PAULING, L. (1960). In *The Nature of the Chemical Bond*. New York: Cornell Univ. Press.
STOMBERG, R. (1984*a*). *Acta Chem. Scand. Ser. A*, **38**, 223–228.
STOMBERG, R. (1984*b*). *Acta Chem. Scand. Ser. A*, **38**, 541–545.
STOMBERG, R. (1986). *Acta Chem. Scand. Ser. A*, **40**, 168–176.
STOMBERG, R. (1988). *J. Crystallogr. Spectrosc. Res.* **18**, 659–669.
SZENTIVANYI, H. & STOMBERG, R. (1983). *Acta Chem. Scand. Ser. A*, **37**, 709–714.
VOLNOV, I. I. (1987). *Peroxo Complexes of Vanadium, Niobium, Tantalum*. Moscow: Nauka.

Acta Cryst. (1993). C**49**, 870–873

Structure of Bis{2′-[α-(2-pyridyl)benzylidene]salicylohydrazido}zinc(II)

BY H. SUR

Department of Physics, B. N. Mahavidyalaya, Itachuna, Hooghly-712 147, *West Bengal, India*

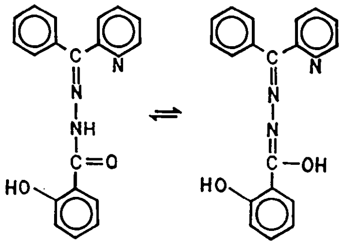
AND S. ROYCHOWDHURI AND S. SETH*

Department of Physics, X-ray Laboratory, Presidency College, College Street, Calcutta 700 073, *West Bengal, India*

(*Received* 17 *April* 1991; *accepted* 23 *November* 1992)

Abstract. $[Zn(C_{19}H_{14}N_3O_2)_2]$, $M_r = 698.06$, monoclinic, $P2_1/c$, $a = 13.460\,(5)$, $b = 9.632\,(3)$, $c = 24.667\,(2)$ Å, $\beta = 107.79\,(6)°$, $V = 3045\,(2)$ Å^3, $Z = 4$, $D_m = 1.54$ (by flotation in xylene/chloroform), $D_x = 1.523$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.877$ mm^{-1}, $F(000) = 1440$, $T = 294$ K, final $R = 0.039$, $wR = 0.039$ for 3626 observed diffractometer data. The two non-planar tridentate $C_{19}H_{14}N_3O_2$ ligands interact differently with the Zn atom. They are stabilized by intramolecular O—H···N hydrogen bonds. The Zn atom has a distorted octahedral environment consisting of two pyridine (py) N atoms [Zn—N(py) = 2.044 (4), 2.206 (4) Å], two azo N atoms [Zn—N(azo) = 2.046 (3), 2.078 (3) Å], a carbonyl (keto) O atom and an enolate O^- atom [Zn—O = 2.070 (4), 1.947 (3) Å]. The two N(py) atoms are *cis* [N(3)—Zn—N(4) = 86.9 (2)°] while the N(azo) atoms are *trans* [N(2)—Zn—N(5) = 164.9 (1)°].

Introduction. Several complexes of the tridentate ligand 2′-[α-(2-pyridyl)benzylidene]salicylohydrazide (pbsH) with transition and non-transition elements and also with lanthanides (Dutta & Hossain, 1984; Dutta & Das, 1984) have been synthesized. The neutral ligand pbsH may exist in tautomeric keto and enol forms. The present work was carried out to determine whether the structural features of the title compound are similar to those in the Ni and Sm complexes (Seth & Chakraborty, 1984; Dan, Seth & Chakraborty, 1989) and to discover the nature of the Zn—N bonding, the planarity of the ligand and the bonding within the molecule.



