Bis[(4-methylphenyl)diphenylphosphine-κP](nitrito-κ²O,O')silver(I)

Kariska Potgieter,a Frederick P. Malan,b Oyekunle Azeez Alimiab and Reinout Meijboom*a

*Department of Chemical Sciences, University of Johannesburg, PO Box 524, Auckland Park, 2006, Johannesburg, South Africa, and bDepartment of Chemistry, University of Pretoria, Lynnwood Road, Hatfield, Pretoria, 0002, South Africa.
*Correspondence e-mail: rmeijboom@uj.ac.za

The title AgI complex, [Ag(NO₂)(C₁₉H₁₇P)₂], reveals a distorted pseudo-trigonal–planar shape around the AgI atom geometry resulting from the coordination of two phosphine ligands, as well as a nitrito-O,O' ligand coordinating to the silver(I) atom through the oxygen atoms; in this description, the two oxygen atoms are assumed to occupy one position, forming an acute O—Ag—O angle of 51.44 (9)°. The plane resulting from the NO₂ coordination to Ag is nearly perpendicular to the plane from the coordination of the phosphine-P atoms to Ag [dihedral angle = 86.43 (9)°].

Structure description

The molecular structure of the title compound is shown in Fig. 1. The complex crystallizes in the monoclinic space group P2₁/c with Z = 4. The asymmetric unit contains one complete silver complex molecule, featuring an AgI atom, two diphenyl-p-tolylphosphine ligands, and one NO₂ coordinating in a bidentate fashion. Near-identical Ag—P bond lengths are observed [Ag1—P1 = 2.4209 (7) A and Ag1—P2 = 2.4251 (8) A]. The nitrito ligand is similarly coordinating in a near symmetric fashion (Ag1—O1 = 2.422 (2), Ag1—O2 = 2.415 (2), N1—O1 = 1.253 (4) and N1–O2 = 1.255 (4) A). As seen in Fig. 1, the four-coordinate silver(I) atom essentially exhibits a pseudo trigonal–planar shape with the three coordinating ligands, with bond angles P1—Ag1—P2 [129.51 (3)°], P1—Ag1—O1 [116.23 (7)°], P1—Ag1—O2 [111.09 (7)°], P2—Ag1—O1 [110.79 (7)°], P2—Ag1—O2 [111.96 (7)°] and O1—Ag1—O2 [51.44 (9)°]; in this description, the two oxygen atoms are assumed to occupy one position. The plane P11 defined by Ag1, O1, O2 and N1 crosses the plane P12 defined by P1, P2 and Ag1 at an angle of 86.43 (9)°. The ipso-carbon atoms of each of the phosphine ligands overlap in a near-eclipsed fashion when viewed.
down the P1—Ag1—P2 plane Pl2. Corresponding torsion angles are Ag1—P1—C1—C2 = 23.4 (3), Ag1—P1—C7—C8 = 51.9 (3), Ag1—P1—C13—C14 = 147.8 (3), Ag1—P2—C20—C21 = 29.0 (3), Ag1—P2—C26—C27 = 133.3 (3), and Ag1—P2—C32—C33 = 132.3 (3). The complex packs in three dimensions as layers of molecules, leaving thin corrugated channels in between the inorganic layers when viewed along the a axis (Fig. 2).

Synthesis and crystallization

Diphenyl-p-tolylphosphine (1 mmol) was dissolved in acetonitrile (10 ml). Silver nitrite (1 mmol) was dissolved in acetonitrile (5 ml). The diphenyl-p-tolylphosphine solution (10 ml) was added to the silver nitrite solution (5 ml), to give a 2:1 molar ratio reaction. The mixture was heated under reflux for 2 h after which the solution was left to crystallize.

Refinement

For full experimental details including crystal data, data collection and structure refinement details, refer to Table 1.

