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{ μ -1,2-Bis[bis(4-methoxyphenyl)-phosphanyl]-1,2-dimethylhydrazine- $\kappa^2P:P'$ }bis[chloridogold(I)] tetrahydrofuran disolvate

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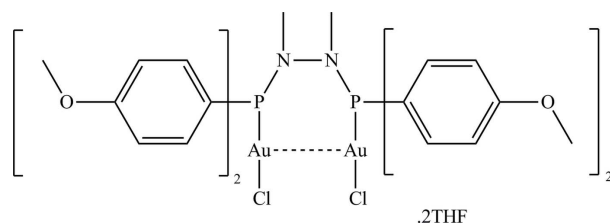
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Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.019; wR factor = 0.049; data-to-parameter ratio = 22.4.

The title compound, $[\text{Au}_2\text{Cl}_2(\text{C}_{30}\text{H}_{34}\text{N}_2\text{O}_4\text{P}_2)] \cdot 2\text{C}_4\text{H}_8\text{O}$, is formed from a bidentate phosphine ligand complexed to two almost linearly coordinated gold(I) atoms [$\text{P}-\text{Au}-\text{Cl} = 175.68$ (3) Å]. The nuclei are 3.122 (2) Å apart. The molecule exhibits a twofold rotation axis.

Related literature

For the synthesis of the parent ligand and related structures utilizing alternative metals, see: Reddy *et al.* (1994, 1995); Slawin *et al.* (2002); Kriel *et al.* (2010*a,b*, 2011*a,b*). For $\text{Au} \cdots \text{Au}$ interactions, see: Holleman & Wiberg (2001).



Experimental

Crystal data

$[\text{Au}_2\text{Cl}_2(\text{C}_{30}\text{H}_{34}\text{N}_2\text{O}_4\text{P}_2)] \cdot 2\text{C}_4\text{H}_8\text{O}$ $V = 4257$ (3) Å³
 $M_r = 1157.57$ $Z = 4$
 Monoclinic, $C2/c$ $\text{Mo } K\alpha$ radiation
 $a = 23.208$ (5) Å $\mu = 7.13$ mm⁻¹
 $b = 9.080$ (5) Å $T = 173$ K
 $c = 20.220$ (5) Å $0.58 \times 0.45 \times 0.10$ mm
 $\beta = 92.414$ (5)°

Data collection

Bruker SMART CCD area-detector 33198 measured reflections
 diffractometer 5264 independent reflections
 Absorption correction: multi-scan 4679 reflections with $I > 2\sigma(I)$
 (SADABS; Bruker, 1999) $R_{\text{int}} = 0.040$
 $T_{\text{min}} = 0.044$, $T_{\text{max}} = 0.567$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.019$ 235 parameters
 $wR(F^2) = 0.049$ H-atom parameters constrained
 $S = 1.04$ $\Delta\rho_{\text{max}} = 1.08$ e Å⁻³
 5264 reflections $\Delta\rho_{\text{min}} = -0.80$ e Å⁻³

Data collection: SMART-NT (Bruker, 1998); cell refinement: SAINT-Plus (Bruker, 1999); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 1997) and Mercury (Macrae *et al.*, 2008); software used to prepare material for publication: WinGX (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2412).

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supplementary materials

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{ μ -1,2-Bis[bis(4-methoxyphenyl)phosphanyl]-1,2-dimethylhydrazine- κ^2 P:P'}bis[chloridogold(I)] tetrahydrofuran disolvate

F. H. Kriel, M. A. Fernandes and J. Coates

Comment

The title compound ($C_{30}H_{34}Au_2Cl_2N_2O_4P_2 \cdot 2(C_4H_8O)$), formed from a bidentate phosphine ligand complexed to two linear gold(I) nuclei, readily crystallizes out of dichloromethane (DCM) with the addition of a few drops of tetrahydrofuran (THF). The crystal structure includes a THF solvent molecule. The complex molecule is bisected by a two fold axis through the N-N' and Au-Au' lines (Fig 1). Gold(I) has an almost linear coordination with a P—Au—Cl angle of 175.68 (3) °. The Au—Au distance within the complex is 3.122 (2) Å, well within the range of aurophilic interactions (described in Holleman *et al.*, 2001, as being normally between 2.7 Å and 3.4 Å). Other bond lengths are within expected ranges.

