Acta Cryst. (1998). C**54**, 727–728

(Nitrito-*O*,*O*′)bis(triphenylphosphine-*P*)-silver(I) Dichloromethane Solvate

FERDINAND BELAJ, ANDREAS TRNOSKA AND EDGAR NACHBAUR

Institut für Anorganische Chemie, Karl-Franzens-Universität Graz, Schubertstr. 1, A-8010 Graz, Austria. E-mail: belaj@kfunigraz.ac.at

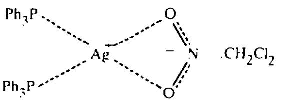
(*Received 8 May 1997; accepted 22 December 1997*)

Abstract

In the title complex, $[Ag(NO_2)\{(C_6H_5)_3P\}_2].CH_2Cl_2$, the Ag^+ cation exhibits a tetrahedral coordination built up by the two O atoms of the anion [Ag—O 2.386 (3) and 2.481 (3) Å] and the two P atoms of the ligands [Ag—P 2.4120 (12) and 2.4401 (12) Å], and lies within the plane of the anion [N—O 1.253 (4) and 1.259 (4) Å, and O—N—O 114.1 (3)°].

Comment

In the course of our work on Ag^+–oxoanion interactions in ordinary salts and coordination compounds (Belaj *et al.*, 1997), we isolated the title compound, (I). The intimate neutral ion pairs (1:1) found in (I) are much more shielded by the triphenylphosphine ligands and the solvent molecules than those in $[(C_6H_5)_3P]_2AgNO_3$ (Barron *et al.*, 1986); whereas the shortest Ag···O distances besides those within the ion pair are 3.075 (3) Å in the latter compound, they are 6.836 (4) Å in (I). The almost planar arrangement of the Ag^+–NO_2^- ion pair in (I) can be conceived as part of the infinite $[\cdots NO_2^- \cdots Ag^+ \cdots]_n$ chains in the lattice of $AgNO_2$ (Ohba & Saito, 1981). The Ag—O [2.441 (3) Å] and N—O [1.248 (3) Å] distances, and the O—N—O angle [114.6 (4)°] in $AgNO_2$ are close to those of (I) (see Table 1). No indication of partial covalency in the Ag—O bond is found in the ion pair of (I); the differences in the two Ag—O distances are accounted for by lattice effects.



(I)

Similar structural fragments are found in the disordered structures of $AgNa(NO_2)_2$ (Ishihara *et al.*, 1987) and $Ag_2Li(NO_2)_3$ (Ohba *et al.*, 1986). In contrast to these structures in which the anion is situated between two Ag^+ ions, no disorder of the NO_2^- anion is detected in (I), since in the packing, the N atom of the anion is surrounded by the phenyl rings and the solvent molecule.

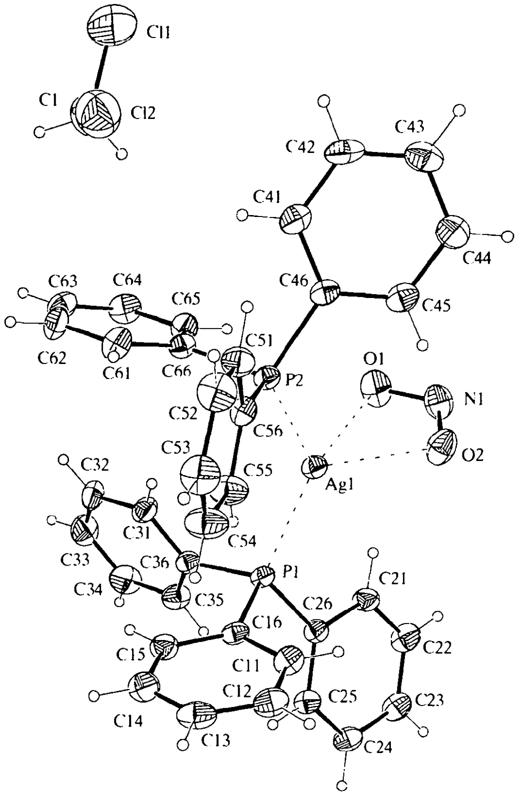


Fig. 1. The asymmetric unit of (I) showing 50% probability displacement ellipsoids. The four shortest contacts to the Ag^+ ion are plotted with dashed bonds.

Experimental

Triphenylphosphine (2.02 g) and $AgNO_2$ (0.59 g) (molar ratio 2:1) were dissolved in 10 ml of CH_2Cl_2 and stirred at 313 K in a closed tube until all the solid had disappeared. On slow cooling, large opaque crystals [m.p. 503.8 K (dec.)] separated from the solution. The measurements were performed with a single crystal which was immersed in oil and immediately cooled to 90 K.

Crystal data

$[Ag(NO_2)(C_{18}H_{15}P)_2]$.-
 CH_2Cl_2
$M_r = 763.39$
Triclinic
$P\bar{1}$
$a = 10.817 (2)$ Å
$b = 12.908 (3)$ Å
$c = 14.080 (3)$ Å
$\alpha = 105.13 (3)°$
$\beta = 105.77 (4)°$
$\gamma = 103.46 (4)°$
$V = 1726.0 (6)$ Å^3
$Z = 2$
$D_x = 1.469$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 55 reflections
$\theta = 15.1–19.8°$
$\mu = 0.865$ mm^{-1}
$T = 90 (2)$ K
Block
0.35 × 0.20 × 0.20 mm
Colourless