(*A*) (*B*)

Experimental. The title compound was synthesized by refluxing an aqueous solution of zinc acetate with an ethanolic solution of the Schiff-base ligand pbsH. Thin plate-shaped yellow crystals were obtained by slow evaporation of a solution in a mixture of acetone and alcohol at room temperature. Approximate lattice constants from rotation and Weissenberg photographs, accurate values by least-squares treatment of 25 randomly chosen reflections with $10.2 < \theta < 13.5°$, Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, crystal *ca* 0.30 × 0.37 × 0.42 mm; intensity data for $4.6 \leq 2\theta \leq 55.0°$, index range $-15 \leq h \leq 15$, $0 \leq k \leq 12$, $0 \leq l \leq 29$, ω–2θ scans; three standard reflections monitored periodically did not vary significantly during data collection; 5896 independent reflections were recorded, 3626 $[I \geq 3\sigma(I)]$ employed for the structure determination. Lorentz–polarization correction but no absorption correction. The approxi-

---

* To whom correspondence should be addressed.

0108-2701/93/050870-04$06.00

mate position of the Zn atom was obtained from an unsharpened three-dimensional Patterson map, the positions of remaining non-H atoms from successive weighted Fourier syntheses. Full-matrix least-squares refinement, anisotropic thermal parameters for C, N, O and Zn atoms; all H atoms included from difference Fourier map and kept fixed during refinement; each H atom given the isotropic temperature factor of its associated non-H atom. Examination of a difference map to assess whether water of crystallization was present confirmed the anhydrous nature of the crystals. Final $R = 0.039$ and $wR = 0.039$ for 3626 observed reflections only; maximum shift/e.s.d. = 0.39, function minimized was $\sum w(|F_o| - |F_c|)^2$, $w = [1/\sigma(F_o)^2]$ (from counting statistics). No correction for secondary extinction was attempted as $(|F_o| - |F_c|)$ values for strong low-order reflections were randomly positive and negative. Final difference map was featureless, with function values between 0.3 and $-0.4\,\text{e}\,\text{Å}^{-3}$. The thermal motion of the molecule is strongly anisotropic with U_{33} larger than the other components for most of the atoms. X-ray scattering factors and anomalous-dispersion corrections for Zn were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). All computations carried out using the Cyber computer of the Regional Computer Centre, Calcutta. Program system used was *XRAYARC* (Vickery, Bright & Mallinson, 1971) for Patterson synthesis. Normal *EXFFT* and *SEARCH* sequences of *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) were used for weighted Fourier syntheses and *SHELX*76 (Sheldrick, 1976) for refinement.

Table 1. *Final fractional atomic coordinates* ($\times 10^4$, *for* Zn $\times 10^5$) *and equivalent values of the anisotropic temperature factor coefficients* ($\text{Å}^2 \times 10^4$) *with e.s.d.'s in parentheses*