The structure exhibits columns of complexes arranged head-to-tail along b, forming channels filled with THF. There is an intercolumnar contact involving chloride atoms in one molecule and hydrogen atoms on the methyl substituted hydrazine bridge of a neighbouring one, in the same column (Cl1 \cdots H1cⁱ: 2.892Å, (i): 1-x, 1+y, 1/2-z, site A in Fig 2). There are also weak intercolumnar H-bonding contacts (O1 \cdots H13ⁱⁱ: 2.629Å, (ii): 3/2-x, -1/2-y, 1-z, site B in Fig 2). Finally, the THF solvato molecule is weakly attached to the columns by a pair of O \cdots H contacts (O3 \cdots H15: 2.608 Å; O3 \cdots H15b: 2.557 Å) (site C in Fig. 2).

Experimental

The complex was synthesized by dissolving tetrahydrothiophenogold(I) chloride [(THT)AuCl] in DCM and adding 0.5 equivalents of the corresponding ligand (bis(di(4-methoxyphenyl)phosphino)-1,2-dimethylhydrazine). The addition of a few drops of THF led to the growth of crystals suitable for use in single-crystal X-Ray analysis. The presence of THF during the initial complexation led to undesirable side products as a result of the breakdown of the ligand.

Refinement

The H atoms were positioned geometrically and allowed to ride on their respective parent atoms, with C—H = 0.93 (CH) or 0.96 (CH₃) Å, and with U_{eq} = 1.2 (CH) or 1.5 (CH₃) $U_{eq}(C)$.

Figures

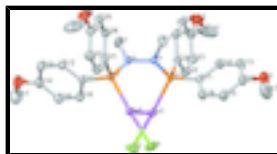


Fig. 1. : Molecular structure of **I** drawn with displacement ellipsoids at the 50% probability level. Hydrogen atoms and solvent THF have been omitted for clarity.

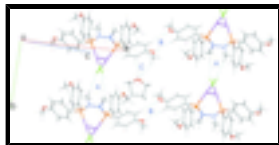


Fig. 2. : Packing of compound **I** showing short contacts.

{ μ -1,2-Bis[bis(4-methoxyphenyl)phosphanyl]-1,2-dimethylhydrazine- $\kappa^2P:P'$ }bis[chloridogold(I)] tetrahydrofuran disolvate

Crystal data

| | |
|--|---|
| $[\text{Au}_2\text{Cl}_2(\text{C}_{30}\text{H}_{34}\text{N}_2\text{O}_4\text{P}_2)] \cdot 2\text{C}_4\text{H}_8\text{O}$ | $F(000) = 2248$ |
| $M_r = 1157.57$ | $D_x = 1.806 \text{ Mg m}^{-3}$ |
| Monoclinic, $C2/c$ | Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$ |
| Hall symbol: $-C 2yc$ | Cell parameters from 7327 reflections |
| $a = 23.208 (5) \text{ \AA}$ | $\theta = 2.4\text{--}28.3^\circ$ |
| $b = 9.080 (5) \text{ \AA}$ | $\mu = 7.13 \text{ mm}^{-1}$ |
| $c = 20.220 (5) \text{ \AA}$ | $T = 173 \text{ K}$ |
| $\beta = 92.414 (5)^\circ$ | Plate, colourless |
| $V = 4257 (3) \text{ \AA}^3$ | $0.58 \times 0.45 \times 0.10 \text{ mm}$ |
| $Z = 4$ | |

