

Dr Maria Rutkowska for the data collection, Doz Z. Gałdecki for his support, Professor W. Robert Scheidt whose computing facilities were used during refinement and finally to a referee for useful comments on the results reported in this paper. This work was financially supported by the Polish Academy of Sciences, project No. MR-I.9, and by NIH grant No. HL-15627 (to W. R. Scheidt), which are gratefully acknowledged.

#### References

- BAILEY, N. A., JENKINS, J. M., MASON, R. & SHAW, B. L. (1965). *J. Chem. Soc. Chem. Commun.* pp. 237–238.
- BARTCZAK, T. J. (1985). *Acta Cryst.* **C41**, 604–607.
- BATTEN, P., HAMILTON, A., JOHNSON, A. W., SHELTON, W. & WARD, D. (1974). *J. Chem. Soc. Chem. Commun.* pp. 550–551.
- CURRIE, M. & DUNITZ, J. D. (1971). *Helv. Chim. Acta*, **54**, 98–112.
- DEEMING, A. J., ROTHWELL, I. P., HURSTHOUSE, M. B. & BACKER-DIRKS, J. D. J. (1979). *J. Chem. Soc. Chem. Commun.* pp. 670–672.
- FLEISCHER, E. B., MILLER, C. K. & WEBB, L. E. (1964). *J. Am. Chem. Soc.* **86**, 2342–2347.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- JOHNSON, A. W., WARD, D., BATTEN, P., HAMILTON, A. L., SHELTON, G. & ELSON, C. M. (1975). *J. Chem. Soc. Perkin Trans. 1*, pp. 2076–2085.
- KONIECZY, A., BAILEY, P. M. & MAITLIS, P. M. (1975). *J. Chem. Soc. Chem. Commun.* pp. 78–79.
- ROBERTS, P. & SHELDRIK, G. M. (1975). *XANADU*. Program for crystallographic calculations. Univ. of Cambridge, England.
- SEMILETOV, S. A., BARANOVA, R. V., KHODYREV, YU. P. & IMANOV, R. M. (1980). *Kristallografiya*, **25**, 1162–1168.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
- STEFFEN, W. L. & PALENIK, G. J. (1976). *Inorg. Chem.* **15**, 2432–2439.

*Acta Cryst.* (1985). **C41**, 869–871

## Structure of (*N,N*-Dimethylbenzylamino-*N,C*)iodo(4-methoxyphenyl)-(4-methylphenyl)lead(IV), [PbI(C<sub>7</sub>H<sub>7</sub>)(C<sub>9</sub>H<sub>12</sub>N)(C<sub>7</sub>H<sub>7</sub>O)]

BY H. O. VAN DER KOOI, W. H. DEN BRINKER AND A. J. DE KOK\*

Department of Chemistry, Gorlaeus Laboratories, State University Leiden, PO Box 9502, 2300 RA Leiden, The Netherlands

(Received 18 July 1984; accepted 4 February 1985)

**Abstract.**  $M_r = 666.56$ , monoclinic,  $P2_1/n$ ,  $a = 10.994$  (3),  $b = 12.491$  (2),  $c = 16.715$  (4) Å,  $\beta = 94.87$  (2)°,  $V = 2287$  (1) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.936$  g cm<sup>-3</sup>,  $Mo K\alpha$ ,  $\lambda = 0.71073$  Å,  $\mu = 87.8$  cm<sup>-1</sup>,  $F(000) = 1256$ , room temperature, final  $R = 0.0283$  for 2129 significant reflexions. The lead atom is five-coordinated by iodine, nitrogen and three carbon atoms in a distorted trigonal bipyramidal geometry with bond lengths Pb–I 2.956 (1), Pb–N 2.685 (7), Pb–C 2.186 (7), 2.206 (7), 2.209 (8) Å. Iodine and nitrogen atoms are apical. The three carbon atoms are equatorial with the lead atom slightly out of their plane in the direction of the iodine atom.