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Bis[(4-methylphenyl)diphenylphosphine-$\kappa P$](nitrito-$\kappa^2O,O'$)silver(I)

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**Bis[(4-methylphenyl)diphenylphosphine-$\kappa P$](nitrito-$\kappa^2O,O'$)silver(I)**

Crystal data

$[\text{Ag(NO}_2\text{(C}_{19}\text{H}_{17}\text{P})_2\text{]}$ $F(000) = 1448$

$M_r = 706.47$ $D_x = 1.414 \text{ Mg m}^{-3}$

Monoclinic, $P2_1/c$ $\text{Cu K}\alpha$ radiation, $\lambda = 1.54184$ \AA

$a = 11.8709$ (2) \AA $\theta = 3.8–78.9^\circ$

$b = 18.6292$ (2) \AA $\mu = 6.05$ mm$^{-1}$

$c = 15.4003$ (2) \AA $T = 150$ K

$\beta = 103.055$ (1)$^\circ$ Block, colourless

$V = 3317.68$ (8) \AA$^3$ $0.24 \times 0.13 \times 0.10$ mm

Data collection

XtaLAB Synergy R, DW system, HyPix

Radiation source: Rotating-anode X-ray tube,

Rigaku (Cu) X-ray Source

Mirror monochromator

Detector resolution: 10.0000 pixels mm$^{-1}$

$\omega$ scans

Absorption correction: multi-scan

(CrysAlisPro; Rigaku OD, 2022)

Refinement

Refinement on $F^2$ $T_{\text{min}} = 0.188, T_{\text{max}} = 1.000$

Least-squares matrix: full $41716$ measured reflections

$R[F^2 > 2\sigma(F^2)] = 0.040$ 7030 independent reflections

$wR(F^2) = 0.104$ 6535 reflections with $I > 2\sigma(I)$

$S = 1.07$ $\theta_{\text{max}} = 79.2^\circ, \theta_{\text{min}} = 3.8^\circ$

$7030$ reflections $h = -14\rightarrow15$

$399$ parameters $k = -23\rightarrow23$

$0$ restraints $l = -18\rightarrow19$

Primary atom site location: dual

Hydrogen site location: inferred from

neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0425P)^2 + 5.6399P]$ $\Delta(\sigma)_{\text{max}} = 0.002$

where $P = (F_o^2 + 2F_c^2)/3$ $\Delta\rho_{\text{max}} = 0.68$ e \AA$^{-3}$

$\Delta\rho_{\text{min}} = -0.82$ e \AA$^{-3}$

Special details

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

**Refinement.** All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms.
Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