Data collection

Stoe four-circle diffractometer
ω scans
Absorption correction: none
8624 measured reflections
7611 independent reflections
6331 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.040$
$\theta_{\text{max}} = 27.5°$
$h = -9 \rightarrow 13$
$k = -16 \rightarrow 9$
$l = -18 \rightarrow 18$
3 standard reflections every 100 reflections intensity decay: 1.52%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.134$
$S = 1.044$
7602 reflections
413 parameters
H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.0776P)^2 + 1.955P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\text{max}} = 0.001$
$\Delta\rho_{\text{max}} = 1.253$ e Å^{-3}
$\Delta\rho_{\text{min}} = -1.670$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Ag1—O2	2.386 (3)	Cl2—C1	1.769 (6)
Ag1—P1	2.4120 (12)	P1—C26	1.815 (3)
Ag1—P2	2.4401 (12)	P1—C16	1.825 (3)
Ag1—O1	2.481 (3)	P1—C36	1.826 (4)
N1—O1	1.253 (4)	P2—C46	1.821 (4)
N1—O2	1.259 (4)	P2—C56	1.826 (3)
Cl1—C1	1.777 (6)	P2—C66	1.826 (4)
O2—Ag1—P1	118.35 (8)	N1—O2—Ag1	99.6 (2)
O2—Ag1—P2	109.99 (8)	Cl2—C1—Cl1	111.3 (3)
P1—Ag1—P2	128.67 (4)	C26—P1—Ag1	117.64 (12)
O2—Ag1—O1	51.25 (10)	C16—P1—Ag1	110.01 (12)
P1—Ag1—O1	120.81 (8)	C36—P1—Ag1	111.74 (12)
P2—Ag1—O1	102.06 (8)	C46—P2—Ag1	115.14 (12)
O1—N1—O2	114.1 (3)	C56—P2—Ag1	116.73 (12)
N1—O1—Ag1	95.1 (2)	C66—P2—Ag1	108.82 (12)
O1—N1—O2—Ag1	−1.3 (4)	P1—Ag1—P2—C46	−179.82 (12)
P2—Ag1—P1—C26	−169.02 (12)		

The C atoms of the phenyl rings were refined without any constraints. The H atoms were constrained to parent sites and common isotropic displacement parameters were refined for the H atoms of the same phenyl group [$U_{\text{iso}} = 0.036\,(5)$–$0.046\,(6)$ Å^2] and for the H atoms of the solvent molecule [$U_{\text{iso}} = 0.091\,(16)$ Å^2]. The strongest peaks (0.63 to 1.253 e Å^{-3}) and the holes deeper than −0.61 e Å^{-3} in the final difference Fourier map were situated 0.75–1.29 Å from the Ag$^+$ ion.

Data collection: local program. Cell refinement: local program. Data reduction: local program. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *SHELXL*93.

FB thanks Professor Dr C. Kratky, Institute of Physical Chemistry, University of Graz, Austria, for the use of the diffractometer.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1123). Services for accessing these data are described at the back of the journal.

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.
Belaj, F., Trnoska, A. & Nachbaur, E. (1997). *Z. Kristallogr.* **212**, 355–361.
Ishihara, M., Ohba, S. & Saito, Y. (1987). *Acta Cryst.* B**43**, 160–164.
Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
Ohba, S., Matsumoto, F., Ishihara, M. & Saito, Y. (1986). *Acta Cryst.* C**42**, 1–4.
Ohba, S. & Saito, Y. (1981). *Acta Cryst.* B**37**, 1911–1913.
Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures.* University of Göttingen, Germany.
Sheldrick, G. M. (1993). *SHELXL93. Program for Refinement of Crystal Structures.* University of Göttingen, Germany.

Acta Cryst. (1998). C**54**, 728–732

8-Methylaminoquinolinium Salts of Tetrachlorodimethylstannate(IV) and Tetrachlorodiphenylstannate(IV)

ALAN HAZELL,[a] LIAN EE KHOO,[b] JIEXIANG OUYANG,[b] BERNHARD J. RAUSCH[a] AND ZARA MIRAVENT TAVARES[a]

[a]*Department of Chemistry, Aarhus University, Langelandsgade 140, DK-8000 Århus C, Denmark, and* [b]*School of Science, National Institute of Education, 469 Bukit Timah Road, Singapore 259756. E-mail: ach@kemi.aau.dk*

(*Received 21 October 1997; accepted 9 January 1998*)

Abstract

The title compounds, $(C_{10}H_{11}N_2)_2[SnCl_4(CH_3)_2]$ and $(C_{10}H_{11}N_2)_2[SnCl_4(C_6H_5)_2]$, have been prepared from the reaction of 8-methylaminoquinoline with diorganotin dichlorides. In the methyl compound, the Sn atom is coordinated to two methyl groups, which are *trans* with respect to each other [Sn—C 2.102 (5) and 2.103 (4) Å], and to four Cl atoms [Sn—Cl 2.442 (1), 2.461 (1), 2.894 (1) and 3.098 (1) Å]. The two long Sn—Cl bonds are *trans* to the short ones and their Cl atoms are hydrogen bonded to the N—H groups of the 8-methylaminoquinolinium cations. The Sn—CH_3 bonds are bent away from the short Sn—Cl bonds so that the CH_3—Sn—CH_3 angle is 155.7 (2)°. A correlation between the C—Sn—C angle and the Sn—Cl bond lengths was observed. The anion of the phenyl compound is close to being octahedral and has exact twofold symmetry; the phenyl groups are *trans* with respect to each other [Sn—C 2.143 (2) Å] and Sn—Cl distances are in the range 2.497 (1)–2.672 (1) Å. Two Cl atoms are hydrogen bonded to N—H groups of the cations.

Acta Crystallographica Section C
ISSN 0108-2701 © 1998