**Introduction.** The title compound was the first chiral triorganolead compound synthesized (van der Kooi, Wolters & van der Gen, 1979). An X-ray structure determination was made to confirm the supposed geometry and to explain the PMR behaviour of the compound. Although this compound has much in

common with bromo(*N,N*-dimethylbenzylamino-*N,C*)diphenyltin bromide (van Koten, Noltes & Spek, 1976), the structures are not isomorphous. The synthesis of the title compound was described in the paper by van der Kooi *et al.* (1979). Initial results of the structure determination were mentioned in that paper. However, the lattice constants are corrected and the numerical values of distances and angles differ slightly from those in the previous paper. The differences are small and have no bearing on the conclusions given by van der Kooi *et al.* (1979) (*e.g.* the PMR behaviour).

**Experimental.** Crystal dimensions  $0.40 \times 0.15 \times 0.10$  mm; for source of material see van der Kooi *et al.* (1979); Enraf–Nonius CAD-4 four-circle diffractometer; graphite-monochromated  $Mo K\alpha$ ; cell constants from setting angles of 24 reflexions; absorption correction applied, transmission varying from 0.47 to 0.59;  $\theta_{max} = 30^\circ$ ; standard reflexions revealed loss in intensity of 16%, polynomial correction applied; overall e.s.d.'s of standard reflexions between 0.012 and 0.018; 7294 reflexions measured, of which 6593 independent,

\* To whom correspondence should be addressed.

2129 [ $I > 2\sigma(I)$ ] significant,  $R_{\text{int}} = 0.043$ , index range  $h -13/12$ ,  $k 0/16$ ,  $l 0/22$ . Structure solved by Patterson and Fourier methods, least-squares refinement on  $F$ ; phenyl H atoms placed at calculated positions and not refined; other H atoms, found in difference Fourier maps, refined with slack constraints (Waser, 1963). 286 parameters;  $R = 0.0283$ ,  $wR = 0.029$ ,  $w = \sigma(F)^{-2}$ ,  $S = 1.5$ . Max. LS shift 5%  $\sigma$  in last refinement cycle; max. and min. in difference-Fourier map 0.77 (19) and -0.44 (19) e Å<sup>-3</sup>; all significant peaks are near Pb and I; no extinction correction. Scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974); Leiden University computers IBM 370/158 and Amdahl V7B

Table 1. Fractional coordinates ( $\times 10^5$  for Pb and I,  $\times 10^4$  for all other atoms) and equivalent isotropic thermal parameters (Å<sup>2</sup>  $\times 10^4$  for Pb, Å<sup>2</sup>  $\times 10^3$  for I and Å<sup>2</sup>  $\times 10^2$  for all other atoms)

E.s.d.'s are given in parentheses.