| Atom | x     | y     | z     | Uiso*/*Ueq |
|------|-------|-------|-------|------------|
| Ag1  | 0.33615 (2) | 0.79867 (2) | 0.56752 (2) | 0.03178 (8) |
| P1   | 0.52794 (6)  | 0.74875 (4)  | 0.61894 (5)  | 0.02991 (16) |
| P2   | 0.14991 (6)  | 0.73885 (4)  | 0.53633 (6)  | 0.03298 (17) |
| O1   | 0.3244 (2)   | 0.91204 (13) | 0.48903 (17) | 0.0459 (6)   |
| O2   | 0.3250 (2)   | 0.91839 (14) | 0.62523 (18) | 0.0506 (6)   |
| N1   | 0.3202 (3)   | 0.95191 (15) | 0.5538 (2)   | 0.0492 (8)   |
| C7   | 0.5331 (3)   | 0.69367 (17) | 0.7177 (2)   | 0.0325 (6)   |
| C1   | 0.6466 (3)   | 0.81224 (16) | 0.6530 (2)   | 0.0321 (6)   |
| C25  | −0.0805 (3)  | 0.77034 (18) | 0.4467 (2)   | 0.0380 (7)   |
| H25  | −0.0857      | 0.7224       | 0.4253       | 0.046*       |
| C20  | 0.0223 (3)   | 0.79524 (16) | 0.5000 (2)   | 0.0334 (6)   |
| C26  | 0.1234 (3)   | 0.69196 (16) | 0.6342 (2)   | 0.0355 (7)   |
| C13  | 0.5720 (3)   | 0.68644 (16) | 0.5419 (2)   | 0.0337 (6)   |
| C6   | 0.7500 (3)   | 0.79432 (18) | 0.7117 (2)   | 0.0385 (7)   |
| H6   | 0.7610       | 0.7473       | 0.7360       | 0.046*       |
| C24  | −0.1757 (3)  | 0.8154 (2)   | 0.4248 (2)   | 0.0443 (8)   |
| H24  | −0.2462      | 0.7980       | 0.3888       | 0.053*       |
| C32  | 0.1398 (3)   | 0.66934 (17) | 0.4515 (2)   | 0.0360 (7)   |
| C2   | 0.6317 (3)   | 0.88145 (17) | 0.6186 (2)   | 0.0352 (7)   |
| H2   | 0.5610       | 0.8943       | 0.5790       | 0.042*       |
| C8   | 0.4942 (3)   | 0.7238 (2)   | 0.7887 (2)   | 0.0413 (7)   |
| H8   | 0.4726       | 0.7729       | 0.7870       | 0.050*       |
| C37  | 0.1822 (3)   | 0.68542 (19) | 0.3764 (2)   | 0.0425 (8)   |
| H37  | 0.2095       | 0.7325       | 0.3690       | 0.051*       |
| C31  | 0.2138 (3)   | 0.65361 (19) | 0.6870 (3)   | 0.0456 (8)   |
| H31  | 0.2859       | 0.6510       | 0.6702       | 0.055*       |
| C5   | 0.8371 (3)   | 0.8450 (2)   | 0.7349 (3)   | 0.0461 (8)   |
| H5   | 0.9076       | 0.8326       | 0.7750       | 0.055*       |
| C18  | 0.4881 (3)   | 0.64259 (19) | 0.4912 (3)   | 0.0440 (8)   |
| H18  | 0.4096       | 0.6485       | 0.4941       | 0.053*       |
| C10  | 0.5184 (3)   | 0.6103 (2)   | 0.8646 (2)   | 0.0459 (8)   |
| H10  | 0.5119       | 0.5816       | 0.9143       | 0.055*       |
| C12  | 0.5662 (3)   | 0.62220 (17) | 0.7224 (2)   | 0.0374 (7)   |
| H12  | 0.5942       | 0.6014       | 0.6750       | 0.045*       |
| C14  | 0.6851 (3)   | 0.6784 (2)   | 0.5339 (3)   | 0.0474 (8)   |
