shift/e.s.d. dropped below 0·01. The final difference map for the $P2_1/c$ structure had a largest peak of 0·8 (2) e Å^{-3}, 1·13 Å from Te(1). For the $I4_1/a$ case, the largest peak was 0·9 (2) e Å^{-3}, 0·89 Å fromTe(2). A summary of details for data acquisition and structure refinement is given in Table 1.

The programs used for data reduction and structure refinement were from the *NRCVAX* crystal structure system (Gabe, Larson, Lee & Le Page, 1984) and used scattering factors and anomalous-dispersion corrections for neutral atoms (*International Tables for X-ray Crystallography,* 1974). Computations were performed with a MicroVAXII computer. Diagrams were generated by the program *SNOOPI* (Davis, 1985). The final positional and equivalent isotropic thermal parameters for the non-H atoms are given in Table 2 ($P2_1/c$) and Table 3 ($I4_1/a$). Selected bond distances and angles are given in Table 4 ($P2_1/c$) and Table 5 ($I4_1/a$).* The molecular structures are shown in Fig. 1 ($P2_1/c$) and Fig. 2 ($I4_1/a$).

This work was supported by NSERC Operating Grants to CHWJ and FWBE.

References

Blecher, A. & Dräger, M. (1979). *Angew. Chem. Int. Ed. Engl.* **18**, 677–678.

Davis, E. K. (1985). *SNOOPI Plot Program.* Chemical Crystallography Laboratory, Univ. of Oxford, England.

Gabe, E. J., Larson, A. C., Lee, F. L. & Le Page, Y. (1984). *NRCVAX Crystal Structure System.* National Research Council, Ottawa, Canada.

Gay, I. D., Jones, C. H. W. & Sharma, R. D. (1989). *J. Magn. Reson.* In the press.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Larson, A. C. (1970). *Crystallographic Computing,* edited by F. R. Ahmed, p. 291. Copenhagen: Munksgaard.

Main, P., Woolfson, M. M. & Germain, G. (1971). *MULTAN. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Meulenaer, J. de & Tompa, H. (1965). *Acta Cryst.* **19**, 1014–1018.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.

---

* Tables of anisotropic thermal parameters, calculated H-atom coordinates and measured and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52073 (29 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1989). C**45**, 1815–1817

Nitratobis(triphenylphosphine)silver(I) Benzene Solvate (1/1)

By Cameron S. W. Harker and Edward R. T. Tiekink*

Jordan Laboratories, Department of Physical and Inorganic Chemistry, University of Adelaide, South Australia 5001, *Australia*

(*Received* 26 *May* 1989; *accepted* 19 *June* 1989)

Abstract. $[Ag(C_{18}H_{15}P)_2NO_3].C_6H_6$, $M_r = 772{\cdot}5$, triclinic, $P\bar{1}$, $a = 12{\cdot}279\,(1)$, $b = 16{\cdot}260\,(1)$, $c = 11{\cdot}134\,(1)$ Å, $\alpha = 102{\cdot}51\,(1)$, $\beta = 114{\cdot}28\,(1)$, $\gamma = 101{\cdot}79\,(1)°$, $U = 1868\,(1)$ Å^3, $Z = 2$, $D_x = 1{\cdot}373$ Mg m^{-3}, Mo $K\bar{\alpha}$ radiation, $\lambda = 0{\cdot}7107$ Å, $\mu = 0{\cdot}623$ mm^{-1}, $F(000) = 792$, $T = 295\,(2)$ K, $R = 0{\cdot}025$ for 5139 observed reflections. The Ag atom in the title compound is coordinated by two P atoms [Ag—P(1) 2·435 (1), Ag—P(2) 2·416 (1) Å, P(1)—Ag—P(2) 139·4 (1)°] and two O atoms derived from a weakly associated nitrate group [Ag—O(1) 2·463 (2), Ag—O(2) 2·572 (2) Å]. In the crystal lattice, layers of solvent benzene molecules are sandwiched between layers of complex molecules.