$$B_{\text{eq}} = \frac{1}{3} \pi^2 \text{trace } \tilde{U}.$$

	x	y	z	B <sub>eq</sub>
Pb	-3085 (3)	24220 (2)	18349 (2)	5187 (9)
I	12986 (6)	15914 (6)	6470 (4)	738 (2)
N	-2122 (7)	3012 (6)	2721 (4)	60 (2)
O	1201 (7)	7307 (5)	1964 (5)	92 (3)
C(1)	-2055 (8)	1963 (6)	1172 (4)	48 (2)
C(2)	-3101 (9)	2400 (6)	1426 (5)	58 (3)
C(3)	-4217 (9)	2084 (8)	1072 (6)	69 (3)
C(4)	-4263 (10)	1352 (8)	457 (6)	69 (3)
C(5)	-3258 (11)	920 (6)	190 (5)	63 (3)
C(6)	-2113 (8)	1217 (6)	548 (4)	57 (3)
C(7)	-3037 (9)	3253 (8)	2074 (7)	72 (3)
C(8)	-2530 (11)	2099 (10)	3172 (6)	87 (4)
C(9)	-1930 (10)	3944 (9)	3264 (6)	89 (4)
C(10)	248 (8)	4102 (6)	1826 (5)	50 (2)
C(11)	-78 (8)	4761 (7)	1184 (4)	61 (3)
C(12)	245 (9)	5825 (7)	1189 (5)	67 (3)
C(13)	865 (8)	6242 (6)	1848 (6)	57 (3)
C(14)	1198 (9)	5617 (8)	2490 (6)	72 (3)
C(15)	891 (9)	4545 (7)	2477 (5)	69 (3)
C(16)	1096 (13)	7892 (10)	1298 (8)	100 (5)
C(17)	443 (7)	1424 (6)	2848 (5)	54 (2)
C(18)	703 (9)	1838 (6)	3597 (5)	66 (3)
C(19)	1158 (9)	1190 (8)	4225 (4)	70 (3)
C(20)	1370 (8)	130 (9)	4128 (6)	68 (3)
C(21)	1118 (8)	-278 (6)	3383 (6)	64 (3)
C(22)	662 (8)	364 (7)	2738 (5)	64 (3)
C(23)	1877 (9)	-560 (9)	4807 (6)	87 (4)

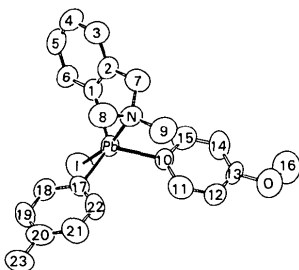


Fig. 1. Molecular structure with atomic numbering (ORTEP II, Johnson, 1965).

with programs written or modified by Mrs E. W. Rutten-Keulemans and Dr R. A. G. de Graaff were used.

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

Pb-I	2.956 (1)	C(10)-C(11)	1.38 (1)
Pb-N	2.685 (7)	C(11)-C(12)	1.38 (1)
Pb-C(1)	2.209 (8)	C(12)-C(13)	1.35 (1)
Pb-C(10)	2.186 (7)	C(13)-C(14)	1.35 (1)
Pb-C(17)	2.206 (7)	C(14)-C(15)	1.38 (1)
N-C(7)	1.44 (1)	C(15)-C(10)	1.36 (1)
N-C(8)	1.46 (1)	C(13)-O	1.39 (1)
N-C(9)	1.48 (1)	O-C(16)	1.33 (1)
C(7)-C(2)	1.52 (1)	C(17)-C(18)	1.36 (1)
C(1)-C(2)	1.37 (1)	C(18)-C(19)	1.39 (1)
C(2)-C(3)	1.37 (1)	C(19)-C(20)	1.36 (1)
C(3)-C(4)	1.37 (1)	C(20)-C(21)	1.35 (1)
C(4)-C(5)	1.34 (1)	C(21)-C(22)	1.40 (1)
C(5)-C(6)	1.40 (1)	C(22)-C(17)	1.36 (1)
C(6)-C(1)	1.40 (1)	C(20)-C(23)	1.49 (1)
I-Pb-N	168.8 (1)	C(4)-C(5)-C(6)	119.2 (7)
I-Pb-C(1)	96.6 (2)	C(5)-C(6)-C(1)	118.7 (8)
I-Pb-C(10)	98.5 (2)	C(6)-C(1)-C(2)	120.6 (8)
I-Pb-C(17)	96.4 (2)	Pb-C(10)-C(15)	120.7 (6)
C(1)-Pb-C(10)	118.5 (3)	Pb-C(10)-C(11)	121.7 (6)
C(10)-Pb-C(17)	117.4 (3)	C(16)-O-C(13)	114.0 (9)
C(1)-Pb-C(17)	119.4 (3)	O-C(13)-C(12)	126.4 (9)
N-Pb-C(1)	72.1 (3)	O-C(13)-C(14)	113.0 (8)
N-Pb-C(10)	87.8 (3)	C(10)-C(11)-C(12)	121.6 (7)
N-Pb-C(17)	88.8 (3)	C(11)-C(12)-C(13)	119.3 (8)
Pb-N-C(7)	98.5 (5)	C(12)-C(13)-C(14)	120.6 (8)
N-C(7)-C(2)	112.0 (7)	C(13)-C(14)-C(15)	121.7 (6)
C(7)-C(2)-C(1)	120.7 (8)	C(14)-C(15)-C(10)	121.1 (8)
C(2)-C(1)-Pb	117.2 (6)	C(15)-C(10)-C(11)	117.5 (7)
Pb-N-C(8)	110.2 (6)	Pb-C(17)-C(18)	121.7 (6)
Pb-N-C(9)	118.7 (6)	Pb-C(17)-C(22)	120.3 (6)
C(8)-N-C(9)	109.3 (7)	C(19)-C(20)-C(23)	121.9 (10)
C(7)-N-C(8)	109.0 (8)	C(21)-C(20)-C(23)	121.1 (10)
C(7)-N-C(9)	110.4 (7)	C(17)-C(18)-C(19)	120.5 (7)
Pb-C(1)-C(6)	122.1 (7)	C(18)-C(19)-C(20)	122.3 (8)
C(7)-C(2)-C(3)	119.6 (9)	C(19)-C(20)-C(21)	119.0 (9)
C(1)-C(2)-C(3)	119.6 (8)	C(20)-C(21)-C(22)	121.6 (8)
C(2)-C(3)-C(4)	119.2 (9)	C(21)-C(22)-C(17)	120.5 (7)
C(3)-C(4)-C(5)	122.6 (9)	C(22)-C(17)-C(18)	118.0 (7)

