

## Bis[ $\mu$ -1,3-bis(diphenylphosphanyl)-propane- $\kappa^2P:P'$ ]digold(I) tetrachlorido-nickelate(II) diethyl ether monosolvate

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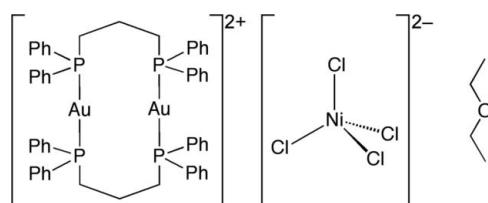
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Key indicators: single-crystal X-ray study;  $T = 200\text{ K}$ ; mean  $\sigma(\text{C}-\text{C}) = 0.009\text{ \AA}$ ; disorder in main residue;  $R$  factor = 0.048;  $wR$  factor = 0.083; data-to-parameter ratio = 19.4.

The title compound,  $[\text{Au}_2(\text{C}_{27}\text{H}_{26}\text{P}_2)_2][\text{NiCl}_4]\cdot\text{C}_4\text{H}_{10}\text{O}$ , consists of a digold(I) complex cation, an  $[\text{NiCl}_4]^{2-}$  complex anion and a diethyl ether solvent molecule. Two 1,3-bis(diphenylphosphanyl)propane (dppp) ligands bridge two  $\text{Au}^{\text{I}}$  atoms, forming a metallacycle in which each of the  $\text{Au}^{\text{I}}$  atoms is coordinated in a slightly distorted linear environment by two P atoms. In the complex anion, the  $\text{Ni}^{\text{II}}$  atom is coordinated by four chloride ligands in a distorted tetrahedral geometry. The complex cation and the complex anion form a cation–anion pair through two  $\text{Au}\cdots\text{Cl}$  contacts of 3.040 (1) and 3.021 (2)  $\text{\AA}$ . One of the phenyl groups of the dppp ligand is disordered over two positions with equal occupancies.

### Related literature

For closely related structures, see: Gruber & Jansen (2010); Brandys & Puddephatt (2001). For related studies, see: Igashira-Kamiyama *et al.* (2012); Lee *et al.* (2012); Lim *et al.* (2011); Hashimoto *et al.* (2010). For the starting material, see: Howard-Lock *et al.* (1986); Blondeau *et al.* (1967); Mirabelli *et al.* (1987). For a description of the Cambridge Structural Database, see: Allen (2002).



### Experimental

#### Crystal data

$[\text{Au}_2(\text{C}_{27}\text{H}_{26}\text{P}_2)_2][\text{NiCl}_4]\cdot\text{C}_4\text{H}_{10}\text{O}$

$M_r = 1493.40$

Monoclinic,  $P2_1/n$   
 $a = 18.9290 (5)\text{ \AA}$   
 $b = 16.1945 (7)\text{ \AA}$   
 $c = 19.0895 (17)\text{ \AA}$   
 $\beta = 97.368 (7)^\circ$   
 $V = 5803.5 (6)\text{ \AA}^3$

$Z = 4$   
Mo  $K\alpha$  radiation  
 $\mu = 5.70\text{ mm}^{-1}$   
 $T = 200\text{ K}$   
 $0.28 \times 0.05 \times 0.03\text{ mm}$

#### Data collection

Rigaku R-AXIS VII diffractometer  
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)  
 $T_{\min} = 0.580$ ,  $T_{\max} = 0.773$   
45449 measured reflections  
13273 independent reflections  
10666 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.042$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$   
 $wR(F^2) = 0.083$   
 $S = 1.14$   
13273 reflections  
685 parameters  
78 restraints  
H-atom parameters constrained  
 $\Delta\rho_{\max} = 1.16\text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.88\text{ e \AA}^{-3}$

**Table 1**  
Selected bond lengths ( $\text{\AA}$ ).

|        |             |         |             |
|--------|-------------|---------|-------------|
| Au1—P1 | 2.3109 (13) | Ni1—Cl4 | 2.2359 (15) |
| Au1—P3 | 2.3129 (13) | Ni1—Cl2 | 2.2548 (16) |
| Au2—P2 | 2.3013 (13) | Ni1—Cl1 | 2.2558 (15) |
| Au2—P4 | 2.3050 (13) | Ni1—Cl3 | 2.2780 (14) |

Data collection: *PROCESS-AUTO* (Rigaku, 2000); cell refinement: *PROCESS-AUTO*; data reduction: *PROCESS-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Yadokari-XG 2009* (Kabuto *et al.*, 2009); software used to prepare material for publication: *Yadokari-XG 2009*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2571).

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# supporting information

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## Bis[ $\mu$ -1,3-bis(diphenylphosphanyl)propane- $\kappa^2P:P'$ ]digold(I) tetrachloridonickelate(II) diethyl ether monosolvate

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### S1. Comment

In recent years, diphosphine-bridged cyclic digold(I) complexes have attracted considerable attention because of their intriguing photophysical properties that originate from the presence of an aurophilic interaction between gold(I) ions. A typical example is  $[Au_2(dppm)_2]^{2+}$  ( $dppm = \text{bis}(\text{diphenylphosphanyl})\text{methane}$ ), which is easily prepared from  $dppm$  having a methylene linker between two P atoms, in combination with an  $Au^I$  ion. Analogous digold(I) complexes having a longer linker between two P atoms, such as 1,2-bis(diphenylphosphanyl)ethane ( $dppe$ ) and 1,3-bis(diphenylphosphanyl)propane ( $dppp$ ), have also been prepared, but only a few of them have been structurally characterized to date (Allen, 2002).

Recently, we started to investigate the creation of diphosphine-bridged digold(I) metalloligands and their coordination behavior toward various metal ions, with the aim of the rational construction of heterometallic multinuclear and metallosupramolecular structures (Lee *et al.*, 2012; Igashira-Kamiyama *et al.*, 2012; Hashimoto *et al.*, 2010). In the course of this study, we found the formation of a diphosphine-bridged digold(I) complex,  $[Au_2(dppp)_2]^{2+}$  ( $dppp = 1,3\text{-bis}(\text{diphenylphosphanyl})\text{propane}$ ), which is cocrystallized with  $[NiCl_4]^{2-}$  to give single-crystals of  $[Au_2(dppp)_2][NiCl_4].Et_2O$  (I). Here, we report the synthesis and crystal structure of (I).

Treatment of  $[Au_2Cl_2(dppp)]$  (Mirabelli *et al.*, 1987) with 1 equiv of  $N,N'$ -ethylene-bis-*D*-penicillamine (Howard-Lock *et al.*, 1986; Blondeau *et al.*, 1967) in ethanol/water in the presence of KOH gave a white powder. When this powder was reacted with  $NiCl_2.6H_2O$  in ethanol, blue needle crystals of  $[Au_2(dppp)_2][NiCl_4].Et_2O$  (I) were obtained by the vapor diffusion of diethylether into the reaction solution.

Compound (I) crystallized in a centrosymmetric space group  $P2_1/n$ , which contains one digold(I) complex cation, one nickel(II) complex anion and one solvate diethylether molecule in the asymmetric unit. The complex cation has a cyclic digold(I) structure in  $[Au_2(dppp)_2]^{2+}$ , in which two  $dppp$  ligands bridge two  $Au^I$  atoms. Each  $Au^I$  atom adopts a slightly distorted linear geometry ( $176.68(5)^\circ, 177.98(5)^\circ$ ) coordinated by two P atoms from two different  $dppp$  ligands (Fig. 1). The Au–P bond distances ( $2.3013(13)$ – $2.3129(13)$  Å) are within the range observed for  $[Au_2(dppp)_2][Mo_8O_{26}]$ ,  $[Au_2(dppp)_2][PMo_8O_{26}]$  and  $[Au_2(dppp)_2](CF_3CO_2)_2$  ( $Au-P = 2.271$ – $2.324$  Å) (Gruber *et al.*, 2010; Brandys *et al.*, 2001). In the complex-cation of (I), two  $Au^I$  atoms and four P atoms are deviated from co-planarity, with the dihedral angle between two  $Au_2P_2$  planes being  $21.94(2)^\circ$ . This structural feature is different from those found in  $[Au_2(dppp)_2][Mo_8O_{26}]$ ,  $[Au_2(dppp)_2][PMo_8O_{26}]$  and  $[Au_2(dppp)_2](CF_3CO_2)_2$ , in which all the  $Au^I$  and P atoms in each complex-cation are located nearly on the same plane (Gruber *et al.*, 2010; Brandys *et al.*, 2001). The distance between two  $Au^I$  atoms in  $[Au_2(dppp)_2]^{2+}$  is  $5.5424(4)$  Å, indicative of the absence of an aurophilic interaction. In (I), a nickel(II) complex-anion,  $[NiCl_4]^{2-}$ , which has a distorted tetrahedral geometry, coexists with  $[Au_2(dppp)_2]^{2+}$  to balance the total charge. Note that  $[Au_2(dppp)_2]^{2+}$  and  $[NiCl_4]^{2-}$  are connected to each other through two  $Au\cdots Cl$  bonding interactions