Data collection

| | |
|---|--|
| Bruker SMART CCD area-detector diffractometer | 5264 independent reflections |
| Radiation source: fine-focus sealed tube graphite | 4679 reflections with $I > 2\sigma(I)$ |
| phi and ω scans | $R_{\text{int}} = 0.040$ |
| Absorption correction: multi-scan (<i>SADABS</i> ; Bruker, 1999) | $\theta_{\text{max}} = 28.3^\circ$, $\theta_{\text{min}} = 1.8^\circ$ |
| $T_{\text{min}} = 0.044$, $T_{\text{max}} = 0.567$ | $h = -30 \rightarrow 30$ |
| 33198 measured reflections | $k = -12 \rightarrow 11$ |
| | $l = -25 \rightarrow 26$ |

Refinement

| | |
|---------------------------------|--|
| Refinement on F^2 | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.019$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.049$ | H-atom parameters constrained |
| $S = 1.04$ | $w = 1/[\sigma^2(F_o^2) + (0.0199P)^2 + 7.2119P]$ |
| 5264 reflections | where $P = (F_o^2 + 2F_c^2)/3$ |
| 235 parameters | $(\Delta/\sigma)_{\text{max}} = 0.004$ |
| 0 restraints | $\Delta\rho_{\text{max}} = 1.08 \text{ e \AA}^{-3}$ |
| | $\Delta\rho_{\text{min}} = -0.80 \text{ e \AA}^{-3}$ |

Special details

Experimental. Reaction: bis(di(4-methoxyphenyl)phosphino)-1,2-dimethylhydrazine: 167 mg (0.29 mmol), (THT)AuCl: 200 mg (0.57 mmol), dichloromethane: 5 ml, tetrahydrofuran: few drops, Yield: 75%. Colourless to grey crystals. ^1H NMR: (CDCl_3 , 300 MHz) δ_{H} 7.79 (t, Arom, $J = 8.2$ Hz, 4H) 7.41 (t, Arom, $J = 8.2$ Hz, 4H), 6.97 (d, Arom, $J = 7.6$ Hz, 4H), 6.81 (d, Arom, $J = 7.6$ Hz, 4H), 3.86 (s, OMe, 6H), 3.81 (s, OMe, 6H), 2.69 (d, NCH_3 , $^3J(^1\text{H}-^{31}\text{P}) = 5.8$ Hz, 6H). ^{13}C NMR: (CDCl_3 , 75 MHz) δ_{C} 163.5 (d, Arom, $J = 33.4$ Hz), 135.9 (m, Arom), 115.3 (m, Arom), 55.52 and 55.45 (s, OCH_3), 35.1 (s, NCH_3). ^{31}P NMR: (CDCl_3 , 121 MHz) δ_{P} 85.1. MS: 977 (83%, $M - \text{Cl}$), 245 (80%, $\text{P}(\text{PhOMe})_2$). EA: Calc: ($\text{Au}_2\text{Cl}_2\text{P}_2\text{O}_4\text{N}_2\text{C}_{30}\text{H}_{34}$) C 35.56%, H 3.38%, N 2.76%. Found: C 36.78%, H 3.79%, N 2.47%. MP: 166 – 170 °C.

