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Structure of Lead(II) *N*,*N*-Diisopropyldithiocarbamate [Bis(*N*,*N*-diisopropyldithiocarbamato)lead(II)]

BY MASAHISA ITO AND HITOSHI IWASAKI

The Institute of Physical and Chemical Research, Wako-shi, Saitama 351, *Japan*

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Abstract. $[Pb\{S_2CN(i\text{-}C_3H_7)_2\}_2]$, $C_{14}H_{28}N_2PbS_4$, monoclinic, $P2_1/a$, $a = 21{\cdot}055$ (3), $b = 8{\cdot}502$ (2), $c = 11{\cdot}720$ (2) Å, $\beta = 96{\cdot}15$ (3)°, $U = 2067{\cdot}7$ Å^3, $D_x = 1{\cdot}798$ Mg m^{-3}, μ(Mo $K\alpha$) = 9·84 mm^{-1}, $Z = 4$. The structure was refined to $R = 0{\cdot}066$ by the block-diagonal least-squares method. The molecules are monomeric with approximate 2 symmetry. The Pb atom is coordinated by four S atoms pyramidally at distances of 2·673 (4), 2·681 (4), 2·843 (5) and 2·859 (5) Å.

Introduction. The crystal structure analysis of lead(II) *N*,*N*-diisopropyldithiocarbamate 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 sodium *N*,*N*-diisopropyldithiocarbamate. By recrystallization from an ether solution, pillar-like yellowish crystals were obtained.

A specimen of approximate dimensions 0·16 × 0·16 × 0·25 mm was mounted on a Rigaku automated four-circle diffractometer. Intensities of the reflexions up to $2\theta \leq 55°$ were measured with Mo $K\alpha$ radiation monochromatized by a graphite plate. The measurements were made in the ω–2θ scan mode with a scanning speed of 4° min^{-1} in 2θ, and 2468 independent reflexions with values of $|F_o|$ greater than 3·5 times the standard deviation were obtained. The intensities were corrected for Lorentz and polarization factors and also for absorption.

The structure was solved by the heavy-atom method, and refined by the block-diagonal least-squares method with the weighting scheme $w = 0{\cdot}333$ for $|F_o| < 30$, 1·0 for $30 \leq |F_o| < 150$ and $(150/|F_o|)^2$ for $|F_o| \geq 150$. The atomic scattering factors and the correction terms for anomalous scattering were taken from *International Tables for X-ray Crystallography* (1974). The final R value was 0·066 without H atoms. The atomic coordinates are given in Table 1.*

Table 1. *Atomic parameters with estimated standard deviations in parentheses* (×10^4)

The B_{eq} values are the equivalent isotropic temperature factors (Å^2) (Hamilton, 1959).

	x	y	z	B_{eq}
Pb	2295 (1)	1543 (1)	4935 (1)	5·8
S(1)	1523 (2)	3793 (5)	3957 (3)	5·3
S(2)	2131 (2)	1594 (6)	2493 (4)	6·9
S(3)	1421 (2)	−773 (5)	4896 (3)	4·8
S(4)	1519 (2)	1519 (6)	6791 (4)	6·5
C(1)	1719 (6)	3326 (15)	2617 (11)	4·0
C(2)	1212 (6)	−204 (16)	6222 (11)	3·9
N(1)	1554 (6)	4268 (16)	1730 (10)	5·5
N(2)	799 (5)	−1057 (13)	6742 (10)	4·6
C(3)	1228 (8)	5777 (22)	1788 (23)	9·6
C(4)	1708 (11)	3770 (36)	541 (16)	11·6
C(5)	510 (7)	−2617 (17)	6381 (13)	5·1
C(6)	574 (9)	−452 (21)	7853 (12)	6·1
C(7)	1608 (9)	7183 (22)	2073 (17)	7·8
C(8)	534 (8)	5668 (25)	1773 (17)	7·6
C(9)	2269 (10)	4473 (31)	191 (19)	10·4
C(10)	1158 (13)	3031 (28)	−151 (20)	11·5
C(11)	1008 (8)	−3935 (18)	6438 (17)	6·4
C(12)	73 (8)	−2500 (24)	5227 (15)	6·6
C(13)	867 (10)	−1525 (26)	8854 (14)	7·8
C(14)	−159 (10)	−328 (29)	7782 (18)	8·9

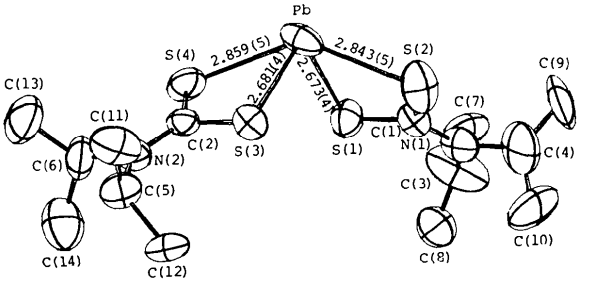

Fig. 1. A perspective view of the molecule, showing the Pb–S lengths (Å). The thermal ellipsoids are the 50% probability surfaces.