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

	x	y	z	U_{eq}
Zn	78049 (4)	21074 (5)	58049 (2)	419 (2)
O(1)	8592 (2)	506 (3)	6190 (1)	478 (12)
O(2)	11563 (2)	1124 (3)	7333 (1)	529 (11)
O(3)	6825 (2)	2226 (4)	6307 (1)	503 (11)
O(4)	3696 (2)	1331 (3)	5531 (1)	542 (12)
N(1)	10007 (3)	1848 (3)	6509 (1)	380 (12)
N(2)	9303 (3)	2729 (4)	6208 (1)	393 (12)
N(3)	7803 (3)	4186 (4)	5637 (2)	498 (15)
N(4)	8034 (3)	1772 (4)	4967 (2)	443 (15)
N(5)	6314 (2)	1865 (3)	5240 (1)	367 (13)
N(6)	5501 (2)	1965 (3)	5461 (1)	372 (11)
C(1)	11228 (4)	−2199 (5)	7543 (2)	525 (19)
C(2)	10284 (4)	−2453 (4)	7137 (2)	507 (18)
C(3)	9742 (3)	−1487 (4)	6800 (2)	429 (15)
C(4)	10142 (3)	−266 (4)	6850 (2)	349 (14)
C(5)	11107 (3)	−29 (4)	7261 (2)	399 (15)
C(6)	11641 (4)	−1000 (5)	7611 (2)	488 (17)
C(7)	9512 (3)	732 (4)	6485 (2)	360 (14)
C(8)	9616 (3)	3854 (4)	6137 (2)	424 (16)
C(9)	10703 (3)	4297 (4)	6352 (2)	458 (17)
C(10)	11205 (4)	4407 (5)	6930 (2)	573 (18)
C(11)	12227 (5)	4825 (6)	7120 (3)	761 (26)
C(12)	12746 (5)	5126 (6)	6743 (4)	928 (33)
C(13)	12257 (5)	5012 (7)	6169 (4)	918 (34)
C(14)	11239 (4)	4606 (6)	5973 (3)	690 (24)
C(15)	8748 (4)	4693 (4)	5816 (2)	451 (17)
C(16)	8905 (4)	5964 (5)	5715 (2)	603 (22)
C(17)	8046 (5)	6670 (5)	5415 (3)	749 (27)
C(18)	7074 (5)	6142 (6)	5243 (3)	727 (24)
C(19)	6977 (4)	4878 (5)	5364 (2)	627 (21)
C(20)	8928 (4)	1597 (5)	4852 (2)	565 (20)
C(21)	8979 (4)	1427 (6)	4310 (2)	630 (23)
C(22)	8066 (5)	1443 (6)	3865 (2)	637 (24)
C(23)	7133 (4)	1610 (5)	3979 (2)	536 (18)
C(24)	7137 (3)	1769 (4)	4536 (2)	410 (16)
C(25)	6173 (3)	1915 (4)	4701 (2)	369 (15)
C(26)	5150 (3)	2127 (5)	4267 (2)	407 (15)
C(27)	4347 (4)	1268 (5)	4179 (2)	510 (18)
C(28)	3396 (4)	1481 (6)	3773 (2)	627 (21)
C(29)	3226 (4)	2565 (6)	3459 (2)	675 (22)
C(30)	4009 (4)	3434 (6)	3544 (2)	667 (23)
C(31)	4971 (4)	3224 (5)	3943 (2)	551 (17)
C(32)	5879 (3)	2092 (4)	6034 (2)	406 (14)
C(33)	5092 (3)	2058 (4)	6338 (2)	454 (15)
C(34)	4063 (3)	1682 (4)	6084 (2)	484 (17)
C(35)	3370 (4)	1646 (5)	6403 (3)	636 (22)
C(36)	3710 (5)	1965 (6)	6971 (3)	789 (24)
C(37)	4719 (5)	2337 (7)	7226 (2)	823 (27)
C(38)	5403 (4)	2376 (6)	6912 (2)	653 (19)

Discussion. The final positional coordinates and equivalent anisotropic thermal parameters for the non-H atoms are given in Table 1.* Table 2 contains selected bond lengths and angles. Fig. 1 is an *ORTEP* (Johnson, 1976) drawing of the title compound. Of the two ligands coordinated to the Zn^{II} acceptor centre, one approximates more closely the deprotonated form of the keto tautomer (*A*) and is attached to Zn^{II} through its carbonyl (keto) O(3) atom, azo N(5) and pyridine N(4) atoms, while the second ligand resembles more closely the deprotonated enol form (*B*) coordinating through the deprotonated enolate O(1), azo N(2) and pyridine N(3) atoms. A distorted octahedral coordination around the Zn^{II} ion is formed by the two dissimilar ligands which are in *cis* positions. The *trans* Zn—N(azo) distances [2.046 (3), 2.078 (3) Å] are almost equal while the mutually *trans* Zn—O(3) and Zn—N(4) bonds to one of the ligands are each more than 0.1 Å longer than the corresponding Zn—O or Zn—N bonds to the other ligand, which has a greater contribution from the enol canonical form (*B*). The differences in the bonding to Zn of the two ligands is, therefore, related to the internal geometry of the complex and this effect was not observed in the Ni and Sm complexes (Seth & Chakraborty, 1984; Dan *et al.*, 1989). The relative shortness of Zn—N(azo) bonds compared to Zn—N(py) bonds in the keto form of the ligand may be due to the differing positions of the N(py) and N(azo) atoms in the octahedron and also to the considerable π-back-bonding power of the azo function or, alternatively,