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|-------------|--------------|----------------------------------|
| C1 | 0.56719 (14) | -0.3148 (4) | 0.26405 (15) | 0.0437 (8) |
| H1A | 0.5974 | -0.3050 | 0.2976 | 0.066* |
| H1B | 0.5830 | -0.3040 | 0.2212 | 0.066* |
| H1C | 0.5497 | -0.4102 | 0.2673 | 0.066* |
| C11 | 0.58836 (10) | -0.0866 (3) | 0.37553 (12) | 0.0238 (5) |
| C12 | 0.60878 (11) | -0.1775 (3) | 0.42700 (13) | 0.0316 (6) |
| H12 | 0.5836 | -0.2413 | 0.4473 | 0.038* |
| C13 | 0.66594 (12) | -0.1732 (3) | 0.44792 (14) | 0.0383 (6) |
| H13 | 0.6794 | -0.2351 | 0.4818 | 0.046* |
| C14 | 0.70353 (11) | -0.0767 (3) | 0.41852 (15) | 0.0360 (6) |
| C15 | 0.68388 (11) | 0.0165 (3) | 0.36871 (13) | 0.0337 (6) |
| H15 | 0.7089 | 0.0826 | 0.3496 | 0.040* |
| C16 | 0.62614 (11) | 0.0104 (3) | 0.34742 (12) | 0.0287 (5) |
| H16 | 0.6127 | 0.0727 | 0.3137 | 0.034* |
| C17 | 0.80122 (14) | -0.0045 (5) | 0.4075 (2) | 0.0714 (12) |
| H17A | 0.8385 | -0.0180 | 0.4291 | 0.107* |
| H17B | 0.7916 | 0.0983 | 0.4069 | 0.107* |
| H17C | 0.8019 | -0.0405 | 0.3629 | 0.107* |
| C21 | 0.47826 (10) | -0.2085 (3) | 0.40186 (12) | 0.0237 (5) |
| C22 | 0.45316 (11) | -0.1382 (3) | 0.45396 (13) | 0.0287 (5) |
| H22 | 0.4534 | -0.0358 | 0.4559 | 0.034* |
| C23 | 0.42742 (12) | -0.2182 (3) | 0.50370 (13) | 0.0336 (6) |
| H23 | 0.4113 | -0.1699 | 0.5390 | 0.040* |
| C24 | 0.42619 (11) | -0.3700 (3) | 0.49983 (14) | 0.0299 (6) |