Discussion. The crystals of the title compound consist of monomeric molecules composed of one Pb atom and two chelating carbamate ligands. The bond

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34803 (17 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 2. *Interatomic distances* (Å) *and bond angles* (°)

Pb–S(1)	2·673 (4)	N(1)–C(4)	1·52 (3)
Pb–S(2)	2·843 (5)	N(2)–C(5)	1·49 (2)
Pb–S(3)	2·681 (4)	N(2)–C(6)	1·52 (2)
Pb–S(4)	2·859 (5)	C(3)–C(7)	1·45 (3)
S(1)–C(1)	1·714 (14)	C(3)–C(8)	1·46 (3)
S(2)–C(1)	1·717 (14)	C(4)–C(9)	1·42 (4)
S(3)–C(2)	1·729 (14)	C(4)–C(10)	1·47 (4)
S(4)–C(2)	1·700 (14)	C(5)–C(11)	1·53 (3)
C(1)–N(1)	1·32 (2)	C(5)–C(12)	1·55 (3)
C(2)–N(2)	1·33 (2)	C(6)–C(13)	1·55 (3)
N(1)–C(3)	1·46 (3)	C(6)–C(14)	1·54 (3)
S(1)–Pb–S(2)	63·9 (2)	C(1)–N(1)–C(4)	119·2 (15)
S(1)–Pb–S(3)	97·7 (2)	C(3)–N(1)–C(4)	115·8 (17)
S(1)–Pb–S(4)	87·8 (2)	C(2)–N(2)–C(5)	128·0 (12)
S(2)–Pb–S(3)	89·4 (2)	C(2)–N(2)–C(6)	119·3 (12)
S(2)–Pb–S(4)	138·6 (2)	C(5)–N(2)–C(6)	112·6 (12)
S(3)–Pb–S(4)	64·0 (2)	N(1)–C(3)–C(7)	118·8 (15)
Pb–S(1)–C(1)	91·8 (5)	N(1)–C(3)–C(8)	114·8 (16)
Pb–S(2)–C(1)	86·2 (5)	C(7)–C(3)–C(8)	125·3 (18)
Pb–S(3)–C(2)	91·3 (5)	N(1)–C(4)–C(9)	114·5 (19)
Pb–S(4)–C(2)	86·0 (5)	N(1)–C(4)–C(10)	112·8 (19)
S(1)–C(1)–S(2)	116·7 (8)	C(9)–C(4)–C(10)	130·0 (19)
S(3)–C(2)–S(4)	117·9 (8)	N(2)–C(5)–C(11)	112·4 (12)
S(1)–C(1)–N(1)	120·8 (11)	N(2)–C(5)–C(12)	111·9 (13)
S(2)–C(1)–N(1)	122·5 (11)	C(11)–C(5)–C(12)	114·9 (14)
S(3)–C(2)–N(2)	120·3 (10)	N(2)–C(6)–C(13)	108·3 (14)
S(4)–C(2)–N(2)	121·7 (10)	N(2)–C(6)–C(14)	112·4 (13)
C(1)–N(1)–C(3)	125·1 (15)	C(13)–C(6)–C(14)	112·9 (16)

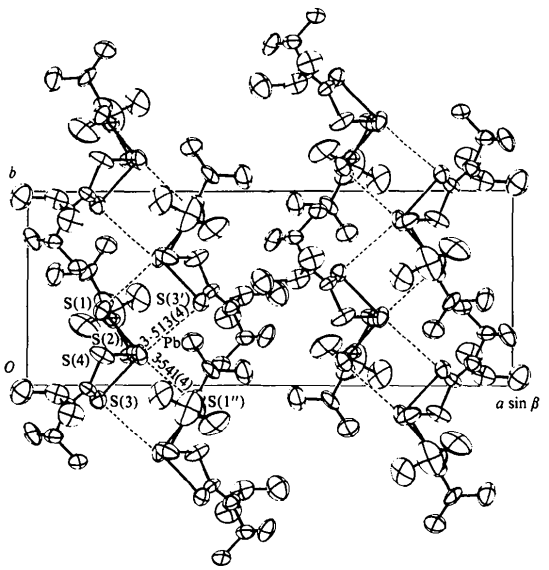

Fig. 2. The crystal structure projected along the *c* axis. The short intermolecular contacts (Å) are shown.

distances and angles are listed in Table 2. A perspective view of the molecule and the crystal structure projected along the *c* axis are shown in Figs. 1 and 2 respectively. The molecule has C_2 (2) symmetry to a good approximation. The Pb atom is coordinated by four S atoms at distances of 2·673 (4), 2·681 (4), 2·843 (5) and 2·859 (5) Å, and the configuration around the metal atom is a distorted pyramid. All bond lengths within the carbamate ligands are normal. As shown in Fig. 2, weak intermolecular Pb···S interactions of 3·513 (4) and 3·541 (4) Å occur between molecules related by a twofold screw axis. As a result, the molecules form a linear-chain structure along the *b* axis and complete an octahedral environment around the Pb atom. Among the six S atoms surrounding the metal atom, two belong to two different neighboring molecules, so that the direction of the linear chain is nearly parallel to the basal plane of the PbS_4 pyramid of the molecule. This mode of chain formation is different from that found in the ethyl analogue, $Pb(S_2CNEt_2)_2$ (Iwasaki & Hagihara, 1972), in which two of the six S atoms in the octahedron belong to one neighboring molecule and the direction of the chain is vertical with respect to the basal plane of the PbS_4 pyramid.

The calculations were performed on a FACOM 230-75 computer of this Institute, using the *UNICS* II program system (Sakurai, Iwasaki, Watanabe, Kobayashi, Bando & Nakamichi, 1974). Part of this work was supported by a Grant-in-Aid for Scientific Research No. 354161 from the Ministry of Education, Science and Culture.

References

HAMILTON, W. C. (1959). *Acta Cryst.* **12**, 609–610.

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

IWASAKI, H. & HAGIHARA, H. (1972). *Acta Cryst.* B**28**, 507–513.

SAKURAI, T., IWASAKI, H., WATANABE, Y., KOBAYASHI, K., BANDO, Y. & NAKAMICHI, Y. (1974). *Rep. Inst. Phys. Chem. Res.* **50**, 75–91.