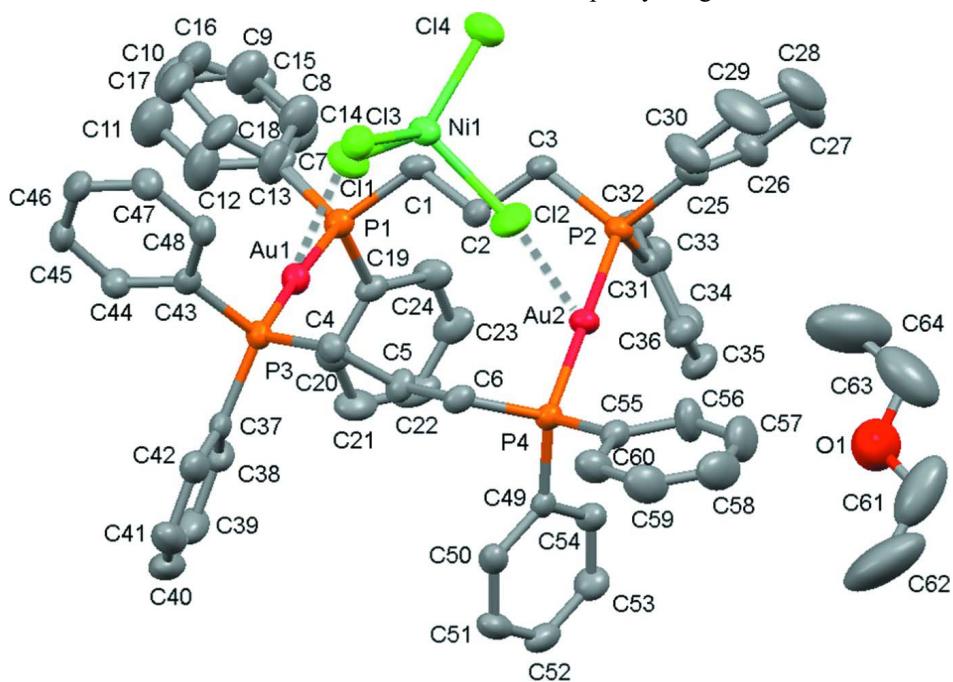
(3.040 (1) Å and 3.021 (2) Å), while any other significant intermolecular interactions do not exist in (I) (Fig. 2).

## S2. Experimental

To a white suspension containing 0.15 g (0.17 mmol) of  $[\text{Au}_2\text{Cl}_2(\text{dppp})]$  in 300 ml of EtOH was added 0.056 g (0.17 mmol) of *N,N'*-ethylene-bis-*D*-penicillamine dissolved in 3 ml of a 0.1 M KOH aqueous solution. The mixture was stirred at room temperature for 3 h. The resulting colorless solution was evaporated to dryness, and then the residue was washed with H<sub>2</sub>O to give a white powder (0.15 g). When 2.5 mg (0.01 mmol) of NiCl<sub>2</sub>.6H<sub>2</sub>O was added to a suspension of this white powder (10 mg) in 1 ml of EtOH, followed by stirring at room temperature for 3 h, a clear red orange solution was obtained. Vapor diffusion of diethylether into the resulting red orange solution afforded a small amount of blue needle crystals of  $[\text{Au}_2(\text{dppp})_2]\text{[NiCl}_4\text{]}\cdot\text{Et}_2\text{O}$ .

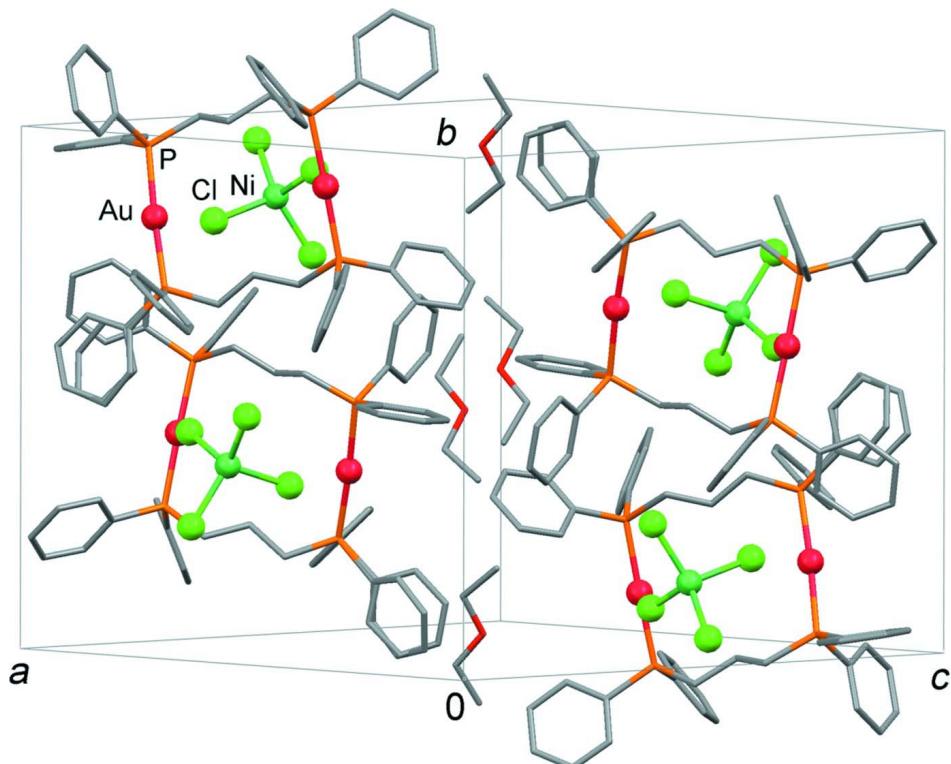
## S3. Refinement

H atoms were placed at calculated positions [C–H = 0.99 (methylene) or 0.95 Å (phenyl)] and refined as riding with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ . One phenyl ring is disordered over two positions (C7–C12 and C13–C18) with site occupancies of 0.5. The FLAT and SIMU restraints were used to model the disordered phenyl ring.



**Figure 1**

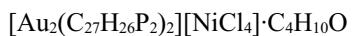
A view of the molecular structure of the title compound with the atom-numbering scheme. H atoms are omitted for clarity.

**Figure 2**

Crystal packing of the title compound. H atoms are omitted for clarity.

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#### Crystal data



$M_r = 1493.40$

Monoclinic,  $P2_1/n$

Hall symbol: -P 2yn

$a = 18.9290 (5)$  Å

$b = 16.1945 (7)$  Å

$c = 19.0895 (17)$  Å

$\beta = 97.368 (7)^\circ$

$V = 5803.5 (6)$  Å<sup>3</sup>

$Z = 4$

$F(000) = 2928$

$D_x = 1.709 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71075$  Å

Cell parameters from 2299 reflections

$\theta = 4.1\text{--}27.6^\circ$

$\mu = 5.70 \text{ mm}^{-1}$

$T = 200$  K

Needle, blue

$0.28 \times 0.05 \times 0.03$  mm

#### Data collection

Rigaku R-AXIS VII  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 10.000 pixels mm<sup>-1</sup>

$\omega$  scans

Absorption correction: multi-scan  
(*ABSCOR*; Higashi, 1995)

$T_{\min} = 0.580$ ,  $T_{\max} = 0.773$

45449 measured reflections

13273 independent reflections

10666 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.042$

$\theta_{\max} = 27.5^\circ$ ,  $\theta_{\min} = 3.0^\circ$

$h = -22 \rightarrow 24$

$k = -20 \rightarrow 21$

$l = -24 \rightarrow 24$

*Refinement*Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.048$  $wR(F^2) = 0.083$  $S = 1.14$ 

13273 reflections

685 parameters

78 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0332P)^2 + 5.0632P]$   
where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\max} = 0.001$  $\Delta\rho_{\max} = 1.16 \text{ e } \text{\AA}^{-3}$  $\Delta\rho_{\min} = -0.88 \text{ e } \text{\AA}^{-3}$ *Special details*