supplementary materials

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|------|--------------|---------------|--------------|--------------|
| C25 | 0.45039 (11) | -0.4422 (3) | 0.44695 (13) | 0.0283 (5) |
| H25 | 0.4490 | -0.5444 | 0.4442 | 0.034* |
| C26 | 0.47638 (11) | -0.3615 (3) | 0.39880 (13) | 0.0271 (5) |
| H26 | 0.4929 | -0.4100 | 0.3638 | 0.032* |
| C27 | 0.3811 (2) | -0.3925 (4) | 0.6031 (2) | 0.0793 (16) |
| H27A | 0.3653 | -0.4664 | 0.6310 | 0.119* |
| H27B | 0.3517 | -0.3223 | 0.5906 | 0.119* |
| H27C | 0.4122 | -0.3431 | 0.6267 | 0.119* |
| C31 | 0.7520 (3) | 0.4212 (6) | 0.2921 (3) | 0.1022 (19) |
| H31A | 0.7121 | 0.4137 | 0.2760 | 0.123* |
| H31B | 0.7767 | 0.3897 | 0.2574 | 0.123* |
| C32 | 0.7659 (4) | 0.5757 (6) | 0.3128 (3) | 0.127 (3) |
| H32A | 0.7899 | 0.6231 | 0.2809 | 0.152* |
| H32B | 0.7309 | 0.6330 | 0.3167 | 0.152* |
| C33 | 0.7965 (3) | 0.5625 (8) | 0.3765 (3) | 0.124 (2) |
| H33A | 0.8348 | 0.6048 | 0.3751 | 0.149* |
| H33B | 0.7757 | 0.6114 | 0.4108 | 0.149* |
| C34 | 0.7993 (5) | 0.4148 (8) | 0.3874 (6) | 0.240 (8) |
| H34A | 0.7915 | 0.3953 | 0.4333 | 0.288* |
| H34B | 0.8381 | 0.3809 | 0.3799 | 0.288* |
| N1 | 0.52377 (8) | -0.2011 (2) | 0.27315 (10) | 0.0240 (4) |
| O1 | 0.75927 (9) | -0.0834 (3) | 0.44229 (12) | 0.0507 (6) |
| O2 | 0.40217 (9) | -0.4604 (2) | 0.54504 (10) | 0.0417 (5) |
| O3 | 0.76126 (16) | 0.3366 (4) | 0.3477 (2) | 0.0940 (11) |
| P1 | 0.51542 (3) | -0.10079 (7) | 0.34180 (3) | 0.02187 (13) |
| Cl1 | 0.41744 (3) | 0.31848 (8) | 0.29583 (4) | 0.04364 (17) |
| Au1 | 0.470213 (4) | 0.109672 (10) | 0.317798 (4) | 0.02473 (4) |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C1 | 0.0542 (17) | 0.0412 (18) | 0.0345 (15) | 0.0252 (15) | -0.0130 (13) | -0.0115 (13) |
| C11 | 0.0244 (11) | 0.0244 (13) | 0.0223 (12) | 0.0004 (9) | -0.0026 (9) | -0.0014 (9) |
| C12 | 0.0331 (13) | 0.0307 (15) | 0.0305 (13) | -0.0038 (11) | -0.0052 (10) | 0.0078 (11) |
| C13 | 0.0369 (14) | 0.0387 (17) | 0.0382 (15) | -0.0021 (13) | -0.0091 (11) | 0.0123 (13) |
| C14 | 0.0283 (12) | 0.0405 (17) | 0.0384 (16) | -0.0014 (12) | -0.0084 (11) | 0.0013 (13) |
| C15 | 0.0300 (13) | 0.0370 (16) | 0.0342 (14) | -0.0065 (11) | 0.0013 (11) | 0.0036 (12) |
| C16 | 0.0310 (12) | 0.0309 (14) | 0.0241 (12) | 0.0002 (11) | -0.0003 (10) | 0.0053 (10) |
| C17 | 0.0293 (15) | 0.090 (3) | 0.094 (3) | -0.0153 (18) | -0.0118 (17) | 0.027 (2) |
| C21 | 0.0246 (11) | 0.0223 (13) | 0.0239 (12) | -0.0024 (9) | -0.0029 (9) | 0.0021 (9) |
| C22 | 0.0330 (13) | 0.0221 (13) | 0.0309 (14) | -0.0019 (10) | 0.0028 (10) | -0.0007 (10) |
| C23 | 0.0398 (14) | 0.0312 (15) | 0.0304 (14) | 0.0001 (12) | 0.0081 (11) | -0.0017 (11) |
| C24 | 0.0275 (12) | 0.0299 (15) | 0.0326 (14) | -0.0018 (10) | 0.0026 (10) | 0.0079 (11) |
| C25 | 0.0303 (12) | 0.0209 (13) | 0.0333 (14) | -0.0026 (10) | -0.0026 (10) | 0.0009 (11) |
| C26 | 0.0287 (12) | 0.0235 (13) | 0.0289 (13) | 0.0007 (10) | -0.0001 (10) | -0.0006 (10) |
| C27 | 0.114 (4) | 0.055 (3) | 0.074 (3) | 0.018 (2) | 0.062 (3) | 0.026 (2) |
| C31 | 0.154 (6) | 0.091 (4) | 0.064 (3) | -0.039 (4) | 0.022 (3) | 0.004 (3) |
| C32 | 0.229 (8) | 0.068 (4) | 0.085 (4) | -0.009 (4) | 0.042 (5) | 0.008 (3) |

| | | | | | | |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C33 | 0.185 (7) | 0.100 (5) | 0.090 (5) | -0.055 (5) | 0.018 (4) | -0.011 (4) |
| C34 | 0.248 (11) | 0.096 (6) | 0.356 (17) | -0.057 (6) | -0.225 (12) | 0.061 (7) |
| N1 | 0.0299 (10) | 0.0220 (11) | 0.0196 (10) | 0.0040 (8) | -0.0054 (8) | -0.0014 (8) |
| O1 | 0.0295 (10) | 0.0591 (15) | 0.0620 (15) | -0.0089 (10) | -0.0154 (10) | 0.0151 (12) |
| O2 | 0.0475 (11) | 0.0327 (12) | 0.0462 (12) | 0.0009 (9) | 0.0172 (9) | 0.0131 (9) |
| O3 | 0.100 (2) | 0.061 (2) | 0.118 (3) | -0.0244 (18) | -0.025 (2) | 0.022 (2) |
| P1 | 0.0249 (3) | 0.0202 (3) | 0.0203 (3) | -0.0005 (2) | -0.0015 (2) | 0.0000 (2) |
| Cl1 | 0.0563 (4) | 0.0254 (4) | 0.0495 (4) | 0.0139 (3) | 0.0055 (3) | 0.0033 (3) |
| Au1 | 0.03095 (6) | 0.01863 (6) | 0.02458 (6) | 0.00191 (4) | 0.00082 (4) | -0.00061 (3) |