---

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, selected torsion angles and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55811 (34 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU0262]

Table 2. *Selected interatomic distances* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

Zn—O(1)	1.947 (3)	Zn—N(3)	2.044 (4)
Zn—O(3)	2.070 (4)	Zn—N(4)	2.206 (4)
Zn—N(2)	2.046 (3)	Zn—N(5)	2.078 (3)
N(1)—N(2)	1.319 (4)	N(5)—N(6)	1.367 (5)
N(1)—C(7)	1.256 (5)	N(2)—C(8)	1.195 (6)
N(3)—C(15)	1.308 (6)	N(3)—C(19)	1.296 (6)
N(4)—C(20)	1.327 (7)	N(4)—C(24)	1.344 (5)
N(5)—C(25)	1.285 (6)	N(6)—C(32)	1.352 (5)
O(1)—C(7)	1.249 (4)	O(2)—C(5)	1.256 (5)
O(3)—C(32)	1.252 (4)	O(4)—C(34)	1.345 (6)
C(1)—C(2)	1.379 (6)	C(1)—C(6)	1.270 (7)
C(2)—C(3)	1.311 (6)	C(3)—C(4)	1.284 (6)
C(4)—C(5)	1.400 (5)	C(4)—C(7)	1.410 (5)
C(5)—C(6)	1.326 (6)	C(8)—C(9)	1.459 (6)
C(8)—C(15)	1.442 (6)	C(9)—C(10)	1.384 (7)
C(9)—C(14)	1.376 (9)	C(10)—C(11)	1.371 (8)
C(11)—C(12)	1.352 (12)	C(12)—C(13)	1.372 (12)
C(13)—C(14)	1.364 (9)	C(15)—C(16)	1.280 (7)
C(16)—C(17)	1.351 (8)	C(17)—C(18)	1.346 (9)
C(18)—C(19)	1.270 (8)	C(20)—C(21)	1.370 (8)
C(21)—C(22)	1.376 (7)	C(22)—C(23)	1.377 (9)
C(23)—C(24)	1.379 (7)	C(24)—C(25)	1.481 (7)
C(25)—C(26)	1.477 (5)	C(26)—C(27)	1.326 (6)
C(26)—C(31)	1.302 (6)	C(27)—C(28)	1.378 (6)
C(28)—C(29)	1.279 (8)	C(29)—C(30)	1.311 (8)
C(30)—C(31)	1.381 (7)	C(32)—C(33)	1.474 (7)
C(33)—C(34)	1.383 (6)	C(33)—C(38)	1.383 (6)
C(34)—C(35)	1.393 (8)	C(35)—C(36)	1.370 (9)
C(36)—C(37)	1.358 (8)	C(37)—C(38)	1.372 (9)
N(2)—Zn—N(3)	75.7 (2)	N(3)—Zn—O(3)	96.0 (2)
N(2)—Zn—N(4)	95.7 (2)	N(4)—Zn—N(5)	74.8 (2)
N(2)—Zn—N(5)	164.9 (1)	N(4)—Zn—O(1)	97.5 (1)
N(2)—Zn—O(1)	71.7 (2)	O(3)—Zn—N(4)	150.0 (2)
N(2)—Zn—O(3)	114.0 (1)	N(5)—Zn—O(1)	120.6 (1)
N(3)—Zn—N(4)	86.9 (2)	O(3)—Zn—N(5)	75.3 (2)
N(3)—Zn—N(5)	91.9 (2)	O(1)—Zn—O(3)	96.0 (1)
N(3)—Zn—O(1)	147.3 (2)	Zn—N(2)—N(1)	121.2 (3)
Zn—N(5)—N(6)	116.8 (3)		
N(2)—N(1)—C(7)	104.1 (4)	C(15)—N(3)—C(19)	125.1 (4)
N(1)—N(2)—C(8)	116.2 (4)	C(20)—N(4)—C(24)	119.1 (4)
N(6)—N(5)—C(25)	121.8 (4)	N(5)—N(6)—C(32)	109.4 (4)
C(2)—C(1)—C(6)	121.8 (5)	C(1)—C(2)—C(3)	123.0 (5)
C(2)—C(3)—C(4)	116.9 (5)	C(3)—C(4)—C(5)	119.2 (4)