**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 ( $\text{\AA}^2$ )*

|     | <i>x</i>      | <i>y</i>      | <i>z</i>      | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|-----|---------------|---------------|---------------|----------------------------------|-----------|
| Au1 | 0.779915 (10) | 0.834674 (12) | 0.074536 (10) | 0.03063 (6)                      |           |
| Au2 | 0.549659 (10) | 0.900739 (12) | 0.218117 (10) | 0.02907 (6)                      |           |
| P1  | 0.73666 (7)   | 0.70192 (8)   | 0.05633 (7)   | 0.0304 (3)                       |           |
| P2  | 0.53747 (7)   | 0.76109 (8)   | 0.23538 (7)   | 0.0285 (3)                       |           |
| P3  | 0.81787 (6)   | 0.96902 (8)   | 0.09657 (7)   | 0.0271 (3)                       |           |
| P4  | 0.56077 (6)   | 1.03989 (8)   | 0.19661 (7)   | 0.0266 (3)                       |           |
| C1  | 0.7040 (3)    | 0.6692 (3)    | 0.1377 (3)    | 0.0324 (12)                      |           |
| H1  | 0.6837        | 0.6129        | 0.1313        | 0.039*                           |           |
| H2  | 0.7443        | 0.6670        | 0.1762        | 0.039*                           |           |
| C2  | 0.6472 (2)    | 0.7282 (3)    | 0.1583 (3)    | 0.0306 (11)                      |           |
| H3  | 0.6057        | 0.7270        | 0.1210        | 0.037*                           |           |
| H4  | 0.6666        | 0.7850        | 0.1605        | 0.037*                           |           |
| C3  | 0.6218 (2)    | 0.7075 (3)    | 0.2294 (2)    | 0.0296 (11)                      |           |
| H5  | 0.6580        | 0.7251        | 0.2686        | 0.036*                           |           |
| H6  | 0.6149        | 0.6471        | 0.2333        | 0.036*                           |           |
| C4  | 0.7729 (2)    | 1.0247 (3)    | 0.1619 (3)    | 0.0289 (11)                      |           |
| H7  | 0.7878        | 1.0008        | 0.2092        | 0.035*                           |           |
| H8  | 0.7881        | 1.0832        | 0.1630        | 0.035*                           |           |
| C5  | 0.6909 (2)    | 1.0206 (3)    | 0.1457 (3)    | 0.0348 (12)                      |           |
| H9  | 0.6760        | 1.0424        | 0.0976        | 0.042*                           |           |
| H10 | 0.6754        | 0.9622        | 0.1466        | 0.042*                           |           |
| C6  | 0.6541 (2)    | 1.0700 (3)    | 0.1987 (3)    | 0.0304 (11)                      |           |
| H11 | 0.6568        | 1.1296        | 0.1877        | 0.036*                           |           |
| H12 | 0.6794        | 1.0610        | 0.2468        | 0.036*                           |           |
| C7  | 0.802 (2)     | 0.623 (3)     | 0.0354 (17)   | 0.055 (4)                        | 0.50      |

|     |             |             |              |             |      |
|-----|-------------|-------------|--------------|-------------|------|
| C8  | 0.7861 (11) | 0.5432 (14) | 0.0384 (9)   | 0.064 (4)   | 0.50 |
| H13 | 0.7424      | 0.5264      | 0.0537       | 0.077*      | 0.50 |
| C9  | 0.8353 (12) | 0.4825 (17) | 0.0181 (13)  | 0.070 (4)   | 0.50 |
| H14 | 0.8255      | 0.4253      | 0.0218       | 0.085*      | 0.50 |
| C10 | 0.8946 (13) | 0.5076 (15) | -0.0057 (11) | 0.075 (4)   | 0.50 |
| H15 | 0.9253      | 0.4681      | -0.0228      | 0.090*      | 0.50 |
| C11 | 0.9112 (9)  | 0.5852 (10) | -0.0060 (10) | 0.078 (3)   | 0.50 |
| H16 | 0.9559      | 0.6015      | -0.0191      | 0.094*      | 0.50 |
| C12 | 0.8637 (8)  | 0.6454 (10) | 0.0129 (9)   | 0.067 (3)   | 0.50 |
| H17 | 0.8751      | 0.7023      | 0.0097       | 0.081*      | 0.50 |
| C13 | 0.7966 (16) | 0.622 (3)   | 0.0378 (12)  | 0.030 (3)   | 0.50 |
| C14 | 0.7708 (9)  | 0.5416 (12) | 0.0162 (8)   | 0.042 (3)   | 0.50 |
| H18 | 0.7210      | 0.5317      | 0.0094       | 0.051*      | 0.50 |
| C15 | 0.8156 (8)  | 0.4794 (15) | 0.0055 (11)  | 0.049 (3)   | 0.50 |
| H19 | 0.7983      | 0.4265      | -0.0099      | 0.059*      | 0.50 |
| C16 | 0.8907 (12) | 0.4968 (14) | 0.0186 (9)   | 0.056 (3)   | 0.50 |
| H20 | 0.9236      | 0.4548      | 0.0101       | 0.067*      | 0.50 |
| C17 | 0.9153 (7)  | 0.5694 (9)  | 0.0421 (8)   | 0.056 (3)   | 0.50 |
| H21 | 0.9652      | 0.5785      | 0.0516       | 0.068*      | 0.50 |
| C18 | 0.8683 (7)  | 0.6319 (9)  | 0.0526 (7)   | 0.045 (3)   | 0.50 |
| H22 | 0.8866      | 0.6833      | 0.0708       | 0.054*      | 0.50 |
| C19 | 0.6622 (3)  | 0.6947 (3)  | -0.0140 (3)  | 0.0304 (11) |      |
| C20 | 0.6632 (3)  | 0.7447 (3)  | -0.0737 (3)  | 0.0430 (14) |      |
| H23 | 0.7014      | 0.7821      | -0.0764      | 0.052*      |      |
| C21 | 0.6076 (3)  | 0.7395 (4)  | -0.1294 (3)  | 0.0500 (16) |      |
| H24 | 0.6087      | 0.7727      | -0.1703      | 0.060*      |      |
| C22 | 0.5512 (3)  | 0.6863 (4)  | -0.1253 (3)  | 0.0525 (17) |      |
| H25 | 0.5130      | 0.6839      | -0.1627      | 0.063*      |      |
| C23 | 0.5506 (3)  | 0.6369 (3)  | -0.0668 (3)  | 0.0469 (15) |      |
| H26 | 0.5123      | 0.5996      | -0.0647      | 0.056*      |      |
| C24 | 0.6050 (3)  | 0.6407 (3)  | -0.0109 (3)  | 0.0404 (13) |      |
| H27 | 0.6034      | 0.6067      | 0.0294       | 0.048*      |      |
| C25 | 0.5129 (3)  | 0.7377 (3)  | 0.3220 (3)   | 0.0354 (12) |      |
| C26 | 0.4619 (3)  | 0.6787 (3)  | 0.3322 (3)   | 0.0456 (15) |      |
| H28 | 0.4399      | 0.6472      | 0.2934       | 0.055*      |      |
| C27 | 0.4431 (4)  | 0.6658 (4)  | 0.3999 (4)   | 0.0574 (18) |      |
| H29 | 0.4071      | 0.6269      | 0.4069       | 0.069*      |      |
| C28 | 0.4769 (4)  | 0.7097 (4)  | 0.4565 (4)   | 0.072 (2)   |      |
| H30 | 0.4649      | 0.7002      | 0.5026       | 0.086*      |      |
| C29 | 0.5281 (5)  | 0.7671 (4)  | 0.4462 (3)   | 0.078 (2)   |      |
| H31 | 0.5512      | 0.7973      | 0.4852       | 0.093*      |      |
| C30 | 0.5462 (4)  | 0.7811 (4)  | 0.3794 (3)   | 0.0577 (18) |      |
| H32 | 0.5816      | 0.8209      | 0.3728       | 0.069*      |      |
| C31 | 0.4727 (2)  | 0.7128 (3)  | 0.1696 (3)   | 0.0279 (11) |      |
| C32 | 0.4813 (3)  | 0.6337 (3)  | 0.1438 (3)   | 0.0432 (14) |      |
| H33 | 0.5202      | 0.6007      | 0.1638       | 0.052*      |      |
| C33 | 0.4338 (3)  | 0.6028 (4)  | 0.0894 (3)   | 0.0498 (15) |      |
| H34 | 0.4401      | 0.5483      | 0.0728       | 0.060*      |      |