Geometric parameters (Å, °)

| | | | |
|-------------|-------------|----------------------|-------------|
| C1—N1 | 1.460 (3) | C24—O2 | 1.365 (3) |
| C1—H1A | 0.9600 | C24—C25 | 1.393 (4) |
| C1—H1B | 0.9600 | C25—C26 | 1.378 (4) |
| C1—H1C | 0.9600 | C25—H25 | 0.9300 |
| C11—C16 | 1.382 (4) | C26—H26 | 0.9300 |
| C11—C12 | 1.395 (4) | C27—O2 | 1.430 (4) |
| C11—P1 | 1.803 (2) | C27—H27A | 0.9600 |
| C12—C13 | 1.376 (4) | C27—H27B | 0.9600 |
| C12—H12 | 0.9300 | C27—H27C | 0.9600 |
| C13—C14 | 1.388 (4) | C31—O3 | 1.371 (6) |
| C13—H13 | 0.9300 | C31—C32 | 1.495 (7) |
| C14—O1 | 1.362 (3) | C31—H31A | 0.9700 |
| C14—C15 | 1.378 (4) | C31—H31B | 0.9700 |
| C15—C16 | 1.391 (3) | C32—C33 | 1.449 (9) |
| C15—H15 | 0.9300 | C32—H32A | 0.9700 |
| C16—H16 | 0.9300 | C32—H32B | 0.9700 |
| C17—O1 | 1.419 (4) | C33—C34 | 1.361 (9) |
| C17—H17A | 0.9600 | C33—H33A | 0.9700 |
| C17—H17B | 0.9600 | C33—H33B | 0.9700 |
| C17—H17C | 0.9600 | C34—O3 | 1.366 (7) |
| C21—C22 | 1.382 (4) | C34—H34A | 0.9700 |
| C21—C26 | 1.391 (4) | C34—H34B | 0.9700 |
| C21—P1 | 1.807 (2) | N1—N1 ⁱ | 1.417 (4) |
| C22—C23 | 1.396 (4) | N1—P1 | 1.678 (2) |
| C22—H22 | 0.9300 | P1—Au1 | 2.2238 (11) |
| C23—C24 | 1.380 (4) | Cl1—Au1 | 2.2905 (11) |
| C23—H23 | 0.9300 | Au1—Au1 ⁱ | 3.1222 (17) |
| N1—C1—H1A | 109.5 | C21—C26—H26 | 119.6 |
| N1—C1—H1B | 109.5 | O2—C27—H27A | 109.5 |
| H1A—C1—H1B | 109.5 | O2—C27—H27B | 109.5 |
| N1—C1—H1C | 109.5 | H27A—C27—H27B | 109.5 |
| H1A—C1—H1C | 109.5 | O2—C27—H27C | 109.5 |
| H1B—C1—H1C | 109.5 | H27A—C27—H27C | 109.5 |
| C16—C11—C12 | 118.8 (2) | H27B—C27—H27C | 109.5 |
| C16—C11—P1 | 119.45 (19) | O3—C31—C32 | 105.7 (5) |
| C12—C11—P1 | 121.61 (19) | O3—C31—H31A | 110.6 |