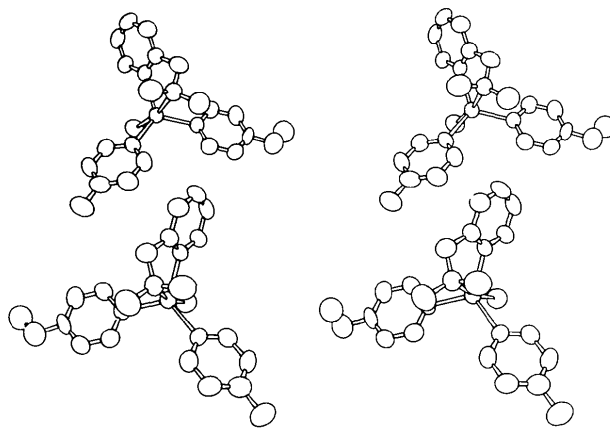


Fig. 2. A stereoview of one half of the unit cell (ORTEP, Johnson, 1965). The monoclinic  $b$  axis lies horizontally in the plane of the picture. The  $a$  axis is turned 30° out of this plane from the vertical position. The view direction is nearly [102].

**Discussion.** Positional parameters and isotropic  $B$  values are listed in Table 1;\* the numbering of the atoms is given in Fig. 1; intramolecular bond distances and valency angles are listed in Table 2. The Pb atom is five-coordinated in a distorted trigonal bipyramidal geometry, I and N being in apical and C(1), C(10) and C(17) in the equatorial positions. The Pb atom lies slightly [0.275 (5) Å] below the plane of these C atoms on the side of the I atom. The 4-methoxyphenyl and 4-methylphenyl groups make angles of 21.8 (3) and 44.8 (3)° respectively with the plane through C(1), C(10) and C(17); the benzylamine group is rotated out of this plane by 73.9 (2)° (Fig. 2).

All these features are also found in the tin compound mentioned with the exception of the 4-methylphenyl

\* Lists of structure amplitudes, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42040 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

C(1), C(10), C(17) dihedral angle, which is 8.3° in the tin compound. The I—Pb—N angle at 168.8 (1)° in the lead compound is slightly smaller than the Br—Sn—N angle (171.0°) in the tin compound.

The authors thank Mr S. Gorter for his assistance in the collection and processing of the diffraction data, Dr G. C. Verschoor for his useful advice and Dr J. Wolters for his stimulating interest in this study.