| H14  | 0.7437       | 0.7089       | 0.5665       | 0.057*       |
| C11  | 0.5589 (3)   | 0.5806 (2)   | 0.7959 (2)   | 0.0447 (8)   |
| H11  | 0.5819       | 0.5317       | 0.7985       | 0.054*       |
| C4   | 0.8218 (3)   | 0.9132 (2)   | 0.7003 (2)   | 0.0453 (8)   |
| H4   | 0.8817       | 0.9478       | 0.7167       | 0.054*       |
| C3   | 0.7192 (3)   | 0.93168 (19) | 0.6415 (2)   | 0.0445 (8)   |
| H3   | 0.7090       | 0.9787       | 0.6171       | 0.053*       |
| C23  | −0.1685 (3)  | 0.8854 (2)   | 0.4552 (3)   | 0.0457 (8)   |
| H23  | −0.2335      | 0.9164       | 0.4398       | 0.055*       |
| C21  | 0.0295 (3)   | 0.86579 (18) | 0.5299 (3)   | 0.0447 (8)   |
| Atom | x      | y      | z      | U11 | U22 | U33 | U12 | U13 | U23 |
|------|--------|--------|--------|-----|-----|-----|-----|-----|-----|
| H21  | 0.1000 | 0.8837 | 0.5653 | 0.054* |
| C27  | 0.0206 (3) | 0.6970 (2) | 0.6615 (3) | 0.0491 (9) |
| H27  | −0.0422 | 0.7235 | 0.6271 | 0.059* |
| C29  | 0.0976 (4) | 0.6233 (2) | 0.7899 (3) | 0.0515 (9) |
| H29  | 0.0878 | 0.5992 | 0.8421 | 0.062* |
| C35  | 0.1463 (3) | 0.5644 (2) | 0.3217 (3) | 0.0482 (8) |
| C9   | 0.4873 (3) | 0.6817 (2) | 0.8616 (2) | 0.0492 (9) |
| H9   | 0.4609 | 0.7023 | 0.9098 | 0.059* |
| C22  | −0.0663 (3) | 0.9098 (2) | 0.5080 (3) | 0.0518 (9) |
| H22  | −0.0615 | 0.9577 | 0.5297 | 0.062* |
| C36  | 0.1850 (3) | 0.6340 (2) | 0.3126 (3) | 0.0465 (8) |
| H36  | 0.2137 | 0.6462 | 0.2617 | 0.056* |
| C30  | 0.2004 (3) | 0.6192 (2) | 0.7633 (3) | 0.0520 (9) |
| H30  | 0.2628 | 0.5924 | 0.7978 | 0.062* |
| C17  | 0.5172 (4) | 0.5902 (2) | 0.4363 (3) | 0.0523 (9) |
| H17  | 0.4585 | 0.5601 | 0.4028 | 0.063* |
| C33  | 0.0991 (3) | 0.60034 (19) | 0.4599 (3) | 0.0488 (9) |
| H33  | 0.0689 | 0.5882 | 0.5101 | 0.059* |
| C15  | 0.7132 (4) | 0.6261 (2) | 0.4787 (3) | 0.0580 (11) |
| H15  | 0.7914 | 0.6210 | 0.4744 | 0.070* |
| C16  | 0.6305 (4) | 0.5809 (2) | 0.4296 (3) | 0.0542 (10) |
| C28  | 0.0095 (4) | 0.6631 (2) | 0.7393 (3) | 0.0579 (10) |
| H28  | −0.0611 | 0.6676 | 0.7581 | 0.069* |
| C34  | 0.1023 (4) | 0.5492 (2) | 0.3954 (3) | 0.0568 (10) |
| H34  | 0.0735 | 0.5025 | 0.4019 | 0.068* |
| C38  | 0.1539 (4) | 0.5072 (3) | 0.2557 (3) | 0.0684 (12) |
| H38A | 0.0815 | 0.5056 | 0.2100 | 0.103* |
| H38B | 0.1668 | 0.4607 | 0.2860 | 0.103* |
| H38C | 0.2183 | 0.5177 | 0.2275 | 0.103* |
| C19  | 0.6634 (5) | 0.5233 (3) | 0.3706 (4) | 0.0806 (16) |
| H19A | 0.6400 | 0.4763 | 0.3889 | 0.121* |
| H19B | 0.7473 | 0.5239 | 0.3763 | 0.121* |
| H19C | 0.6242 | 0.5325 | 0.3085 | 0.121* |