|     |            |            |             |             |
|-----|------------|------------|-------------|-------------|
| C34 | 0.3779 (3) | 0.6490 (4) | 0.0588 (3)  | 0.0518 (16) |
| H35 | 0.3464     | 0.6274     | 0.0204      | 0.062*      |
| C35 | 0.3675 (3) | 0.7272 (4) | 0.0841 (3)  | 0.0485 (15) |
| H36 | 0.3277     | 0.7591     | 0.0644      | 0.058*      |
| C36 | 0.4150 (3) | 0.7590 (3) | 0.1381 (3)  | 0.0366 (12) |
| H37 | 0.4083     | 0.8135     | 0.1542      | 0.044*      |
| C37 | 0.8062 (2) | 1.0336 (3) | 0.0176 (3)  | 0.0303 (11) |
| C38 | 0.7747 (3) | 1.0013 (4) | -0.0457 (3) | 0.0444 (14) |
| H38 | 0.7611     | 0.9448     | -0.0481     | 0.053*      |
| C39 | 0.7627 (3) | 1.0501 (5) | -0.1058 (3) | 0.0565 (18) |
| H39 | 0.7405     | 1.0277     | -0.1491     | 0.068*      |
| C40 | 0.7836 (3) | 1.1317 (5) | -0.1014 (4) | 0.0618 (19) |
| H40 | 0.7763     | 1.1655     | -0.1423     | 0.074*      |
| C41 | 0.8145 (3) | 1.1645 (4) | -0.0391 (4) | 0.0562 (18) |
| H41 | 0.8276     | 1.2211     | -0.0367     | 0.067*      |
| C42 | 0.8268 (3) | 1.1151 (3) | 0.0208 (3)  | 0.0422 (14) |
| H42 | 0.8495     | 1.1377     | 0.0639      | 0.051*      |
| C43 | 0.9125 (2) | 0.9708 (3) | 0.1293 (3)  | 0.0269 (11) |
| C44 | 0.9617 (3) | 0.9585 (3) | 0.0813 (3)  | 0.0345 (12) |
| H43 | 0.9457     | 0.9566     | 0.0320      | 0.041*      |
| C45 | 1.0339 (3) | 0.9489 (3) | 0.1052 (3)  | 0.0396 (13) |
| H44 | 1.0666     | 0.9401     | 0.0721      | 0.048*      |
| C46 | 1.0583 (3) | 0.9520 (3) | 0.1762 (3)  | 0.0377 (13) |
| H45 | 1.1076     | 0.9455     | 0.1920      | 0.045*      |
| C47 | 1.0108 (3) | 0.9648 (3) | 0.2241 (3)  | 0.0363 (12) |
| H46 | 1.0276     | 0.9679     | 0.2731      | 0.044*      |
| C48 | 0.9377 (3) | 0.9733 (3) | 0.2010 (3)  | 0.0318 (11) |
| H47 | 0.9052     | 0.9809     | 0.2345      | 0.038*      |
| C49 | 0.5141 (2) | 1.0717 (3) | 0.1118 (2)  | 0.0265 (10) |
| C50 | 0.5378 (3) | 1.1376 (3) | 0.0733 (3)  | 0.0341 (12) |
| H48 | 0.5804     | 1.1664     | 0.0900      | 0.041*      |
| C51 | 0.4972 (3) | 1.1601 (3) | 0.0095 (3)  | 0.0434 (14) |
| H49 | 0.5123     | 1.2050     | -0.0170     | 0.052*      |
| C52 | 0.4360 (3) | 1.1183 (4) | -0.0153 (3) | 0.0420 (14) |
| H50 | 0.4093     | 1.1340     | -0.0588     | 0.050*      |
| C53 | 0.4130 (3) | 1.0534 (3) | 0.0231 (3)  | 0.0409 (14) |
| H51 | 0.3702     | 1.0251     | 0.0064      | 0.049*      |
| C54 | 0.4522 (3) | 1.0298 (3) | 0.0853 (3)  | 0.0343 (12) |
| H52 | 0.4368     | 0.9842     | 0.1108      | 0.041*      |
| C55 | 0.5201 (3) | 1.1036 (3) | 0.2596 (3)  | 0.0320 (12) |
| C56 | 0.5434 (3) | 1.1835 (3) | 0.2746 (3)  | 0.0463 (15) |
| H53 | 0.5833     | 1.2046     | 0.2549      | 0.056*      |
| C57 | 0.5084 (4) | 1.2322 (4) | 0.3184 (3)  | 0.0583 (18) |
| H54 | 0.5246     | 1.2869     | 0.3289      | 0.070*      |
| C58 | 0.4510 (4) | 1.2029 (5) | 0.3467 (4)  | 0.067 (2)   |
| H55 | 0.4272     | 1.2376     | 0.3763      | 0.081*      |
| C59 | 0.4271 (4) | 1.1241 (5) | 0.3329 (4)  | 0.069 (2)   |
| H56 | 0.3869     | 1.1040     | 0.3528      | 0.083*      |

|     |             |              |             |              |
|-----|-------------|--------------|-------------|--------------|
| C60 | 0.4624 (3)  | 1.0732 (4)   | 0.2892 (3)  | 0.0530 (16)  |
| H57 | 0.4468      | 1.0181       | 0.2800      | 0.064*       |
| Ni1 | 0.79494 (3) | 0.85982 (4)  | 0.34081 (3) | 0.03311 (16) |
| Cl1 | 0.81798 (9) | 0.81747 (9)  | 0.23347 (7) | 0.0496 (4)   |
| Cl2 | 0.68553 (8) | 0.91672 (11) | 0.32021 (9) | 0.0624 (5)   |
| Cl3 | 0.87604 (7) | 0.96027 (8)  | 0.37596 (7) | 0.0377 (3)   |
| Cl4 | 0.77899 (8) | 0.74535 (9)  | 0.40236 (8) | 0.0508 (4)   |
| O1  | 0.2306 (3)  | 0.4928 (4)   | 0.2151 (3)  | 0.0986 (18)  |
| C61 | 0.2757 (5)  | 0.5546 (8)   | 0.2001 (6)  | 0.125 (4)    |
| H58 | 0.2643      | 0.5702       | 0.1498      | 0.150*       |
| H59 | 0.3252      | 0.5337       | 0.2072      | 0.150*       |
| C62 | 0.2710 (6)  | 0.6288 (8)   | 0.2443 (8)  | 0.171 (6)    |
| H60 | 0.3041      | 0.6709       | 0.2312      | 0.257*       |
| H61 | 0.2835      | 0.6142       | 0.2942      | 0.257*       |
| H62 | 0.2223      | 0.6505       | 0.2369      | 0.257*       |
| C63 | 0.2334 (7)  | 0.4189 (7)   | 0.1756 (5)  | 0.125 (4)    |
| H63 | 0.2798      | 0.3912       | 0.1889      | 0.151*       |
| H64 | 0.2286      | 0.4319       | 0.1246      | 0.151*       |
| C64 | 0.1743 (8)  | 0.3632 (7)   | 0.1905 (7)  | 0.174 (6)    |
| H65 | 0.1764      | 0.3118       | 0.1637      | 0.260*       |
| H66 | 0.1285      | 0.3905       | 0.1764      | 0.260*       |
| H67 | 0.1793      | 0.3507       | 0.2411      | 0.260*       |

*Atomic displacement parameters ( $\text{\AA}^2$ )*

|     | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$     | $U^{13}$    | $U^{23}$     |
|-----|--------------|--------------|--------------|--------------|-------------|--------------|
| Au1 | 0.02931 (11) | 0.03070 (12) | 0.03187 (11) | -0.00375 (8) | 0.00394 (8) | -0.00447 (8) |
| Au2 | 0.03258 (11) | 0.02486 (11) | 0.02932 (11) | 0.00187 (8)  | 0.00220 (8) | 0.00207 (8)  |
| P1  | 0.0302 (7)   | 0.0293 (7)   | 0.0310 (7)   | -0.0001 (6)  | 0.0014 (6)  | -0.0036 (6)  |
| P2  | 0.0322 (7)   | 0.0262 (7)   | 0.0271 (7)   | 0.0005 (6)   | 0.0038 (5)  | 0.0034 (5)   |
| P3  | 0.0232 (7)   | 0.0300 (7)   | 0.0283 (7)   | -0.0020 (5)  | 0.0043 (5)  | -0.0013 (5)  |
| P4  | 0.0250 (7)   | 0.0248 (7)   | 0.0297 (7)   | 0.0018 (5)   | 0.0029 (5)  | -0.0008 (5)  |
| C1  | 0.038 (3)    | 0.023 (3)    | 0.034 (3)    | 0.003 (2)    | -0.004 (2)  | 0.002 (2)    |
| C2  | 0.026 (3)    | 0.029 (3)    | 0.036 (3)    | 0.000 (2)    | 0.003 (2)   | 0.002 (2)    |
| C3  | 0.031 (3)    | 0.026 (3)    | 0.031 (3)    | 0.004 (2)    | 0.000 (2)   | 0.004 (2)    |
| C4  | 0.029 (3)    | 0.028 (3)    | 0.029 (3)    | -0.005 (2)   | 0.004 (2)   | -0.003 (2)   |
| C5  | 0.027 (3)    | 0.038 (3)    | 0.039 (3)    | -0.001 (2)   | 0.002 (2)   | -0.009 (2)   |
| C6  | 0.032 (3)    | 0.027 (3)    | 0.031 (3)    | 0.007 (2)    | 0.000 (2)   | -0.007 (2)   |
| C7  | 0.046 (8)    | 0.048 (7)    | 0.077 (8)    | 0.005 (6)    | 0.026 (6)   | -0.011 (7)   |
| C8  | 0.057 (8)    | 0.055 (7)    | 0.084 (8)    | 0.013 (6)    | 0.019 (6)   | -0.008 (7)   |
| C9  | 0.063 (8)    | 0.054 (7)    | 0.098 (9)    | 0.015 (7)    | 0.020 (7)   | -0.007 (7)   |
| C10 | 0.065 (7)    | 0.061 (7)    | 0.104 (9)    | 0.019 (6)    | 0.026 (7)   | -0.011 (7)   |
| C11 | 0.063 (7)    | 0.064 (7)    | 0.111 (8)    | 0.004 (6)    | 0.023 (7)   | -0.009 (7)   |
| C12 | 0.051 (7)    | 0.055 (7)    | 0.100 (8)    | -0.002 (5)   | 0.026 (6)   | -0.012 (6)   |
| C13 | 0.023 (6)    | 0.036 (6)    | 0.030 (5)    | 0.003 (5)    | -0.005 (5)  | -0.006 (5)   |
| C14 | 0.030 (6)    | 0.042 (6)    | 0.053 (6)    | 0.001 (5)    | -0.001 (5)  | -0.010 (5)   |
| C15 | 0.035 (6)    | 0.044 (6)    | 0.067 (7)    | 0.006 (5)    | 0.005 (5)   | -0.003 (5)   |
| C16 | 0.040 (6)    | 0.057 (7)    | 0.072 (7)    | 0.014 (5)    | 0.012 (6)   | 0.005 (6)    |