#### References

- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)  
 JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee.  
 KOOI, H. O. VAN DER, WOLTERS, J. & VAN DER GEN, A. (1979). *Recl Trav. Chim. Pays-Bas*, **98**, 353–355.  
 KOTEN, G. VAN, NOLTES, J. G. & SPEK, A. J. (1976). *J. Organomet. Chem.* **118**, 183–189.  
 WASER, J. (1963). *Acta Cryst.* **16**, 1091–1094.

*Acta Cryst.* (1985). **C41**, 871–873

## Structure of $\mu$ -Oxo-bis{[*N*-(2-mercaptoethyl)-*N,N'*-dimethylethylenediamino]-dioxomolybdenum(VI)}, [Mo<sub>2</sub>O<sub>5</sub>{(CH<sub>3</sub>)<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>NHCH<sub>2</sub>CH<sub>2</sub>S}<sub>2</sub>]

BY KASSU GEBREYES, SHAHID N. SHAIKH AND JON ZUBIETA\*

*Department of Chemistry, State University of Albany at New York, Albany, New York 12222, USA*

(Received 6 November 1984; accepted 4 February 1985)

**Abstract.**  $M_r = 565.9$ , monoclinic,  $C2/c$ ,  $a = 11.801$  (2),  $b = 8.521$  (2),  $c = 21.319$  (4) Å,  $\beta = 106.72$  (1)°,  $V = 2053.0$  (8) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.82$  g cm<sup>-3</sup>, Mo  $K\alpha$ ,  $\lambda = 0.71069$  Å,  $\mu = 14.22$  cm<sup>-1</sup>,  $F(000) = 1144$ , room temperature, final  $R = 0.055$  for 1060 reflections. The Mo atom displays distorted octahedral geometry with a facial stereochemistry for the bridging and terminal oxo groups. The angle at the bridging oxo group Mo—O(3)—Mo' is non-linear, 147.0 (5)°, possibly as a consequence of weak intramolecular hydrogen bonding between N(2)···O(2') and N(2')···O(2).

**Introduction.** Oxo complexes of Mo<sup>VI</sup> have been under intense study (Bruce, Corbin, Dahlstrom, Hyde, Minelli, Stiefel, Spence & Zubieta, 1982) as potential model systems for Mo sites in enzymes (Stiefel, 1977). Ligands containing S donors are most relevant as there is strong spectroscopic evidence for mercapto coordi-

nation to the enzymic Mo site. As part of a continuing investigation of the structural and synthetic chemistry of molybdenum–thiolate systems, we have been studying the chemistry of Mo–oxo moieties with tridentate ligands (Hyde, Magin & Zubieta, 1980; Hyde & Zubieta, 1982). Tridentate ligands are capable of stabilizing complexes with vacant coordination sites, as in [Mo<sub>2</sub>O<sub>3</sub>(SCH<sub>2</sub>CH<sub>2</sub>SCH<sub>2</sub>CH<sub>2</sub>S)<sub>2</sub>], which may potentially bind substrate or inhibitor molecules. Furthermore, our interest in the reactivity of the oxo functionality toward substituted hydrazines prompted the preparation and characterization of new Mo–oxo complexes as synthetic precursors. In this paper, we report the structure of one of these complexes, [Mo<sub>2</sub>O<sub>5</sub>(Me<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>NHCH<sub>2</sub>CH<sub>2</sub>S)<sub>2</sub>].

**Experimental.** *N*-(2-Mercaptoethyl)-*N,N'*-dimethylethylenediamine, (CH<sub>3</sub>)<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>NHCH<sub>2</sub>CH<sub>2</sub>SH, was prepared from the reaction of ethylene sulfide and *N,N*-dimethylethylenediamine in toluene (Karlín & Lippard, 1976). A solution of the ligand (30 mmol) in

\* To whom correspondence should be addressed.