Experimental. The title compound was the major isolable product from the reaction of [Ag(adeninate)]NO_3 and triphenylphosphine (1/2) in refluxing $CHCl_3$ solution (Lobana, Bhatia & Tiekink, 1989). Crystals were obtained from benzene solution as a (1/1) benzene solvate; m.p. 394–395 K. Enraf–Nonius CAD-4F diffractometer controlled by a PDP8/A computer, graphite-monochromated Mo $K\bar{\alpha}$ radiation; $\omega{:}2\theta$ scan technique. Cell parameters on crystal $0{\cdot}44 \times 0{\cdot}31 \times 0{\cdot}65$ mm by least squares on 25 reflections ($8 \leq \theta \leq 12°$) (de Boer & Duisenberg, 1984). Analytical absorption correction (Sheldrick, 1976) applied; max. and min. transmission factors 0·838 and 0·727. Total of 7524 reflections ($1{\cdot}5 \leq \theta \leq 25{\cdot}0°$) measured in the range $-14 \leq h \leq 14$, $-19 \leq k \leq 19$, $-13 \leq l \leq 1$. No significant

---

* Author for correspondence.

variation in the net intensities of three reference reflections ($\bar{4}4\bar{3}$, $\bar{2}4\bar{3}$, $\bar{1}3\bar{4}$) measured every 7200 s. 6571 unique reflections ($R_{\rm int}$ 0·014) and 5139 satisfied $I \geq 2{\cdot}5\sigma(I)$. Structure solved by Patterson method, block-matrix least-squares refinement of 558 parameters based on F (Sheldrick, 1976). Anisotropic thermal parameters for non-H atoms and H atoms included in their calculated positions (C—H 0·97 Å). At convergence $R = 0{\cdot}025$, $wR = 0{\cdot}029$, $w = [\sigma^2(F) + 0{\cdot}0009F^2]^{-1}$, $S = 1{\cdot}12$, $(\Delta/\sigma)_{\rm max} \leq 0{\cdot}002$, $(\Delta\rho)_{\rm max} = 0{\cdot}37$, $(\Delta\rho)_{\rm min} = -0{\cdot}44$ e Å^{-3}; no extinction correction. Scattering factors for H, C, N, O and P given in *SHELX76* (Sheldrick, 1976) and those for neutral Ag corrected for f' and f'' (*International Tables for X-ray Crystallography*, 1974). All calculations on a SUN4/280 computer system. Atomic parameters are given in Table 1 and selected interatomic parameters in Table 2.* The numbering scheme used is shown in Fig. 1, which was drawn with *ORTEP*II (Johnson, 1971) at 25% probability ellipsoids.

Related literature. The Ag-atom geometry in the present compound is essentially as found in the unsolvated $[Ag(PPh_3)_2NO_3]$ complex with only minor differences in Ag–ligand parameters (Barron, Dyason, Healy, Engelhardt, Skelton & White, 1986). Notable in the present complex is the presence of a solvent benzene molecule of crystallization, Fig. 2, and the absence of an Ag—O′ intermolecular contact [3·075(3) Å] such as found in the unsolvated complex (Barron, Dyason, Healy, Engelhardt, Skelton & White, 1986).

Table 1. *Fractional atomic coordinates and B_{eq} values* (Å^2)

