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Trigonal–planar silver coordination in (3,5-dinitrobenzoato)bis(triphenylphosphine)silver(I)

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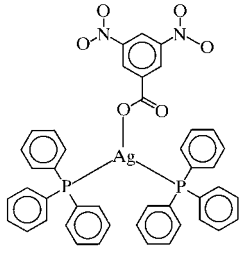
The three-coordinate Ag atom in the title compound, $[Ag(C_7H_3N_2O_6)(C_{18}H_{15}P)_2]$, shows trigonal–planar coordination [P—Ag—P = 147.1 (1)° and Σ_{Ag} = 359.0 (3)°]. Adjacent molecules are linked through the O atoms of adjacent nitro groups [Ag···O = 3.205 (3) and 3.302 (4) Å] into a zigzag chain running parallel to the *c* axis.

Comment

Silver carboxylates yield 1:1 and 1:2 adducts with triphenylphosphine; with the 1:2 complexes, the carboxyl CO_2 entity chelates to the Ag atom and renders it four-coordinate in a distorted tetrahedral geometry, as noted in a variety of carboxylates, *e.g.* acetate [Ag—O = 2.420 (2) and 2.438 (2) Å; Ng & Othman, 1997], trifluoroacetate [Ag—O = 2.542 (4) and 2.526 (3) Å; Ng, 1998], lactate [2.425 (3) and 2.508 (4) Å; Hanna & Ng, 2000] and 2,4-dichlorophenoxyacetate [2.436 (2) and 2.571 (2) Å; Subramanian *et al.*, 2000]. The adducts are synthesized by treating the silver carboxylate with triphenylphosphine in the appropriate stoichiometry; however, the direct reaction cannot be used for a large number of silver carboxylates. A more general synthesis involves the reaction of the hydrated 1:1 silver acetate complex with a carboxylic acid; the dimeric compound (Ng & Othman, 1997) reacts with a variety of organic acids to afford the corresponding complexes (Othman, Fun & Sivakumar, 1996; Othman, Fun, Sivakumar *et al.*, 1996; Othman, Goh *et al.*, 1996). The title complex, (I), was prepared by reacting the dimer with 3,5-dinitrobenzoic acid. In the 3,5-dinitrobenzoate adduct, the carboxylato group is monodentate [Ag—O = 2.285 (3) Å], as the double-bond carbonyl atom is more than 3 Å away from the Ag atom. A monodentate mode for the carboxylato group is also noted in the 1:3 complex with α-oxobenzeneacetate; in this compound, three phosphine ligands are linked to the Ag atom, their steric bulk preventing the carbonyl O atom from approaching the Ag atom (Ng, 1997). Three-coordination for silver has not been previously noted for 1:2 silver carboxylate phosphine adducts. A T-shaped geometry has been found in the 1:1 adduct with silver phenylacetate, this geometry arises from bridging by the carboxyl entity, which results in the formation of a centrosymmetric dinuclear compound (Hong *et al.*, 1996). The hemihydrated 1:1 adduct with silver acetate exists as a non-centrosymmetric dimer; the geometry of both atoms is closer to trigonal–planar rather than T-shaped (Ng & Othman, 1997). The Ag atom in the 3,5-dinitrobenzoate adduct is also assigned a trigonal–planar geometry.



(I)

Experimental

Bis[acetato(triphenylphosphine)silver] hemihydrate (Ng & Othman, 1997) and 3,5-nitrobenzoic acid (1:2 molar ratio) were dissolved in warm ethanol to give a yellow solution. The solution deposited the 1:2 adduct on cooling; the compound was recrystallized from a 1:1 methanol–chloroform mixture (m.p. 459 K).