supplementary materials

| | | | |
|-----------------|-------------|----------------------------|--------------|
| C13—C12—C11 | 120.4 (3) | C32—C31—H31A | 110.6 |
| C13—C12—H12 | 119.8 | O3—C31—H31B | 110.6 |
| C11—C12—H12 | 119.8 | C32—C31—H31B | 110.6 |
| C12—C13—C14 | 120.1 (3) | H31A—C31—H31B | 108.7 |
| C12—C13—H13 | 120.0 | C33—C32—C31 | 105.2 (5) |
| C14—C13—H13 | 120.0 | C33—C32—H32A | 110.7 |
| O1—C14—C15 | 124.4 (3) | C31—C32—H32A | 110.7 |
| O1—C14—C13 | 115.2 (3) | C33—C32—H32B | 110.7 |
| C15—C14—C13 | 120.4 (2) | C31—C32—H32B | 110.7 |
| C14—C15—C16 | 119.1 (3) | H32A—C32—H32B | 108.8 |
| C14—C15—H15 | 120.4 | C34—C33—C32 | 104.1 (6) |
| C16—C15—H15 | 120.4 | C34—C33—H33A | 110.9 |
| C11—C16—C15 | 121.2 (2) | C32—C33—H33A | 110.9 |
| C11—C16—H16 | 119.4 | C34—C33—H33B | 110.9 |
| C15—C16—H16 | 119.4 | C32—C33—H33B | 110.9 |
| O1—C17—H17A | 109.5 | H33A—C33—H33B | 108.9 |
| O1—C17—H17B | 109.5 | C33—C34—O3 | 113.0 (6) |
| H17A—C17—H17B | 109.5 | C33—C34—H34A | 109.0 |
| O1—C17—H17C | 109.5 | O3—C34—H34A | 109.0 |
| H17A—C17—H17C | 109.5 | C33—C34—H34B | 109.0 |
| H17B—C17—H17C | 109.5 | O3—C34—H34B | 109.0 |
| C22—C21—C26 | 118.8 (2) | H34A—C34—H34B | 107.8 |
| C22—C21—P1 | 119.4 (2) | N1 ⁱ —N1—C1 | 116.05 (18) |
| C26—C21—P1 | 121.73 (19) | N1 ⁱ —N1—P1 | 115.65 (17) |
| C21—C22—C23 | 121.0 (3) | C1—N1—P1 | 126.33 (17) |
| C21—C22—H22 | 119.5 | C14—O1—C17 | 117.6 (3) |
| C23—C22—H22 | 119.5 | C24—O2—C27 | 117.0 (3) |
| C24—C23—C22 | 119.2 (3) | C34—O3—C31 | 105.4 (5) |
| C24—C23—H23 | 120.4 | N1—P1—C11 | 102.26 (11) |
| C22—C23—H23 | 120.4 | N1—P1—C21 | 109.71 (12) |
| O2—C24—C23 | 124.8 (3) | C11—P1—C21 | 104.57 (11) |
| O2—C24—C25 | 114.8 (3) | N1—P1—Au1 | 110.83 (8) |
| C23—C24—C25 | 120.4 (2) | C11—P1—Au1 | 116.63 (9) |
| C26—C25—C24 | 119.6 (3) | C21—P1—Au1 | 112.20 (9) |
| C26—C25—H25 | 120.2 | P1—Au1—Cl1 | 175.68 (3) |
| C24—C25—H25 | 120.2 | P1—Au1—Au1 ⁱ | 88.241 (19) |
| C25—C26—C21 | 120.9 (2) | Cl1—Au1—Au1 ⁱ | 94.74 (2) |
| C25—C26—H26 | 119.5 | | |
| C16—C11—C12—C13 | -1.9 (4) | C13—C14—O1—C17 | 170.4 (3) |
| P1—C11—C12—C13 | 174.3 (2) | C23—C24—O2—C27 | -5.4 (5) |
| C11—C12—C13—C14 | 1.0 (5) | C25—C24—O2—C27 | 174.8 (3) |
| C12—C13—C14—O1 | -178.9 (3) | C33—C34—O3—C31 | 26.4 (12) |
| C12—C13—C14—C15 | 0.6 (5) | C32—C31—O3—C34 | -23.6 (9) |
| O1—C14—C15—C16 | 178.3 (3) | N1 ⁱ —N1—P1—C11 | -165.00 (15) |
| C13—C14—C15—C16 | -1.3 (5) | C1—N1—P1—C11 | 31.7 (3) |
| C12—C11—C16—C15 | 1.3 (4) | N1 ⁱ —N1—P1—C21 | 84.44 (16) |
| P1—C11—C16—C15 | -175.0 (2) | C1—N1—P1—C21 | -78.9 (3) |