$$B_{\rm eq} = 8\pi^2(U_{11} + U_{22} + U_{33})/3.$$

	x	y	z	$B_{\rm eq}$
Ag	0·27566 (2)	0·24257 (1)	0·13002 (2)	4·02
P(1)	0·29858 (5)	0·14249 (4)	0·26612 (6)	3·45
P(2)	0·17605 (5)	0·35455 (3)	0·08833 (5)	3·25
N(1)	0·4846 (2)	0·2458 (1)	0·0675 (2)	4·38
O(1)	0·3718 (2)	0·2017 (1)	−0·0196 (2)	4·47
O(2)	0·5081 (2)	0·2960 (1)	0·1837 (2)	5·19
O(3)	0·5697 (2)	0·2415 (2)	0·0376 (3)	7·49
C(111)	0·1509 (2)	0·0641 (1)	0·2309 (2)	3·49
C(112)	0·1346 (3)	−0·0234 (2)	0·2246 (3)	5·20
C(113)	0·0211 (3)	−0·0774 (2)	0·2035 (4)	6·14
C(114)	−0·0764 (3)	−0·0445 (2)	0·1889 (3)	5·73
C(115)	−0·0615 (3)	0·0416 (2)	0·1954 (4)	6·91
C(116)	0·0516 (3)	0·0957 (2)	0·2141 (4)	5·92
C(121)	0·3701 (2)	0·2068 (1)	0·4512 (2)	3·57
C(122)	0·3264 (2)	0·1836 (2)	0·5393 (2)	4·37
C(123)	0·3876 (3)	0·2357 (2)	0·6806 (3)	5·82
C(124)	0·4904 (3)	0·3114 (2)	0·7305 (3)	5·20
C(125)	0·5332 (3)	0·3352 (2)	0·6439 (3)	4·78
C(126)	0·4746 (2)	0·2836 (2)	0·5042 (3)	4·31
C(131)	0·3981 (2)	0·0750 (2)	0·2573 (2)	4·12
C(132)	0·3811 (3)	0·0297 (2)	0·1277 (3)	5·18
C(133)	0·4489 (3)	−0·0268 (2)	0·1136 (4)	6·73
C(134)	0·5388 (3)	−0·0355 (2)	0·2305 (4)	7·83
C(135)	0·5594 (3)	0·0116 (2)	0·3599 (4)	7·28
C(136)	0·4904 (3)	0·0676 (2)	0·3760 (3)	5·57
C(211)	0·2278 (2)	0·4252 (1)	0·0022 (2)	3·19
C(212)	0·2935 (3)	0·3985 (2)	−0·0646 (3)	6·12
C(213)	0·3320 (3)	0·4497 (2)	−0·1335 (3)	7·45
C(214)	0·3054 (3)	0·5273 (2)	−0·1368 (3)	4·79
C(215)	0·2395 (3)	0·5539 (2)	−0·0717 (3)	4·76
C(216)	0·2006 (2)	0·5039 (2)	−0·0017 (3)	4·41
C(221)	0·0042 (2)	0·3085 (1)	−0·0188 (2)	3·32
C(222)	−0·0570 (2)	0·3070 (2)	−0·1551 (3)	4·33
C(223)	−0·1873 (3)	0·2640 (2)	−0·2360 (3)	5·10
C(224)	−0·2543 (2)	0·2232 (2)	−0·1796 (4)	5·10
C(225)	−0·1954 (3)	0·2252 (2)	−0·0445 (3)	5·25
C(226)	−0·0650 (2)	0·2676 (2)	0·0362 (3)	4·34
C(231)	0·2007 (2)	0·4310 (1)	0·2504 (2)	3·45
C(232)	0·1238 (2)	0·4818 (2)	0·2556 (3)	4·20
C(233)	0·1507 (3)	0·5420 (2)	0·3814 (3)	5·15
C(234)	0·2549 (3)	0·5515 (2)	0·5028 (3)	5·44
C(235)	0·3303 (3)	0·5011 (2)	0·5000 (3)	5·08
C(236)	0·3046 (2)	0·4406 (2)	0·3751 (3)	4·45
C(311)	0·1459 (6)	0·7242 (4)	0·6602 (4)	9·28
C(312)	0·0184 (7)	0·6793 (4)	0·5899 (7)	11·46
C(313)	−0·0565 (5)	0·7074 (5)	0·4929 (6)	10·14
C(314)	−0·0107 (7)	0·7778 (6)	0·4655 (6)	13·22
C(315)	0·1143 (7)	0·8184 (4)	0·5299 (7)	11·58
C(316)	0·1916 (5)	0·7933 (4)	0·6294 (5)	8·92

Table 2. *Selected interatomic distances* (Å) *and bond angles* (°)