|     |             |             |             |             |             |             |
|-----|-------------|-------------|-------------|-------------|-------------|-------------|
| C17 | 0.034 (5)   | 0.067 (6)   | 0.068 (6)   | 0.009 (5)   | 0.006 (5)   | -0.002 (5)  |
| C18 | 0.030 (5)   | 0.052 (6)   | 0.052 (5)   | 0.010 (4)   | -0.003 (5)  | -0.008 (5)  |
| C19 | 0.030 (3)   | 0.030 (3)   | 0.029 (3)   | 0.007 (2)   | -0.003 (2)  | -0.005 (2)  |
| C20 | 0.047 (3)   | 0.040 (3)   | 0.043 (3)   | 0.002 (3)   | 0.008 (3)   | -0.002 (3)  |
| C21 | 0.064 (4)   | 0.056 (4)   | 0.028 (3)   | 0.014 (3)   | -0.003 (3)  | 0.001 (3)   |
| C22 | 0.058 (4)   | 0.050 (4)   | 0.043 (4)   | 0.017 (3)   | -0.021 (3)  | -0.015 (3)  |
| C23 | 0.039 (3)   | 0.034 (3)   | 0.063 (4)   | -0.003 (3)  | -0.010 (3)  | -0.013 (3)  |
| C24 | 0.045 (3)   | 0.026 (3)   | 0.047 (3)   | -0.005 (2)  | -0.007 (3)  | -0.002 (2)  |
| C25 | 0.048 (3)   | 0.029 (3)   | 0.031 (3)   | 0.007 (2)   | 0.010 (2)   | 0.007 (2)   |
| C26 | 0.054 (4)   | 0.042 (4)   | 0.043 (3)   | -0.001 (3)  | 0.015 (3)   | 0.004 (3)   |
| C27 | 0.078 (5)   | 0.043 (4)   | 0.059 (4)   | -0.001 (3)  | 0.036 (4)   | 0.010 (3)   |
| C28 | 0.129 (7)   | 0.053 (4)   | 0.040 (4)   | 0.007 (5)   | 0.038 (4)   | 0.005 (3)   |
| C29 | 0.138 (7)   | 0.066 (5)   | 0.029 (4)   | -0.020 (5)  | 0.009 (4)   | -0.004 (3)  |
| C30 | 0.087 (5)   | 0.051 (4)   | 0.035 (3)   | -0.019 (4)  | 0.006 (3)   | 0.003 (3)   |
| C31 | 0.026 (3)   | 0.027 (3)   | 0.032 (3)   | -0.003 (2)  | 0.009 (2)   | 0.002 (2)   |
| C32 | 0.046 (3)   | 0.032 (3)   | 0.049 (4)   | -0.002 (3)  | -0.005 (3)  | 0.002 (3)   |
| C33 | 0.052 (4)   | 0.035 (3)   | 0.061 (4)   | -0.005 (3)  | 0.003 (3)   | -0.010 (3)  |
| C34 | 0.048 (4)   | 0.061 (4)   | 0.043 (4)   | -0.009 (3)  | -0.004 (3)  | -0.006 (3)  |
| C35 | 0.034 (3)   | 0.062 (4)   | 0.048 (4)   | 0.009 (3)   | -0.003 (3)  | -0.002 (3)  |
| C36 | 0.035 (3)   | 0.038 (3)   | 0.037 (3)   | 0.004 (2)   | 0.002 (2)   | -0.004 (2)  |
| C37 | 0.023 (3)   | 0.037 (3)   | 0.033 (3)   | 0.006 (2)   | 0.011 (2)   | 0.005 (2)   |
| C38 | 0.034 (3)   | 0.064 (4)   | 0.034 (3)   | 0.003 (3)   | 0.002 (2)   | 0.009 (3)   |
| C39 | 0.043 (4)   | 0.092 (6)   | 0.035 (3)   | 0.016 (4)   | 0.006 (3)   | 0.010 (3)   |
| C40 | 0.058 (4)   | 0.077 (5)   | 0.053 (4)   | 0.034 (4)   | 0.018 (3)   | 0.030 (4)   |
| C41 | 0.063 (4)   | 0.049 (4)   | 0.062 (4)   | 0.019 (3)   | 0.028 (4)   | 0.020 (3)   |
| C42 | 0.043 (3)   | 0.042 (4)   | 0.045 (3)   | 0.013 (3)   | 0.019 (3)   | 0.004 (3)   |
| C43 | 0.028 (3)   | 0.019 (3)   | 0.033 (3)   | 0.001 (2)   | 0.005 (2)   | -0.002 (2)  |
| C44 | 0.035 (3)   | 0.040 (3)   | 0.029 (3)   | 0.004 (2)   | 0.005 (2)   | 0.001 (2)   |
| C45 | 0.030 (3)   | 0.044 (3)   | 0.047 (3)   | 0.005 (2)   | 0.016 (3)   | 0.008 (3)   |
| C46 | 0.023 (3)   | 0.038 (3)   | 0.051 (4)   | 0.000 (2)   | 0.003 (2)   | 0.004 (3)   |
| C47 | 0.035 (3)   | 0.039 (3)   | 0.034 (3)   | -0.004 (2)  | 0.002 (2)   | -0.001 (2)  |
| C48 | 0.030 (3)   | 0.030 (3)   | 0.037 (3)   | 0.000 (2)   | 0.010 (2)   | 0.000 (2)   |
| C49 | 0.025 (3)   | 0.027 (3)   | 0.029 (3)   | 0.006 (2)   | 0.008 (2)   | -0.001 (2)  |
| C50 | 0.035 (3)   | 0.027 (3)   | 0.042 (3)   | 0.003 (2)   | 0.011 (2)   | 0.002 (2)   |
| C51 | 0.056 (4)   | 0.039 (3)   | 0.036 (3)   | 0.014 (3)   | 0.012 (3)   | 0.012 (3)   |
| C52 | 0.041 (3)   | 0.052 (4)   | 0.032 (3)   | 0.021 (3)   | 0.001 (3)   | -0.002 (3)  |
| C53 | 0.039 (3)   | 0.046 (4)   | 0.036 (3)   | 0.001 (3)   | -0.003 (3)  | -0.006 (3)  |
| C54 | 0.033 (3)   | 0.031 (3)   | 0.039 (3)   | 0.002 (2)   | 0.003 (2)   | 0.003 (2)   |
| C55 | 0.034 (3)   | 0.035 (3)   | 0.028 (3)   | 0.008 (2)   | 0.005 (2)   | -0.002 (2)  |
| C56 | 0.062 (4)   | 0.037 (4)   | 0.041 (3)   | 0.009 (3)   | 0.013 (3)   | 0.000 (3)   |
| C57 | 0.087 (5)   | 0.043 (4)   | 0.048 (4)   | 0.014 (4)   | 0.017 (4)   | -0.008 (3)  |
| C58 | 0.089 (6)   | 0.067 (5)   | 0.051 (4)   | 0.036 (4)   | 0.027 (4)   | -0.002 (4)  |
| C59 | 0.066 (5)   | 0.084 (6)   | 0.065 (5)   | 0.013 (4)   | 0.037 (4)   | 0.002 (4)   |
| C60 | 0.051 (4)   | 0.051 (4)   | 0.063 (4)   | -0.003 (3)  | 0.031 (3)   | -0.005 (3)  |
| Ni1 | 0.0343 (4)  | 0.0337 (4)  | 0.0296 (4)  | -0.0045 (3) | -0.0027 (3) | 0.0019 (3)  |
| Cl1 | 0.0703 (10) | 0.0470 (9)  | 0.0309 (7)  | -0.0074 (7) | 0.0042 (7)  | -0.0045 (6) |
| Cl2 | 0.0373 (8)  | 0.0790 (12) | 0.0660 (11) | 0.0071 (8)  | -0.0121 (7) | 0.0174 (9)  |
| Cl3 | 0.0343 (7)  | 0.0340 (7)  | 0.0437 (8)  | -0.0041 (6) | 0.0011 (6)  | -0.0060 (6) |

|     |             |            |            |             |             |            |
|-----|-------------|------------|------------|-------------|-------------|------------|
| Cl4 | 0.0627 (10) | 0.0424 (9) | 0.0474 (9) | -0.0060 (7) | 0.0077 (7)  | 0.0110 (7) |
| O1  | 0.106 (5)   | 0.114 (5)  | 0.080 (4)  | -0.022 (4)  | 0.031 (4)   | -0.001 (4) |
| C61 | 0.063 (6)   | 0.172 (11) | 0.138 (10) | -0.017 (7)  | 0.002 (6)   | 0.068 (9)  |
| C62 | 0.128 (10)  | 0.147 (11) | 0.220 (16) | -0.082 (9)  | -0.050 (10) | 0.032 (11) |
| C63 | 0.191 (12)  | 0.114 (9)  | 0.075 (7)  | 0.044 (8)   | 0.029 (7)   | -0.010 (6) |
| C64 | 0.281 (18)  | 0.105 (9)  | 0.134 (11) | -0.074 (11) | 0.023 (11)  | -0.022 (8) |