Atomic displacement parameters (Å²)

|  | U11   | U22   | U33   | U12  | U13  | U23  |
|---|-------|-------|-------|------|------|------|
| Ag  | 0.02537 (12) | 0.02500 (12) | 0.04510 (14) | −0.00004 (7) | 0.00826 (9) | 0.00223 (8) |
| P   | 0.0263 (3) | 0.0251 (3) | 0.0388 (4) | 0.0012 (3) | 0.0084 (3) | 0.0051 (3) |
| O1  | 0.0571 (15) | 0.0328 (12) | 0.0506 (14) | 0.0036 (11) | 0.0179 (12) | 0.0019 (10) |
| O2  | 0.0616 (16) | 0.0403 (14) | 0.0527 (15) | −0.0017 (12) | 0.0191 (12) | −0.0120 (12) |
| N1  | 0.0524 (18) | 0.0248 (14) | 0.071 (2) | 0.0032 (12) | 0.0154 (15) | −0.0042 (14) |
| C7  | 0.0261 (14) | 0.0343 (16) | 0.0376 (16) | −0.0021 (11) | 0.0080 (12) | 0.0028 (12) |
| C1  | 0.0288 (14) | 0.0299 (15) | 0.0390 (16) | 0.0013 (12) | 0.0104 (12) | 0.0028 (12) |
| C25 | 0.0315 (16) | 0.0320 (16) | 0.0496 (19) | 0.0001 (12) | 0.0075 (13) | −0.0020 (14) |
| C20 | 0.0252 (14) | 0.0295 (15) | 0.0462 (17) | −0.0008 (11) | 0.0098 (12) | 0.0019 (12) |
| C26 | 0.0319 (15) | 0.0267 (14) | 0.0482 (18) | −0.0009 (12) | 0.0097 (13) | −0.0024 (13) |
### Geometric parameters (Å, °)