Ag—P(1)	2·435 (1)	Ag—P(2)	2·416 (1)
Ag—O(1)	2·463 (2)	Ag—O(2)	2·572 (2)
P(1)—C(111)	1·822 (2)	P(2)—C(211)	1·823 (3)
P(1)—C(121)	1·821 (2)	P(2)—C(221)	1·826 (2)
P(1)—C(131)	1·818 (3)	P(2)—C(231)	1·821 (2)
N(1)—O(1)	1·249 (2)	N(1)—O(2)	1·249 (3)
N(1)—O(3)	1·230 (4)		
P(1)—Ag—P(2)	139·4 (1)	P(1)—Ag—O(1)	105·4 (1)
P(1)—Ag—O(2)	96·7 (1)	P(2)—Ag—O(1)	114·9 (1)
P(2)—Ag—O(2)	112·1 (1)	O(1)—Ag—O(2)	50·4 (1)
Ag—P(1)—C(111)	115·3 (1)	Ag—P(2)—C(211)	116·2 (1)
Ag—P(1)—C(121)	109·7 (1)	Ag—P(2)—C(221)	113·5 (1)
Ag—P(1)—C(131)	117·2 (1)	Ag—P(2)—C(231)	111·8 (1)
C(111)—P(1)—C(121)	103·6 (1)	C(211)—P(2)—C(221)	104·7 (1)
C(111)—P(1)—C(131)	105·1 (1)	C(211)—P(2)—C(231)	105·5 (1)
C(121)—P(1)—C(131)	104·5 (1)	C(221)—P(2)—C(231)	104·1 (1)
Ag—O(1)—N(1)	98·1 (2)	Ag—O(2)—N(1)	92·9 (1)
O(1)—N(1)—O(2)	118·4 (3)	O(1)—N(1)—O(3)	120·5 (2)
O(2)—N(1)—O(3)	121·1 (2)		

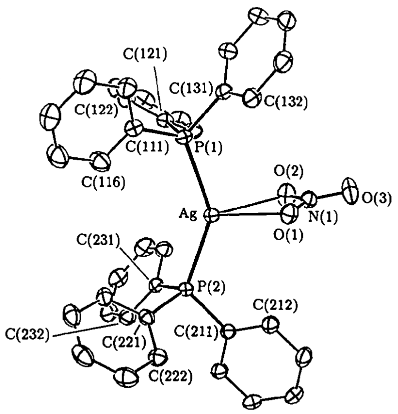

Fig. 1. Molecular structure and numbering scheme for $[Ag(PPh_3)_2NO_3]$ (*ORTEP*II, Johnson, 1971).

---

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, and all bond distances and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52057 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

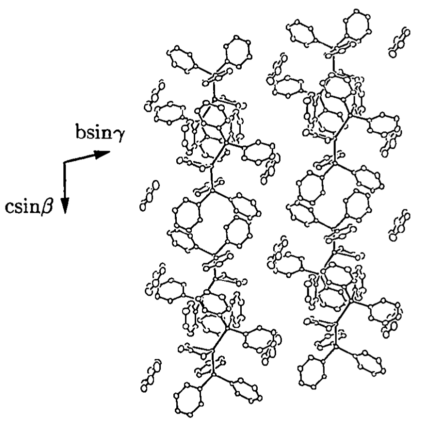

Fig. 2. Crystal lattice for $[Ag(PPh_3)_2NO_3].C_6H_6$ viewed down the [100] direction.

The Australian Research Grants Scheme is thanked for support.

References

Barron, P. F., Dyason, J. C., Healy, P. C., Engelhardt, L. M., Skelton, B. W. & White, A. H. (1986) *J. Chem. Soc. Dalton Trans.* pp. 1965–1970

Boer, J.L. de & Duisenberg, A. J. M. (1984). *Enraf-Nonius CAD-4F Diffractometer Software Update*, February 1984. Enraf–Nonius, Groningen and Utrecht, The Netherlands.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 99, 149. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Johnson, C. K. (1971). *ORTEP*II. Report ORNL-3794, Oak Ridge National Laboratory, Tennessee, USA.

Lobana, T. S., Bhatia, P. K. & Tiekink, E. R. T. (1989). *J. Chem. Soc. Dalton Trans.* pp. 749-751.