C(3)—C(4)—C(7)	114.2 (4)	C(5)—C(4)—C(7)	126.5 (4)
O(2)—C(5)—C(4)	123.6 (4)	O(2)—C(5)—C(6)	112.8 (4)
C(4)—C(5)—C(6)	123.7 (4)	C(1)—C(6)—C(5)	115.2 (5)
N(1)—C(7)—O(1)	126.4 (4)	N(1)—C(7)—C(4)	110.3 (4)
O(1)—C(7)—C(4)	123.3 (4)	N(2)—C(8)—C(9)	124.9 (4)
N(2)—C(8)—C(15)	109.0 (4)	C(9)—C(8)—C(15)	126.1 (4)
C(8)—C(9)—C(10)	121.2 (5)	C(8)—C(9)—C(14)	119.5 (5)
C(10)—C(9)—C(14)	119.4 (5)	C(9)—C(10)—C(11)	119.9 (5)
C(10)—C(11)—C(12)	120.2 (7)	C(11)—C(12)—C(13)	120.5 (7)
C(12)—C(13)—C(14)	120.1 (7)	C(9)—C(14)—C(13)	120.0 (6)
N(3)—C(15)—C(8)	121.2 (4)	N(3)—C(15)—C(16)	119.6 (5)
C(8)—C(15)—C(16)	119.3 (5)	C(15)—C(16)—C(17)	115.1 (5)
C(16)—C(17)—C(18)	124.7 (5)	C(17)—C(18)—C(19)	116.7 (6)
N(3)—C(19)—C(18)	118.8 (6)	N(4)—C(20)—C(21)	122.8 (5)
C(20)—C(21)—C(22)	118.5 (6)	C(21)—C(22)—C(23)	119.1 (5)
C(22)—C(23)—C(24)	119.4 (5)	N(4)—C(24)—C(23)	121.1 (5)
N(4)—C(24)—C(25)	115.7 (4)	C(23)—C(24)—C(25)	123.2 (4)
N(5)—C(25)—C(24)	114.6 (4)	N(5)—C(25)—C(26)	124.5 (4)
C(24)—C(25)—C(26)	121.0 (4)	C(25)—C(26)—C(27)	124.4 (4)
C(25)—C(26)—C(31)	121.0 (4)	C(27)—C(26)—C(31)	114.6 (5)
C(26)—C(27)—C(28)	123.8 (5)	C(27)—C(28)—C(29)	121.1 (6)
C(28)—C(29)—C(30)	116.0 (6)	C(29)—C(30)—C(31)	123.6 (5)
C(26)—C(31)—C(30)	120.9 (5)	N(6)—C(32)—O(3)	124.6 (5)
N(6)—C(32)—C(33)	115.5 (4)	O(3)—C(32)—C(33)	120.0 (4)
C(32)—C(33)—C(34)	123.5 (4)	C(32)—C(33)—C(38)	118.5 (5)
C(34)—C(33)—C(38)	118.0 (5)	O(4)—C(34)—C(33)	122.1 (5)
O(4)—C(34)—C(35)	117.8 (5)	C(33)—C(34)—C(35)	120.1 (5)
C(34)—C(35)—C(36)	120.1 (6)	C(35)—C(36)—C(37)	120.4 (6)
C(36)—C(37)—C(38)	119.7 (6)	C(33)—C(38)—C(37)	121.8 (5)

due to constraints involved in chelate-ring formation with transition elements. Similar trends are observed in the Ni and Sm complexes and also in two isomers of dichlorobis(2-phenylazopyridine)ruthenium(II) (Seal & Ray, 1984). The double-bond character of the N—N and the adjacent C—N bonds is preserved in the enol form while it is significantly reduced in the keto form. Unequal distribution of strain in coordination polyhedra, resulting from steric conditions which give rise to some distorted angles, may influence both the tautomeric resonance and the bond lengths.