*Geometric parameters ( $\text{\AA}$ ,  $\text{^{\circ}}$ )*

|        |             |         |           |
|--------|-------------|---------|-----------|
| Au1—P1 | 2.3109 (13) | C29—H31 | 0.9500    |
| Au1—P3 | 2.3129 (13) | C30—H32 | 0.9500    |
| Au2—P2 | 2.3013 (13) | C31—C32 | 1.389 (7) |
| Au2—P4 | 2.3050 (13) | C31—C36 | 1.395 (7) |
| P1—C13 | 1.78 (4)    | C32—C33 | 1.378 (7) |
| P1—C19 | 1.820 (5)   | C32—H33 | 0.9500    |
| P1—C1  | 1.822 (5)   | C33—C34 | 1.364 (8) |
| P1—C7  | 1.87 (5)    | C33—H34 | 0.9500    |
| P2—C25 | 1.815 (5)   | C34—C35 | 1.378 (8) |
| P2—C31 | 1.816 (5)   | C34—H35 | 0.9500    |
| P2—C3  | 1.833 (5)   | C35—C36 | 1.377 (7) |
| P3—C43 | 1.820 (5)   | C35—H36 | 0.9500    |
| P3—C37 | 1.824 (5)   | C36—H37 | 0.9500    |
| P3—C4  | 1.834 (5)   | C37—C42 | 1.377 (7) |
| P4—C49 | 1.816 (5)   | C37—C38 | 1.379 (7) |
| P4—C6  | 1.828 (5)   | C38—C39 | 1.388 (8) |
| P4—C55 | 1.829 (5)   | C38—H38 | 0.9500    |
| C1—C2  | 1.528 (7)   | C39—C40 | 1.378 (9) |
| C1—H1  | 0.9900      | C39—H39 | 0.9500    |
| C1—H2  | 0.9900      | C40—C41 | 1.363 (9) |
| C2—C3  | 1.535 (6)   | C40—H40 | 0.9500    |
| C2—H3  | 0.9900      | C41—C42 | 1.390 (8) |
| C2—H4  | 0.9900      | C41—H41 | 0.9500    |
| C3—H5  | 0.9900      | C42—H42 | 0.9500    |
| C3—H6  | 0.9900      | C43—C48 | 1.391 (7) |
| C4—C5  | 1.545 (6)   | C43—C44 | 1.402 (7) |
| C4—H7  | 0.9900      | C44—C45 | 1.393 (7) |
| C4—H8  | 0.9900      | C44—H43 | 0.9500    |
| C5—C6  | 1.526 (6)   | C45—C46 | 1.375 (7) |
| C5—H9  | 0.9900      | C45—H44 | 0.9500    |
| C5—H10 | 0.9900      | C46—C47 | 1.379 (7) |
| C6—H11 | 0.9900      | C46—H45 | 0.9500    |
| C6—H12 | 0.9900      | C47—C48 | 1.405 (7) |
| C7—C8  | 1.33 (5)    | C47—H46 | 0.9500    |
| C7—C12 | 1.34 (4)    | C48—H47 | 0.9500    |
| C8—C9  | 1.44 (3)    | C49—C54 | 1.392 (7) |
| C8—H13 | 0.9500      | C49—C50 | 1.403 (7) |
| C9—C10 | 1.33 (3)    | C50—C51 | 1.402 (7) |
| C9—H14 | 0.9500      | C50—H48 | 0.9500    |

|            |             |             |             |
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| C10—C11    | 1.30 (3)    | C51—C52     | 1.373 (8)   |
| C10—H15    | 0.9500      | C51—H49     | 0.9500      |
| C11—C12    | 1.40 (2)    | C52—C53     | 1.383 (8)   |
| C11—H16    | 0.9500      | C52—H50     | 0.9500      |
| C12—H17    | 0.9500      | C53—C54     | 1.372 (7)   |
| C13—C18    | 1.36 (3)    | C53—H51     | 0.9500      |
| C13—C14    | 1.44 (4)    | C54—H52     | 0.9500      |
| C14—C15    | 1.35 (2)    | C55—C60     | 1.383 (7)   |
| C14—H18    | 0.9500      | C55—C56     | 1.384 (7)   |
| C15—C16    | 1.44 (3)    | C56—C57     | 1.379 (8)   |
| C15—H19    | 0.9500      | C56—H53     | 0.9500      |
| C16—C17    | 1.32 (2)    | C57—C58     | 1.359 (9)   |
| C16—H20    | 0.9500      | C57—H54     | 0.9500      |
| C17—C18    | 1.380 (17)  | C58—C59     | 1.368 (10)  |
| C17—H21    | 0.9500      | C58—H55     | 0.9500      |
| C18—H22    | 0.9500      | C59—C60     | 1.401 (8)   |
| C19—C24    | 1.400 (7)   | C59—H56     | 0.9500      |
| C19—C20    | 1.401 (7)   | C60—H57     | 0.9500      |
| C20—C21    | 1.399 (7)   | Ni1—Cl4     | 2.2359 (15) |
| C20—H23    | 0.9500      | Ni1—Cl2     | 2.2548 (16) |
| C21—C22    | 1.382 (8)   | Ni1—Cl1     | 2.2558 (15) |
| C21—H24    | 0.9500      | Ni1—Cl3     | 2.2780 (14) |
| C22—C23    | 1.375 (8)   | O1—C61      | 1.370 (10)  |
| C22—H25    | 0.9500      | O1—C63      | 1.418 (10)  |
| C23—C24    | 1.386 (7)   | C61—C62     | 1.477 (15)  |
| C23—H26    | 0.9500      | C61—H58     | 0.9900      |
| C24—H27    | 0.9500      | C61—H59     | 0.9900      |
| C25—C30    | 1.384 (7)   | C62—H60     | 0.9800      |
| C25—C26    | 1.390 (7)   | C62—H61     | 0.9800      |
| C26—C27    | 1.399 (8)   | C62—H62     | 0.9800      |
| C26—H28    | 0.9500      | C63—C64     | 1.492 (14)  |
| C27—C28    | 1.380 (9)   | C63—H63     | 0.9900      |
| C27—H29    | 0.9500      | C63—H64     | 0.9900      |
| C28—C29    | 1.375 (9)   | C64—H65     | 0.9800      |
| C28—H30    | 0.9500      | C64—H66     | 0.9800      |
| C29—C30    | 1.379 (8)   | C64—H67     | 0.9800      |
| <br>       |             |             |             |
| P1—Au1—P3  | 176.68 (5)  | C29—C28—H30 | 120.0       |
| P2—Au2—P4  | 177.98 (5)  | C27—C28—H30 | 120.0       |
| C13—P1—C19 | 105.0 (8)   | C28—C29—C30 | 120.4 (6)   |
| C13—P1—C1  | 104.7 (10)  | C28—C29—H31 | 119.8       |
| C19—P1—C1  | 107.1 (2)   | C30—C29—H31 | 119.8       |
| C19—P1—C7  | 105.8 (11)  | C29—C30—C25 | 120.4 (6)   |
| C1—P1—C7   | 106.9 (12)  | C29—C30—H32 | 119.8       |
| C13—P1—Au1 | 118.7 (11)  | C25—C30—H32 | 119.8       |
| C19—P1—Au1 | 113.35 (17) | C32—C31—C36 | 117.5 (5)   |
| C1—P1—Au1  | 107.16 (16) | C32—C31—P2  | 122.9 (4)   |
| C7—P1—Au1  | 116.0 (14)  | C36—C31—P2  | 119.3 (4)   |

