ENDRES, H. (1980*b*). *Acta Cryst.* B**36**, 1347–1350.
ENDRES, H. & SCHLICKSUPP, L. (1979). *Acta Cryst.* B**35**, 3035–3036.
ENDRES, H. & SCHLICKSUPP, L. (1980). *Acta Cryst.* B**36**, 715–716.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.
JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.
SHELDRICK, G. M. (1979). *SHELXTL*. An integrated system for solving, refining and displaying crystal structures from diffraction data. Univ. of Göttingen, Federal Republic of Germany.
STEWART, J. M., KUNDELL, F. A. & BALDWIN, J. C. (1970). The XRAY 70 system. Computer Science Center, Univ. of Maryland, College Park, Maryland.
TSCHUGAEFF, L. & SURENJANZ, J. (1907). *Chem. Ber.* **40**, 181–185.

Acta Cryst. (1980). B**36**, 2138–2139

Structure of Bis(*N*,*N*-dimethyldithiocarbamato)lead(II)

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Abstract. $[Pb\{S_2CN(CH_3)_2\}_2]$, $C_6H_{12}N_2PbS_4$, M_r = 447·6, orthorhombic, *Fdd*2, a = 21·073 (7), b = 27·422 (9), c = 4·214 (4) Å, U = 2435 Å^3, D_x = 2·44 Mg m^{-3}, μ(Mo $K\alpha$) = 16·7 mm^{-1}, Z = 8. The structure was refined to R = 0·045 by the block-diagonal least-squares method. The molecule has exact 2 symmetry. Four S atoms are coordinated to the Pb atom pyramidally, the two independent Pb—S distances being 2·779 (5) and 2·873 (6) Å.

Introduction. The crystal structure analysis of the title compound was undertaken as part of a series of studies on coordination compounds with metal–sulfur bonds. The compound was prepared by adding lead acetate to an aqueous solution of potassium *N*,*N*-dimethyldithiocarbamate. Pale-yellow needle crystals elongated along the *c* axis were grown from an acetone solution. They are extremely fragile and very easily cleaved into fibers.

A specimen of approximate dimensions 0·01 × 0·01 × 0·2 mm was mounted on a Rigaku four-circle automated diffractometer. Intensities of reflexions up to $2\theta \leq 55°$ were measured with Mo $K\alpha$ radiation monochromated by a graphite plate. The measurement was made in the ω–2θ scan mode with a scanning speed of 2° min^{-1} in 2θ, and 788 independent reflexions with values of $|F_o|$ larger than three times the standard deviation were obtained. The intensities were corrected for Lorentz, polarization and absorption effects.

The structure was solved by the heavy-atom technique, and refined by the block-diagonal least-squares method with the weighting scheme $w = 0{\cdot}5$ for $|F_o| < 80$, 1·0 for $80 \leq |F_o| < 250$ and $(250/F_o)^2$ for $|F_o| \geq 250$. The atomic form factors and the correction terms for anomalous scattering were taken from *International Tables for X-ray Crystallography* (1974). The final R value was 0·045 without H atoms.* All the calculations were performed on a Facom 230-75 computer of this Institute using the *UNICS* 3 program system (Sakurai & Kobayashi, 1979).

Discussion. The atomic coordinates are shown in Table 1, and bond lengths and angles are listed in Table 2. The crystals consist of monomeric molecules composed of one Pb atom and two chelating carbamate ligands. The Pb atom lies on a crystallographic twofold axis, so that the molecule has exact 2 symmetry. The two carbamate ligands are coordinated through S atoms to the metal atom pyramidally, and in each chelate ring one Pb—S bond is significantly shorter than the other. The Pb atom is approximately on the plane of the ligand, the deviation being 0·58 (3) Å. The dihedral angle between the two ligand planes is 38 (1)°. These features are similar to those observed in the crystals of $[Pb(S_2CNEt_2)_2]$ (Iwasaki & Hagihara, 1972), $[Pb\{S_2CN(iPr)_2\}_2]$ (*i*Pr = isopropyl; Ito & Iwasaki, 1980), and also of other dithioacid complexes of Pb (Hagihara & Yamashita, 1966; Hagihara, Watanabe & Yamashita, 1968; Ito, 1972), although the exact twofold symmetry of the present molecule is rather rare. The geometry within the ligand is normal.

The crystal structure is characterized by the stacking of molecules along the *c* axis (Fig. 1). Within this stack, each Pb atom is in contact at 3·361 (6) Å with two S

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35280 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Atomic parameters with estimated standard deviations in parentheses*

Positional parameters are multiplied by 10^4. B_{eq} is the equivalent isotropic temperature factor.

	x	*y*	*z*	B_{eq} (Å^2)
Pb	0	0	0 (9)	3·1
S(1)	1217 (2)	59 (1)	3051 (17)	4·6
S(2)	325 (2)	−767 (1)	3993 (11)	3·0
N	1537 (6)	−788 (5)	5634 (40)	3·3
C(1)	1082 (7)	−521 (5)	4354 (36)	2·7
C(2)	2168 (7)	−588 (6)	6158 (62)	4·3
C(3)	1419 (8)	−1286 (5)	6762 (46)	3·5

Table 2. *Interatomic distances* (Å) *and bond angles* (°) *with their standard deviations*

An asterisk denotes the atom at (−*x*,−*y*,*z*).