| | | | |
|-----------------|-------------|----------------------------|--------------|
| C14—C15—C16—C11 | 0.3 (4) | N1 ⁱ —N1—P1—Au1 | -40.01 (16) |
| C26—C21—C22—C23 | 1.4 (4) | C1—N1—P1—Au1 | 156.7 (2) |
| P1—C21—C22—C23 | -175.7 (2) | C16—C11—P1—N1 | 76.7 (2) |
| C21—C22—C23—C24 | -1.3 (4) | C12—C11—P1—N1 | -99.5 (2) |
| C22—C23—C24—O2 | -179.7 (3) | C16—C11—P1—C21 | -168.9 (2) |
| C22—C23—C24—C25 | 0.1 (4) | C12—C11—P1—C21 | 14.9 (3) |
| O2—C24—C25—C26 | -179.3 (2) | C16—C11—P1—Au1 | -44.4 (2) |
| C23—C24—C25—C26 | 0.9 (4) | C12—C11—P1—Au1 | 139.4 (2) |
| C24—C25—C26—C21 | -0.7 (4) | C22—C21—P1—N1 | -162.70 (19) |
| C22—C21—C26—C25 | -0.5 (4) | C26—C21—P1—N1 | 20.2 (2) |
| P1—C21—C26—C25 | 176.63 (19) | C22—C21—P1—C11 | 88.3 (2) |
| O3—C31—C32—C33 | 14.1 (8) | C26—C21—P1—C11 | -88.8 (2) |
| C31—C32—C33—C34 | 1.1 (10) | C22—C21—P1—Au1 | -39.0 (2) |
| C32—C33—C34—O3 | -16.7 (13) | C26—C21—P1—Au1 | 143.88 (19) |
| C15—C14—O1—C17 | -9.2 (5) | | |

Symmetry codes: (i) $-x+1, y, -z+1/2$.

Fig. 1

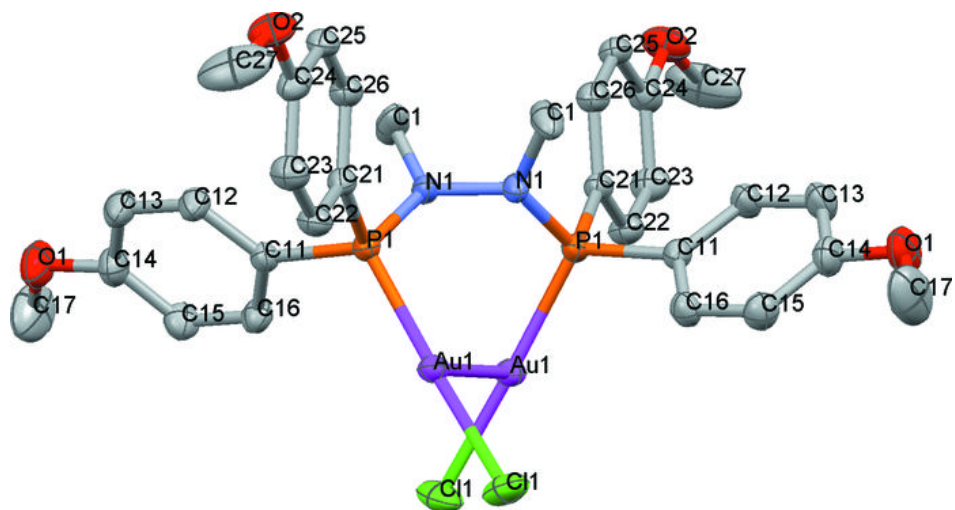


Fig. 2