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| C25—P2—C31 | 108.1 (2)   | C33—C32—C31 | 120.5 (5) |
| C25—P2—C3  | 106.5 (2)   | C33—C32—H33 | 119.8     |
| C31—P2—C3  | 105.6 (2)   | C31—C32—H33 | 119.8     |
| C25—P2—Au2 | 112.19 (17) | C34—C33—C32 | 121.2 (6) |
| C31—P2—Au2 | 113.28 (16) | C34—C33—H34 | 119.4     |
| C3—P2—Au2  | 110.65 (16) | C32—C33—H34 | 119.4     |
| C43—P3—C37 | 106.5 (2)   | C33—C34—C35 | 119.5 (6) |
| C43—P3—C4  | 106.4 (2)   | C33—C34—H35 | 120.2     |
| C37—P3—C4  | 105.2 (2)   | C35—C34—H35 | 120.2     |
| C43—P3—Au1 | 110.24 (15) | C36—C35—C34 | 119.7 (5) |
| C37—P3—Au1 | 112.76 (18) | C36—C35—H36 | 120.1     |
| C4—P3—Au1  | 115.17 (16) | C34—C35—H36 | 120.1     |
| C49—P4—C6  | 107.6 (2)   | C35—C36—C31 | 121.5 (5) |
| C49—P4—C55 | 103.2 (2)   | C35—C36—H37 | 119.2     |
| C6—P4—C55  | 108.8 (2)   | C31—C36—H37 | 119.2     |
| C49—P4—Au2 | 112.99 (16) | C42—C37—C38 | 119.4 (5) |
| C6—P4—Au2  | 111.52 (16) | C42—C37—P3  | 120.6 (4) |
| C55—P4—Au2 | 112.28 (17) | C38—C37—P3  | 119.9 (4) |
| C2—C1—P1   | 111.4 (3)   | C37—C38—C39 | 121.0 (6) |
| C2—C1—H1   | 109.4       | C37—C38—H38 | 119.5     |
| P1—C1—H1   | 109.4       | C39—C38—H38 | 119.5     |
| C2—C1—H2   | 109.4       | C40—C39—C38 | 118.7 (6) |
| P1—C1—H2   | 109.4       | C40—C39—H39 | 120.6     |
| H1—C1—H2   | 108.0       | C38—C39—H39 | 120.6     |
| C1—C2—C3   | 113.7 (4)   | C41—C40—C39 | 120.9 (6) |
| C1—C2—H3   | 108.8       | C41—C40—H40 | 119.5     |
| C3—C2—H3   | 108.8       | C39—C40—H40 | 119.5     |
| C1—C2—H4   | 108.8       | C40—C41—C42 | 120.1 (6) |
| C3—C2—H4   | 108.8       | C40—C41—H41 | 120.0     |
| H3—C2—H4   | 107.7       | C42—C41—H41 | 120.0     |
| C2—C3—P2   | 109.0 (3)   | C37—C42—C41 | 119.9 (6) |
| C2—C3—H5   | 109.9       | C37—C42—H42 | 120.1     |
| P2—C3—H5   | 109.9       | C41—C42—H42 | 120.1     |
| C2—C3—H6   | 109.9       | C48—C43—C44 | 118.4 (4) |
| P2—C3—H6   | 109.9       | C48—C43—P3  | 122.4 (4) |
| H5—C3—H6   | 108.3       | C44—C43—P3  | 118.7 (4) |
| C5—C4—P3   | 112.7 (3)   | C45—C44—C43 | 120.5 (5) |
| C5—C4—H7   | 109.1       | C45—C44—H43 | 119.7     |
| P3—C4—H7   | 109.1       | C43—C44—H43 | 119.7     |
| C5—C4—H8   | 109.1       | C46—C45—C44 | 120.7 (5) |
| P3—C4—H8   | 109.1       | C46—C45—H44 | 119.7     |
| H7—C4—H8   | 107.8       | C44—C45—H44 | 119.7     |
| C6—C5—C4   | 112.2 (4)   | C45—C46—C47 | 119.7 (5) |
| C6—C5—H9   | 109.2       | C45—C46—H45 | 120.2     |
| C4—C5—H9   | 109.2       | C47—C46—H45 | 120.2     |
| C6—C5—H10  | 109.2       | C46—C47—C48 | 120.4 (5) |
| C4—C5—H10  | 109.2       | C46—C47—H46 | 119.8     |
| H9—C5—H10  | 107.9       | C48—C47—H46 | 119.8     |

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| C5—C6—P4    | 111.6 (3)  | C43—C48—C47 | 120.4 (5)  |
| C5—C6—H11   | 109.3      | C43—C48—H47 | 119.8      |
| P4—C6—H11   | 109.3      | C47—C48—H47 | 119.8      |
| C5—C6—H12   | 109.3      | C54—C49—C50 | 119.4 (5)  |
| P4—C6—H12   | 109.3      | C54—C49—P4  | 118.5 (4)  |
| H11—C6—H12  | 108.0      | C50—C49—P4  | 122.1 (4)  |
| C8—C7—C12   | 120 (4)    | C51—C50—C49 | 118.5 (5)  |
| C8—C7—P1    | 119 (3)    | C51—C50—H48 | 120.8      |
| C12—C7—P1   | 121 (3)    | C49—C50—H48 | 120.8      |
| C7—C8—C9    | 119 (2)    | C52—C51—C50 | 121.0 (5)  |
| C7—C8—H13   | 120.4      | C52—C51—H49 | 119.5      |
| C9—C8—H13   | 120.4      | C50—C51—H49 | 119.5      |
| C10—C9—C8   | 119 (2)    | C51—C52—C53 | 120.1 (5)  |
| C10—C9—H14  | 120.4      | C51—C52—H50 | 120.0      |
| C8—C9—H14   | 120.4      | C53—C52—H50 | 120.0      |
| C11—C10—C9  | 121 (2)    | C54—C53—C52 | 119.9 (5)  |
| C11—C10—H15 | 119.5      | C54—C53—H51 | 120.0      |
| C9—C10—H15  | 119.5      | C52—C53—H51 | 120.0      |
| C10—C11—C12 | 120.6 (17) | C53—C54—C49 | 121.0 (5)  |
| C10—C11—H16 | 119.7      | C53—C54—H52 | 119.5      |
| C12—C11—H16 | 119.7      | C49—C54—H52 | 119.5      |
| C7—C12—C11  | 120 (3)    | C60—C55—C56 | 119.7 (5)  |
| C7—C12—H17  | 120.0      | C60—C55—P4  | 119.3 (4)  |
| C11—C12—H17 | 120.0      | C56—C55—P4  | 120.9 (4)  |
| C18—C13—C14 | 117 (3)    | C57—C56—C55 | 119.7 (6)  |
| C18—C13—P1  | 121 (2)    | C57—C56—H53 | 120.2      |
| C14—C13—P1  | 121 (2)    | C55—C56—H53 | 120.2      |
| C15—C14—C13 | 121.8 (18) | C58—C57—C56 | 120.8 (6)  |
| C15—C14—H18 | 119.1      | C58—C57—H54 | 119.6      |
| C13—C14—H18 | 119.1      | C56—C57—H54 | 119.6      |
| C14—C15—C16 | 116.9 (19) | C57—C58—C59 | 120.7 (6)  |
| C14—C15—H19 | 121.5      | C57—C58—H55 | 119.7      |
| C16—C15—H19 | 121.5      | C59—C58—H55 | 119.7      |
| C17—C16—C15 | 122 (2)    | C58—C59—C60 | 119.4 (6)  |
| C17—C16—H20 | 119.0      | C58—C59—H56 | 120.3      |
| C15—C16—H20 | 119.0      | C60—C59—H56 | 120.3      |
| C16—C17—C18 | 119.7 (15) | C55—C60—C59 | 119.7 (6)  |
| C16—C17—H21 | 120.1      | C55—C60—H57 | 120.1      |
| C18—C17—H21 | 120.1      | C59—C60—H57 | 120.1      |
| C13—C18—C17 | 122 (2)    | C14—Ni1—Cl2 | 104.33 (6) |
| C13—C18—H22 | 118.9      | C14—Ni1—Cl1 | 106.27 (6) |
| C17—C18—H22 | 118.9      | Cl2—Ni1—Cl1 | 104.24 (7) |
| C24—C19—C20 | 119.1 (5)  | C14—Ni1—Cl3 | 124.65 (6) |
| C24—C19—P1  | 122.6 (4)  | Cl2—Ni1—Cl3 | 109.20 (6) |
| C20—C19—P1  | 118.3 (4)  | Cl1—Ni1—Cl3 | 106.45 (6) |
| C21—C20—C19 | 119.8 (5)  | C61—O1—C63  | 116.0 (8)  |
| C21—C20—H23 | 120.1      | O1—C61—C62  | 113.0 (9)  |
| C19—C20—H23 | 120.1      | O1—C61—H58  | 109.0      |