Pb–S(1)	2·873 (6)	S(1)–Pb–S(2)	63·3 (1)
Pb–S(2)	2·779 (5)	S(1)–Pb–S(1*)	126·8 (2)
S(1)–C(1)	1·707 (15)	S(1)–Pb–S(2*)	84·7 (2)
S(2)–C(1)	1·736 (15)	S(2)–Pb–S(2*)	105·5 (2)
C(1)–N	1·32 (2)	Pb–S(1)–C(1)	86·7 (5)
N–C(2)	1·46 (2)	Pb–S(2)–C(1)	89·2 (5)
N–C(3)	1·47 (2)	S(1)–C(1)–S(2)	119·1 (9)
S(1)···S(1*)	5·138 (7)	S(1)–C(1)–N	121·7 (11)
S(1)···S(2*)	3·806 (6)	S(2)–C(1)–N	119·2 (11)
S(2)···S(2*)	4·423 (6)	C(1)–N–C(2)	121·2 (13)
Pb···S(2, −*c*)	3·361 (6)	C(1)–N–C(3)	121·6 (13)
Pb···S(1, −*c*)	3·895 (7)	C(2)–N–C(3)	117·1 (14)

atoms of the succeeding molecule, and the six-coordination around the metal atom is completed. This rather strong intermolecular interaction results in the formation of a chain-like structure along the *c* axis. All the contacts between the chains are of van der Waals type. Such a structure is undoubtedly responsible for the crystal habit of the complex. The mode of molecular linkage in a chain is similar to that found in the crystals of [Pb(S_2CNEt_2)$_2$] (Iwasaki & Hagihara, 1972), except that the molecules in a chain are successively related by the operation of the lattice translation, whereas those in the ethyl analog are related by the operation of a glide plane. The mode of linkage in [Pb{$S_2CN(i\mathrm{Pr})_2$}$_2$] is somewhat different (Ito & Iwasaki, 1980).

Since the crystal is polar and intensities were collected only for reflexions with $l \geq 0$, an inverse structure in which the *z* parameters were reversed was examined. However, on refinement it gave a final *R* of 0·050, a significantly higher value than the 0·045 for the parameters given in Table 1. On the basis of an *R*-ratio test (Hamilton, 1965) and of a close check of individual reflexions, the atomic parameters reported in Table 1 were safely concluded to be correct. In accordance with Cruickshank & McDonald (1967), refinement with the incorrect absolute structure caused a shift of the Pb position by 0·12 Å along the *c* axis, and this led to a seriously incorrect determination of the molecular geometry: the difference in corresponding Pb–S distances in the two refinements, for example, amounted to as much as 0·07 Å, which is ten times each standard deviation (Iwasaki & Watanabe, 1980).

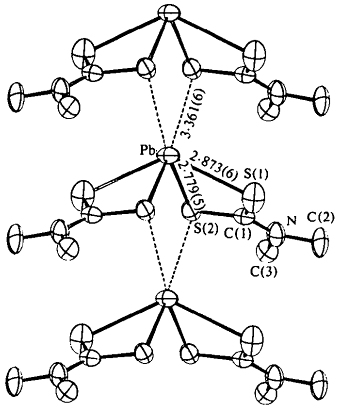

Fig. 1. The atomic numbering scheme and the molecular arrangement in a chain.

References

Cruickshank, D. W. J. & McDonald, W. S. (1967). *Acta Cryst.* **23**, 9–11.

Hagihara, H., Watanabe, Y. & Yamashita, S. (1968). *Acta Cryst.* B**24**, 960–966.

Hagihara, H. & Yamashita, S. (1966). *Acta Cryst.* **21**, 350–358.

Hamilton, W. C. (1965). *Acta Cryst.* **18**, 502–510.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 73–93, 149–150. Birmingham: Kynoch Press.

Ito, M. & Iwasaki, H. (1980). *Acta Cryst.* B**36**, 443–444.

Ito, T. (1972). *Acta Cryst.* B**28**, 1034–1049.

Iwasaki, H. & Hagihara, H. (1972). *Acta Cryst.* B**28**, 507–513.

Iwasaki, H. & Watanabe, Y. (1980). *Rikagaku Kenkyusho Hokoku*, **56**, 42–54.

Sakurai, T. & Kobayashi, K. (1979). *Rikagaku Kenkyusho Kokoku*, **55**, 69–77.