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

Acta Cryst. (1989). C**45**, 1817–1818

Structure of Bis(μ-diethyllead)-bis(tetracarbonyliron)

By Colin Campbell and Louis J. Farrugia*

Department of Chemistry, Univeristy of Glasgow, Glasgow G12 8QQ, Scotland

(*Received* 16 *May* 1989; *accepted* 15 *June* 1989)

Abstract. $[Fe_2(CO)_8\{\mu\text{-}Pb(C_2H_5)_2\}_2]$, $M_r = 866{\cdot}4$, orthorhombic, *Pbca*, $a = 9{\cdot}685\,(2)$, $b = 13{\cdot}824\,(6)$, $c = 17{\cdot}940\,(7)$ Å, $V = 2402\,(1)$ Å^3, $Z = 4$, $D_x = 2{\cdot}40$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71069$ Å, $\mu = 153{\cdot}0$ cm^{-1}, $F(000) = 1584$, $T = 298$ K, $R = 0{\cdot}0526$ for 1038 unique observed reflections. The structure consists of a centrosymmetric planar dimer of $Fe(CO)_4$ and $Pb(C_2H_5)_2$ units, with two crystallographically independent Fe—Pb distances of 2·734 (4) and 2·718 (4) Å. The $Fe(CO)_4$ unit shows a distortion towards tetrahedral geometry, with C(1)—Fe—C(2) and C(3)—Fe—C(4) angles of 158·4 (11) and 98·7 (12) °, respectively.

Experimental. Prepared by photolysis of $Fe(CO)_5$ and $PbEt_4$ in hexane. Red prisms from hexane solution: crystal dimensions *ca* $0{\cdot}6 \times 0{\cdot}4 \times 0{\cdot}2$ mm; systematic absences: $0kl$, $k = 2n+1$; $h0l$, $l = 2n+1$; $hk0$, $h = 2n+1$; Enraf–Nonius CAD-4F diffractometer; graphite monochromator; $\theta/2\theta$ scan mode; cell parameters refined by least-squares methods from setting angles of 25 independent θ reflections ($11 < \theta < 13°$); intensities measured to $\theta = 25{\cdot}0°$ over hkl range 0 to 11, 0 to 16, −1 to 21; $\bar{6}\bar{1}\bar{3}$ and $1\bar{7}4$ measured every 2 h with a 2% decay over 26 h data collection; 2589 data measured, 2109 independent data with 1038 having $I > 2{\cdot}5\sigma(I)$ considered observed and used in structure determination and refinement; R_{int} before absorption correction 0·057, after correction 0·033; corrected for Lorentz–

Table 1. *Final positional parameters (fractional coordinates) with e.s.d.'s in parentheses, and equivalent isotropic thermal parameters* (Å^2)

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

	x	y	z	U_{eq}
Pb	−0·04195 (9)	0·47058 (7)	0·58964 (5)	0·049
Fe	0·2022 (3)	0·4441 (2)	0·5159 (2)	0·050
O(1)	0·0580 (19)	0·2699 (12)	0·4666 (11)	0·092
O(2)	0·2457 (17)	0·6438 (13)	0·5655 (10)	0·084
O(3)	0·337 (2)	0·355 (1)	0·646 (1)	0·117
O(4)	0·4368 (18)	0·4412 (15)	0·4143 (11)	0·107
C(1)	0·116 (2)	0·341 (2)	0·485 (1)	0·060
C(2)	0·225 (2)	0·565 (2)	0·545 (1)	0·059
C(3)	0·287 (3)	0·390 (2)	0·596 (1)	0·062
C(4)	0·343 (2)	0·442 (2)	0·452 (1)	0·061
C(5)	−0·015 (3)	0·551 (3)	0·685 (2)	0·137
C(6)	−0·133 (5)	0·575 (3)	0·726 (2)	0·146
C(7)	−0·127 (3)	0·332 (2)	0·627 (2)	0·095
C(8)†	−0·045 (5)	0·287 (3)	0·677 (3)	0·08 (1)
C(8A)‡	−0·216 (9)	0·330 (6)	0·663 (5)	0·11 (3)

† Site occupancy 0·6.
‡ Site occupancy 0·4.

---

* To whom correspondence should be addressed.

0108-2701/89/111817-02$03.00