → 16

2 standard reflections

frequency: 100 min

intensity decay: none

### Refinement

Refinement on *F*<sup>2</sup>

*R*(*F*) = 0.043

*wR*(*F*<sup>2</sup>) = 0.063

*S* = 1.043

3090 reflections

196 parameters

H atoms fixed

*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.0192*P*)<sup>2</sup>]

where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3

(Δ/σ)<sub>max</sub> = 0.001

Δρ<sub>max</sub> = 0.331 e Å<sup>-3</sup>

Δρ<sub>min</sub> = -0.363 e Å<sup>-3</sup>

Extinction correction:

*SHELXL93* (Sheldrick, 1993)

Extinction coefficient:

0.00015 (3)

Scattering factors from

*International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters (Å, °)

Sn1—C17	2.107 (4)	N3—C2	1.360 (5)
Sn1—S1	2.469 (2)	C2—C11	1.470 (6)
S1—C4	1.716 (4)	C4—C10	1.409 (5)
N1—C2	1.281 (5)	C17—C18	1.510 (5)
N1—C9	1.363 (5)	C18—C19	1.507 (6)
N3—C4	1.313 (5)	C19—C20	1.484 (6)
C17'—Sn1—C17	131.1 (2)	N1—C2—N3	126.3 (4)
C17'—Sn1—S1	105.22 (12)	N1—C2—C11	117.6 (4)
C17—Sn1—S1	109.61 (12)	C18—C17—Sn1	113.3 (3)
S1—Sn1—S1'	87.44 (8)	C19—C18—C17	113.7 (4)
C4—S1—Sn1	91.9 (2)	C20—C19—C18	113.9 (4)

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

Non-H atoms were refined anisotropically; atomic coordinates of all H atoms were calculated and their parameters were fixed.

Data collection: *P2<sub>1</sub> Software* (Syntex, 1973). Cell refinement: *P2<sub>1</sub> Software*. Data reduction: *XP21* (Pavelčík, 1987). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL93*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: KA1265). Services for accessing these data are described at the back of the journal.

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