The four five-membered chelate rings and the individual ligands are non-planar. Each ligand, excluding its phenyl substituent, has two aromatic rings, pyridyl and salicyloyl, which are reasonably planar in the keto form and are almost planar to within ± 0.02 Å in the enol form. They are almost parallel to each other [dihedral angles 19.8 (1) and 18.1 (2)°] within the same ligand. The phenyl rings are strictly planar and are twisted out of the associated chelate rings [torsion angles N(2)—C(8)—C(9)—C(14) = 114.5 (6), N(5)—C(25)—C(26)—C(31) = 116.5 (6)°]. Owing to steric effects, a coplanar arrangement of the pyridyl and phenyl rings within the same ligand is impossible.

Non-bonded O···N distances [O(2)—N(1) = 2.530 (4), O(4)—N(6) = 2.561 (5) Å] indicate that each ligand is stabilized by a strong intramolecular O—H···N hydrogen bond. The molecules are packed in a series of layers parallel to the *ab* plane and are displaced with respect to one another to relieve short intermolecular contacts, so that packing forces are unlikely to influence the geometry of the molecule to any appreciable extent.

The authors thank Professor Siddhartha Ray, IACS, Jadavpur, Calcutta, for valuable discussions, Professor R. L. Dutta, Department of Chemistry, University of Burdwan, for providing the sample used in this investigation and the Regional Sophisticated Instrumentation Centre, Calcutta, for the diffractometer data collection. Financial assistance

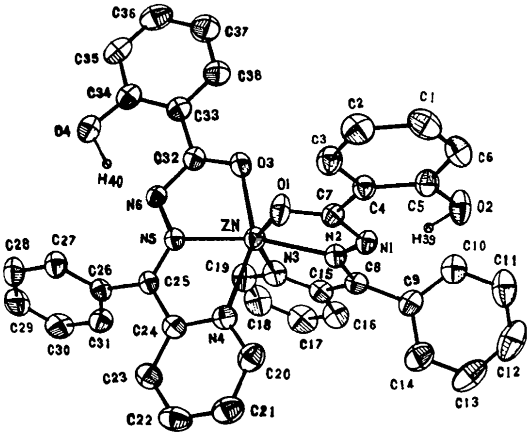


Fig. 1. An *ORTEP* (Johnson, 1976) drawing of the title compound showing the atom-numbering scheme.

from the University Grants Commission, New Delhi, is also gratefully acknowledged.

References

DAN, J., SETH, S. & CHAKRABORTY, S. (1989). *Acta Cryst.* C**45**, 1018–1021.

DUTTA, R. L. & DAS, B. R. (1984). *Indian J. Chem.* **23**A, 654–656.

DUTTA, R. L. & HOSSAIN, MD. M. (1984). *Indian J. Chem.* **23**A, 30–32.

JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

SEAL, A. & RAY, S. (1984). *Acta Cryst.* C**40**, 929–932.

SETH, S. & CHAKRABORTY, S. (1984). *Acta Cryst.* C**40**, 1530–1533.

SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

VICKERY, B. L., BRIGHT, D. & MALLINSON, P. R. (1971). *XRAYARC*. IBM 1130 program system, modified for a Cyber computer.