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| C22—C21—C20   | 120.4 (6)   | C62—C61—H58     | 109.0      |
| C22—C21—H24   | 119.8       | O1—C61—H59      | 109.0      |
| C20—C21—H24   | 119.8       | C62—C61—H59     | 109.0      |
| C23—C22—C21   | 119.7 (5)   | H58—C61—H59     | 107.8      |
| C23—C22—H25   | 120.2       | C61—C62—H60     | 109.5      |
| C21—C22—H25   | 120.2       | C61—C62—H61     | 109.5      |
| C22—C23—C24   | 121.2 (6)   | H60—C62—H61     | 109.5      |
| C22—C23—H26   | 119.4       | C61—C62—H62     | 109.5      |
| C24—C23—H26   | 119.4       | H60—C62—H62     | 109.5      |
| C23—C24—C19   | 119.8 (5)   | H61—C62—H62     | 109.5      |
| C23—C24—H27   | 120.1       | O1—C63—C64      | 109.2 (9)  |
| C19—C24—H27   | 120.1       | O1—C63—H63      | 109.8      |
| C30—C25—C26   | 119.5 (5)   | C64—C63—H63     | 109.8      |
| C30—C25—P2    | 118.2 (4)   | O1—C63—H64      | 109.8      |
| C26—C25—P2    | 122.4 (4)   | C64—C63—H64     | 109.8      |
| C25—C26—C27   | 119.7 (6)   | H63—C63—H64     | 108.3      |
| C25—C26—H28   | 120.1       | C63—C64—H65     | 109.5      |
| C27—C26—H28   | 120.1       | C63—C64—H66     | 109.5      |
| C28—C27—C26   | 119.9 (6)   | H65—C64—H66     | 109.5      |
| C28—C27—H29   | 120.1       | C63—C64—H67     | 109.5      |
| C26—C27—H29   | 120.1       | H65—C64—H67     | 109.5      |
| C29—C28—C27   | 120.1 (6)   | H66—C64—H67     | 109.5      |
| <br>          |             |                 |            |
| C13—P1—C1—C2  | -175.8 (10) | C26—C25—C30—C29 | -1.1 (9)   |
| C19—P1—C1—C2  | -64.6 (4)   | P2—C25—C30—C29  | 178.4 (5)  |
| C7—P1—C1—C2   | -177.7 (13) | C25—P2—C31—C32  | -91.2 (5)  |
| Au1—P1—C1—C2  | 57.3 (4)    | C3—P2—C31—C32   | 22.5 (5)   |
| P1—C1—C2—C3   | -175.6 (3)  | Au2—P2—C31—C32  | 143.8 (4)  |
| C1—C2—C3—P2   | -163.2 (3)  | C25—P2—C31—C36  | 95.1 (4)   |
| C25—P2—C3—C2  | -177.4 (3)  | C3—P2—C31—C36   | -151.1 (4) |
| C31—P2—C3—C2  | 67.7 (4)    | Au2—P2—C31—C36  | -29.9 (4)  |
| Au2—P2—C3—C2  | -55.2 (4)   | C36—C31—C32—C33 | -0.4 (8)   |
| C43—P3—C4—C5  | -173.8 (3)  | P2—C31—C32—C33  | -174.2 (4) |
| C37—P3—C4—C5  | 73.4 (4)    | C31—C32—C33—C34 | 0.9 (9)    |
| Au1—P3—C4—C5  | -51.4 (4)   | C32—C33—C34—C35 | -1.9 (10)  |
| P3—C4—C5—C6   | -177.7 (3)  | C33—C34—C35—C36 | 2.4 (9)    |
| C4—C5—C6—P4   | -162.3 (4)  | C34—C35—C36—C31 | -2.0 (9)   |
| C49—P4—C6—C5  | -67.3 (4)   | C32—C31—C36—C35 | 1.0 (8)    |
| C55—P4—C6—C5  | -178.5 (4)  | P2—C31—C36—C35  | 175.0 (4)  |
| Au2—P4—C6—C5  | 57.1 (4)    | C43—P3—C37—C42  | -58.1 (4)  |
| C19—P1—C7—C8  | -67 (2)     | C4—P3—C37—C42   | 54.6 (4)   |
| C1—P1—C7—C8   | 47 (2)      | Au1—P3—C37—C42  | -179.1 (3) |
| Au1—P1—C7—C8  | 166.4 (17)  | C43—P3—C37—C38  | 123.6 (4)  |
| C19—P1—C7—C12 | 108 (2)     | C4—P3—C37—C38   | -123.8 (4) |
| C1—P1—C7—C12  | -138 (2)    | Au1—P3—C37—C38  | 2.5 (4)    |
| Au1—P1—C7—C12 | -18 (3)     | C42—C37—C38—C39 | -0.9 (8)   |
| C12—C7—C8—C9  | 1 (3)       | P3—C37—C38—C39  | 177.4 (4)  |
| P1—C7—C8—C9   | 176.4 (18)  | C37—C38—C39—C40 | 0.7 (8)    |

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| C7—C8—C9—C10    | -3 (2)      | C38—C39—C40—C41 | -1.0 (9)   |
| C8—C9—C10—C11   | 5 (4)       | C39—C40—C41—C42 | 1.4 (9)    |
| C9—C10—C11—C12  | -6 (4)      | C38—C37—C42—C41 | 1.4 (8)    |
| C8—C7—C12—C11   | -1 (4)      | P3—C37—C42—C41  | -177.0 (4) |
| P1—C7—C12—C11   | -176.9 (16) | C40—C41—C42—C37 | -1.6 (9)   |
| C10—C11—C12—C7  | 4 (3)       | C37—P3—C43—C48  | 141.5 (4)  |
| C19—P1—C13—C18  | 146.0 (17)  | C4—P3—C43—C48   | 29.6 (5)   |
| C1—P1—C13—C18   | -101 (2)    | Au1—P3—C43—C48  | -95.9 (4)  |
| C7—P1—C13—C18   | 40 (31)     | C37—P3—C43—C44  | -46.8 (4)  |
| Au1—P1—C13—C18  | 18 (2)      | C4—P3—C43—C44   | -158.7 (4) |
| C19—P1—C13—C14  | -43.2 (19)  | Au1—P3—C43—C44  | 75.8 (4)   |
| C1—P1—C13—C14   | 69.5 (16)   | C48—C43—C44—C45 | 0.2 (7)    |
| Au1—P1—C13—C14  | -171.1 (13) | P3—C43—C44—C45  | -171.8 (4) |
| C18—C13—C14—C15 | -5 (2)      | C43—C44—C45—C46 | -0.6 (8)   |
| P1—C13—C14—C15  | -176.5 (17) | C44—C45—C46—C47 | 0.0 (8)    |
| C13—C14—C15—C16 | 2 (2)       | C45—C46—C47—C48 | 1.0 (8)    |
| C14—C15—C16—C17 | 2 (3)       | C44—C43—C48—C47 | 0.8 (7)    |
| C15—C16—C17—C18 | -2 (3)      | P3—C43—C48—C47  | 172.5 (4)  |
| C14—C13—C18—C17 | 5 (3)       | C46—C47—C48—C43 | -1.4 (7)   |
| P1—C13—C18—C17  | 176.5 (14)  | C6—P4—C49—C54   | 155.1 (4)  |
| C16—C17—C18—C13 | -2 (2)      | C55—P4—C49—C54  | -89.9 (4)  |
| C13—P1—C19—C24  | 85.3 (12)   | Au2—P4—C49—C54  | 31.6 (4)   |
| C1—P1—C19—C24   | -25.6 (5)   | C6—P4—C49—C50   | -26.1 (5)  |
| C7—P1—C19—C24   | 88.2 (15)   | C55—P4—C49—C50  | 88.8 (4)   |
| Au1—P1—C19—C24  | -143.6 (4)  | Au2—P4—C49—C50  | -149.6 (4) |
| C13—P1—C19—C20  | -94.0 (12)  | C54—C49—C50—C51 | 1.1 (7)    |
| C1—P1—C19—C20   | 155.1 (4)   | P4—C49—C50—C51  | -177.7 (4) |
| C7—P1—C19—C20   | -91.0 (15)  | C49—C50—C51—C52 | -0.6 (8)   |
| Au1—P1—C19—C20  | 37.1 (4)    | C50—C51—C52—C53 | 0.6 (8)    |
| C24—C19—C20—C21 | -0.6 (8)    | C51—C52—C53—C54 | -1.2 (8)   |
| P1—C19—C20—C21  | 178.7 (4)   | C52—C53—C54—C49 | 1.7 (8)    |
| C19—C20—C21—C22 | 1.1 (8)     | C50—C49—C54—C53 | -1.7 (7)   |
| C20—C21—C22—C23 | -1.5 (9)    | P4—C49—C54—C53  | 177.1 (4)  |
| C21—C22—C23—C24 | 1.4 (9)     | C49—P4—C55—C60  | 92.3 (5)   |
| C22—C23—C24—C19 | -0.9 (9)    | C6—P4—C55—C60   | -153.6 (5) |
| C20—C19—C24—C23 | 0.5 (8)     | Au2—P4—C55—C60  | -29.7 (5)  |
| P1—C19—C24—C23  | -178.7 (4)  | C49—P4—C55—C56  | -83.7 (5)  |
| C31—P2—C25—C30  | -167.5 (5)  | C6—P4—C55—C56   | 30.4 (5)   |
| C3—P2—C25—C30   | 79.4 (5)    | Au2—P4—C55—C56  | 154.3 (4)  |
| Au2—P2—C25—C30  | -41.8 (5)   | C60—C55—C56—C57 | -0.7 (9)   |
| C31—P2—C25—C26  | 12.0 (5)    | P4—C55—C56—C57  | 175.3 (5)  |
| C3—P2—C25—C26   | -101.1 (5)  | C55—C56—C57—C58 | -0.4 (9)   |
| Au2—P2—C25—C26  | 137.7 (4)   | C56—C57—C58—C59 | 0.7 (11)   |
| C30—C25—C26—C27 | 2.1 (8)     | C57—C58—C59—C60 | 0.0 (11)   |
| P2—C25—C26—C27  | -177.4 (4)  | C56—C55—C60—C59 | 1.4 (9)    |
| C25—C26—C27—C28 | -2.2 (9)    | P4—C55—C60—C59  | -174.6 (5) |
| C26—C27—C28—C29 | 1.3 (11)    | C58—C59—C60—C55 | -1.1 (10)  |
| C27—C28—C29—C30 | -0.2 (12)   | C63—O1—C61—C62  | -178.7 (9) |

## supporting information

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C28—C29—C30—C25

0.1 (11)

C61—O1—C63—C64

−171.8 (9)

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