Crystal data

$[Ag(C_7H_3N_2O_6)(C_{18}H_{15}P)_2]$
M_r = 843.52
Monoclinic, $C2/c$
a = 44.44 (2) Å
b = 9.651 (4) Å
c = 19.527 (7) Å
β = 111.70 (3)°
V = 7781 (6) $Å^3$
Z = 8

D_x = 1.440 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 7.0–15.0°
μ = 0.651 mm^{-1}
T = 298 (2) K
Parallelepiped, yellow
0.54 × 0.50 × 0.42 mm

Data collection

Siemens *R*3*m* four-circle diffractometer
w scans
Absorption correction: empirical *via* ψ scan (North *et al.*, 1968)
T_{min} = 0.701, T_{max} = 0.761
8588 measured reflections
8480 independent reflections
6095 reflections with $I > 2\sigma(I)$

R_{int} = 0.025
θ_{max} = 27°
$h = 0 \rightarrow 56$
$k = 0 \rightarrow 12$
$l = -24 \rightarrow 23$
2 standard reflections every 120 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.110
S = 1.029
8480 reflections
487 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0555P)^2 + 3.6993P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.55 e $Å^{-3}$
$\Delta\rho_{min}$ = −0.38 e $Å^{-3}$

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Table 1
Selected geometric parameters (Å, °).

Ag1—O1	2.285 (3)	P1—C13	1.830 (3)
Ag1—P1	2.432 (1)	P2—C19	1.827 (3)
Ag1—P2	2.466 (1)	P2—C25	1.824 (4)
P1—C1	1.826 (3)	P2—C31	1.828 (3)
P1—C7	1.826 (3)		
O1—Ag1—P1	115.5 (1)	C13—P1—Ag1	114.5 (1)
O1—Ag1—P2	96.4 (1)	C19—P2—C25	103.9 (2)
P1—Ag1—P2	147.3 (1)	C19—P2—C31	102.9 (2)
C1—P1—C7	104.2 (1)	C25—P2—C31	103.1 (2)
C1—P1—C13	103.5 (1)	C19—P2—Ag1	115.6 (1)
C7—P1—C13	104.9 (1)	C25—P2—Ag1	118.2 (1)
C1—P1—Ag1	116.3 (1)	C31—P2—Ag1	111.4 (1)
C7—P1—Ag1	112.2 (1)		

H atoms were refined as riding on their parent C atoms, with $U(\mathrm{H}) = 1.5U_{\mathrm{eq}}(\mathrm{C})$.

Data collection: *SHELXTL-Plus* (Sheldrick, 1990); cell refinement: *SHELXTL-Plus*; data reduction: *SHELXTL-Plus*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *SHELXL*97.

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References

Hanna, J. V. & Ng, S. W. (2000). *Acta Cryst.* C**56**, 24–25.

Hong, M.-C., Mak, T. C. W., Zhou, Z.-Y., Xue, F., Wu, D.-E., Zhen, Y. & Liu, H.-Q. (1996). *J. Chem. Crystallogr.* **26**, 553–557.

Ng, S. W. (1997). *Z. Kristallogr. New Cryst. Struct.* **212**, 279–281.

Ng, S. W. (1998). *Acta Cryst.* C**54**, 743–744.

Ng, S. W. & Othman, A. H. (1997). *Acta Cryst.* C**53**, 1396–1400.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Othman, A. H., Fun, H.-K. & Sivakumar, K. (1996). *Acta Cryst.* C**52**, 843–845.

Othman, A. H., Fun, H.-K., Sivakumar, S., Yang Farina, A. A. & Baba, I. (1996). *Acta Cryst.* C**52**, 1933–1935.

Othman, A. H., Goh, S. C., Fun, H.-K. & Sivakumar, S. (1996). *Acta Cryst.* C**52**, 2760–2763.

Sheldrick, G. M. (1990). *SHELXTL-Plus.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.

Subramanian, R. R., Shanmuga, S. A., Kwek, K. H., Low, K. S., Shanmuga Sundara Raj, S., Fun, H.-K., Razak, I. A., Hanna, J. V. & Ng, S. W. (2000). *Acta Cryst.* C**56**, e292–294.

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