Acta Cryst. (1993). C**49**, 873–875

Structure of Di(n-butyl)bis(morpholinocarbodithioato-κ^2S,S')tin(IV)

BY V. VRÁBEL AND E. KELLÖ

Department of Analytical Chemistry, Faculty of Chemical Technology, Slovak Technical University,
*CS-*812 37 *Bratislava, Czechoslovakia*

(*Received* 14 *May* 1992; *accepted* 10 *December* 1992)

Abstract. $[\mathrm{Sn}(\mathrm{C_4H_9})_2(\mathrm{C_5H_8NOS_2})_2]$, $M_r = 557.41$, monoclinic, $C2/c$, $a = 17.152\,(14)$, $b = 7.146\,(5)$, $c = 20.995\,(28)$ Å, $\beta = 93.00\,(9)^\circ$, $V = 2570\,(4)$ Å^3, $Z = 4$, $D_m = 1.43\,(2)$, $D_x = 1.44$ Mg m^{-3}, $\lambda(\mathrm{Mo}\ K\alpha) = 0.71069$ Å, $\mu = 1.32$ mm^{-1}, $F(000) = 1144$, room temperature, $R = 0.057$ for 1895 unique observed reflections. The structure consists of discrete hexacoordinated tin complexes. The goemetry around the Sn atom is distorted octahedral with two anisobidentate dithiocarbamate-like (dtc) ligands in equatorial positions and two C atoms of the butyl groups in axial positions. The dtc ligand has a short Sn—S(1) distance of 2.525 (2) Å and longer Sn—S(2) of 3.001 (3) Å. The Bu—Sn—Bu angle is 139.7 (2)°.

Introduction. Great attention has been paid to the study of crystal structures of dtc complexes in the past few years. Interest has centred on their practical applications (Hagihara & Yamashita, 1966; Klug, 1966) and on the nature of the metal–ligand bonding. According to the X-ray studies the geometry about the Sn atom is strongly distorted from the *trans* octahedral one seen in diorganotin(IV)–dtc complexes (Morris & Schlemper, 1979*a*,*b*; Lockhart, Manders, Schlemper & Zuckerman, 1986; Lokaj, Kellö, Kettmann, Vrábel & Rattay, 1986). The largest deviations from octahedral geometry are due to the steric requirements of the four-membered chelate rings. In a continuing effort to understand the coordination chemistry of such complexes we have now determined the crystal structure of di(n-butyl)bis(morpholinocarbodithioato-κ^2S,S')tin(IV).

Experimental. The title compound was synthesized as reported previously (Vrábel, Lokaj, Kellö, Garaj, Batsanov & Struchkov, 1992). On recrystallization from n-heptane colourless needle-like crystals were obtained. Analysis found: N, 5.08; C, 38.89; H, 6.14%. Calculated: N, 5.03; C, 38.79; H, 6.15%. Density measured by flotation (in $\mathrm{ZnSO_4}$ solution). Data collected on a crystal of size 0.35 × 0.15 × 0.3 mm. Space group $C2/c$ from systematic absences: hkl, $h + k = 2n + 1$; $h0l$, $l = 2n + 1$; $0k0$, $k = 2n + 1$. Approximate values of lattice parameters from rotation and Weissenberg patterns. Accurate lattice parameters were refined by least squares on the basis of 15 precisely centred reflections with $4 < 2\theta < 11^\circ$ on a Syntex $P2_1$ four-circle diffractometer using Mo $K\alpha$ radiation filtered by a graphite monochromator. Intensities of 2959 unique reflections ($R_{\mathrm{int}} = 0.034$) collected in the range $2\theta < 55^\circ$, $h = 0$ to 22, $k = 0$ to 8, $l = -25$ to 26 by $\theta/2\theta$ scans, variable rate 4.9 to 29.3° min^{-1} in 2θ, background to scan time ratio 1.0, scan width 2° plus $\alpha_1 - \alpha_2$ dispersion. No significant variation in intensities of two standard reflections (202, 402) measured after 98 reflections. Data reduction performed with the program *XP*21 (Pavelčík, 1987). All intensities corrected for Lorentz and polarization effects but not for absorption or extinction. The structure was solved by the heavy-

0108-2701/93/050873-03$06.00