|     |     |     |     |     |     |
|-----|-----|-----|-----|-----|-----|
|     |     |     |     |     |     |
| Ag1—P1 | 2.4209 (7) | C32—C37 | 1.395 (5) |
| Ag1—P2 | 2.4251 (8) | C32—C33 | 1.390 (5) |
| Ag1—O1 | 2.422 (2)  | C2—C3   | 1.383 (5) |
| Ag1—O2 | 2.415 (2)  | C8—C9   | 1.386 (5) |
| P1—C7  | 1.825 (3)  | C37—C36 | 1.377 (5) |
| P1—C1  | 1.823 (3)  | C31—C30 | 1.380 (5) |
| P1—C13 | 1.820 (3)  | C5—C4   | 1.373 (5) |
| P2—C20 | 1.824 (3)  | C18—C17 | 1.385 (5) |
| P2—C26 | 1.830 (3)  | C10—C11 | 1.373 (5) |
| O1—N1  | 1.253 (4)  | C12—C11 | 1.389 (5) |
| O2—N1  | 1.255 (4)  | C14—C15 | 1.382 (5) |

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| Bond          | Distance (Å) | Bond          | Distance (Å) | Bond          | Distance (Å) |
|--------------|--------------|--------------|--------------|--------------|--------------|
| C7—C8        | 1.396 (5)    | C4—C3        | 1.387 (5)    |               |              |
| C7—C12       | 1.386 (4)    | C23—C22      | 1.377 (5)    |               |              |
| C1—C6        | 1.391 (4)    | C21—C22      | 1.380 (5)    |               |              |
| C1—C2        | 1.390 (4)    | C27—C28      | 1.386 (6)    |               |              |
| C25—C20      | 1.388 (4)    | C29—C30      | 1.374 (6)    |               |              |
| C25—C24      | 1.387 (5)    | C29—C28      | 1.374 (6)    |               |              |
| C20—C21      | 1.389 (4)    | C35—C36      | 1.394 (5)    |               |              |
| C26—C31      | 1.388 (5)    | C35—C34      | 1.381 (6)    |               |              |
| C26—C27      | 1.381 (5)    | C35—C38      | 1.489 (6)    |               |              |
| C13—C18      | 1.384 (5)    | C17—C16      | 1.383 (6)    |               |              |
| C13—C14      | 1.384 (5)    | C33—C34      | 1.383 (5)    |               |              |
| C6—C5        | 1.386 (5)    | C15—C16      | 1.381 (6)    |               |              |
| C24—C23      | 1.381 (5)    | C16—C19      | 1.513 (6)    |               |              |
| P1—Ag1—P2    | 129.51 (3)   | C5—C6—C1    | 120.1 (3)    |               |              |
| P1—Ag1—O1    | 116.23 (7)   | C23—C24—C25 | 120.3 (3)    |               |              |
| O1—Ag1—P2    | 110.79 (7)   | C37—C32—P2  | 117.7 (2)    |               |              |
| O2—Ag1—P1    | 111.09 (7)   | C33—C32—P2  | 123.9 (3)    |               |              |
| O2—Ag1—P2    | 111.96 (7)   | C33—C32—P2  | 118.3 (3)    |               |              |
| O2—Ag1—O1    | 51.44 (9)    | C3—C2—C1    | 120.4 (3)    |               |              |
| C7—P1—Ag1    | 109.92 (10)  | C9—C8—C7    | 119.8 (3)    |               |              |
| C1—P1—Ag1    | 116.95 (10)  | C36—C37—C32 | 120.9 (3)    |               |              |
| C1—P1—C7     | 104.28 (14)  | C30—C31—C26 | 121.0 (3)    |               |              |
| C13—P1—Ag1   | 114.79 (11)  | C4—C5—C6    | 120.3 (3)    |               |              |
| C13—P1—C7    | 103.00 (14)  | C13—C18—C17 | 120.9 (3)    |               |              |
| C13—P1—C1    | 106.52 (14)  | C11—C10—C9  | 119.9 (3)    |               |              |
| C20—P2—Ag1   | 116.91 (10)  | C7—C12—C11  | 120.6 (3)    |               |              |
| C20—P2—C26   | 104.02 (15)  | C15—C14—C13 | 120.3 (4)    |               |              |
| C26—P2—Ag1   | 112.02 (11)  | C10—C11—C12 | 120.1 (3)    |               |              |
| C32—P2—Ag1   | 112.17 (10)  | C5—C4—C3    | 120.2 (3)    |               |              |
| C32—P2—C20   | 105.88 (15)  | C2—C3—C4    | 119.8 (3)    |               |              |
| C32—P2—C26   | 104.80 (15)  | C22—C23—C24 | 119.4 (3)    |               |              |
| N1—O1—Ag1    | 97.3 (2)     | C22—C21—C20 | 119.8 (3)    |               |              |
| N1—O2—Ag1    | 97.59 (19)   | C26—C27—C28 | 119.7 (4)    |               |              |
| O1—N1—O2     | 113.6 (3)    | C28—C29—C30 | 118.3 (4)    |               |              |
| C8—C7—P1     | 118.2 (2)    | C36—C35—C38 | 121.7 (4)    |               |              |
| C12—C7—P1    | 122.7 (3)    | C34—C35—C36 | 117.8 (3)    |               |              |
| C12—C7—C8    | 119.0 (3)    | C34—C35—C38 | 120.5 (4)    |               |              |
| C6—C1—P1     | 122.8 (2)    | C10—C9—C8   | 120.6 (3)    |               |              |
| C2—C1—P1     | 117.9 (2)    | C23—C22—C21 | 121.0 (3)    |               |              |
| C2—C1—C6     | 119.2 (3)    | C37—C36—C35 | 121.0 (3)    |               |              |
| C24—C25—C20  | 120.1 (3)    | C29—C30—C31 | 120.6 (4)    |               |              |
| C25—C20—P2   | 123.2 (2)    | C16—C17—C18 | 121.0 (4)    |               |              |
| C25—C20—C21  | 119.4 (3)    | C34—C33—C32 | 120.2 (4)    |               |              |
| C21—C20—P2   | 117.3 (2)    | C16—C15—C14 | 121.7 (4)    |               |              |
| C31—C26—P2   | 118.3 (3)    | C17—C16—C19 | 121.4 (4)    |               |              |
| C27—C26—P2   | 123.1 (3)    | C15—C16—C17 | 117.8 (4)    |               |              |
| C27—C26—C31  | 118.4 (3)    | C15—C16—C19 | 120.8 (4)    |               |              |
| Bond                  | Angle (deg) | Bond                  | Angle (deg) | Bond                  | Angle (deg) |
|----------------------|-------------|----------------------|-------------|----------------------|-------------|
| C18—C13—P1           | 117.9 (3)   | C29—C28—C27          | 121.8 (4)   |
| C14—C13—P1           | 123.7 (3)   | C35—C34—C33          | 121.8 (4)   |
| C14—C13—C18          | 118.3 (3)   |                      |             |