

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

# Bis(triphenylphosphine- $\kappa P$ )(tropolonato- $\kappa^2 O, O'$ )silver(I) dichloromethane solvate

Gideon Steyl\* and Tania N. Hill

Department of Chemistry, University of the Free State, Bloemfontein 9300, South Africa

Correspondence e-mail: steylg.sci@ufs.ac.za

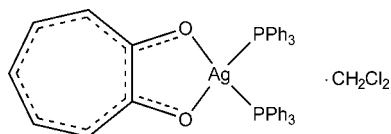
Received 6 January 2009; accepted 22 January 2009

 Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(C-C) = 0.003$  Å;  $R$  factor = 0.030;  $wR$  factor = 0.070; data-to-parameter ratio = 17.8.

The title compound,  $[Ag(C_7H_5O_2)(C_{18}H_{15}P)_2] \cdot CH_2Cl_2$ , crystallizes with a distorted tetrahedral geometry about the  $Ag^I$  atom, defined by two O atoms from one tropolonate ligand and two P atoms from two triphenylphosphine ligands. It is an example of a new type of tropolone derivative that has not been characterized *via* solid-state methods.

## Related literature

For general background, see: Crous *et al.* (2005); Dewar (1945). For structurally related oxalate derivatives, see: Dean *et al.* (2001). For related diketonate complexes, see: Hill & Steyl (2008); Steyl (2006).



## Experimental

### Crystal data

 $[Ag(C_7H_5O_2)(C_{18}H_{15}P)_2] \cdot CH_2Cl_2$ 
 $M_r = 838.45$ 

 Triclinic,  $P\bar{1}$ 
 $a = 12.0175$  (3) Å

 $b = 12.9925$  (4) Å

 $c = 13.8394$  (7) Å

 $\alpha = 100.487$  (2)°

 $\beta = 93.760$  (2)°

 $\gamma = 116.809$  (1)°

 $V = 1869.33$  (12) Å<sup>3</sup>
 $Z = 2$ 

 Mo  $K\alpha$  radiation

 $\mu = 0.81$  mm<sup>-1</sup>
 $T = 100$  (2) K

 $0.19 \times 0.11 \times 0.08$  mm

### Data collection

Bruker APEXII CCD

diffractometer

Absorption correction: multi-scan

(SADABS; Bruker, 2001)

 $T_{\min} = 0.862$ ,  $T_{\max} = 0.938$ 

33544 measured reflections

8178 independent reflections

 6931 reflections with  $I > 2\sigma(I)$ 
 $R_{\text{int}} = 0.046$ 

### Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.030$ 
 $wR(F^2) = 0.070$ 
 $S = 1.05$ 

8178 reflections

460 parameters

H-atom parameters constrained

 $\Delta\rho_{\max} = 0.51$  e Å<sup>-3</sup>
 $\Delta\rho_{\min} = -0.41$  e Å<sup>-3</sup>
**Table 1**

Selected geometric parameters (Å, °).

|          |             |          |              |
|----------|-------------|----------|--------------|
| Ag—O1    | 2.3612 (16) | Ag—P1    | 2.4070 (6)   |
| Ag—O2    | 2.3342 (16) | Ag—P2    | 2.4981 (5)   |
| O2—Ag—O1 | 68.67 (6)   | O2—Ag—P2 | 103.97 (4)   |
| O2—Ag—P1 | 121.43 (4)  | O1—Ag—P2 | 98.81 (4)    |
| O1—Ag—P1 | 128.35 (4)  | P1—Ag—P2 | 122.391 (19) |

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT-Plus* (Bruker, 2007); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Putz, 1999); software used to prepare material for publication: *SHELXL97*.

Financial assistance from the University of the Free State and Professor A. Roodt is gratefully acknowledged. Mr L. Kirsten is acknowledged for the data collection. Part of this research is based on work supported by the South African National Research Foundation (NRF) (grant No. GUN2068915). Opinions, findings, conclusions or recommendations expressed in this material are those of the authors and do not necessarily reflect the views of the NRF.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2179).

## References

- Brandenburg, K. & Putz, H. (1999). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
- Bruker (2001). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2007). *APEX2* and *SAINT-Plus*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Crous, R., Datt, M., Foster, D., Bennie, L., Steenkamp, C., Huyser, J., Kirsten, L., Steyl, G. & Roodt, A. (2005). *Dalton Trans.* pp. 1108–1115.
- Dean, P. A. W., Scudder, M., Craig, D. & Dance, I. (2001). *CrystEngComm*, **3**, 84–90.
- Dewar, M. J. S. (1945). *Nature (London)*, **155**, 141–145.
- Hill, T. N. & Steyl, G. (2008). *Acta Cryst.* **E64**, m1580–m1581.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Steyl, G. (2006). *Acta Cryst.* **E62**, m650–m652.

## supporting information

*Acta Cryst.* (2009). E65, m233 [doi:10.1107/S1600536809002785]

## Bis(triphenylphosphine- $\kappa P$ )(tropolonato- $\kappa^2 O, O'$ )silver(I) dichloromethane solvate

Gideon Steyl and Tania N. Hill

### S1. Comment

Tropolone type compounds have been of interest since its first discovery in the early 1940's (Dewar, 1945), with applications in pharmacology (Hill & Steyl, 2008) and catalysis (Crous *et al.*, 2005). Although this type of compounds have been extensively studied, to date no structural data exist, which contain both a tropolone moiety and a silver metal centre. In this regard, we present a bis(triphenylphosphine)silver(I) complex of tropolone.

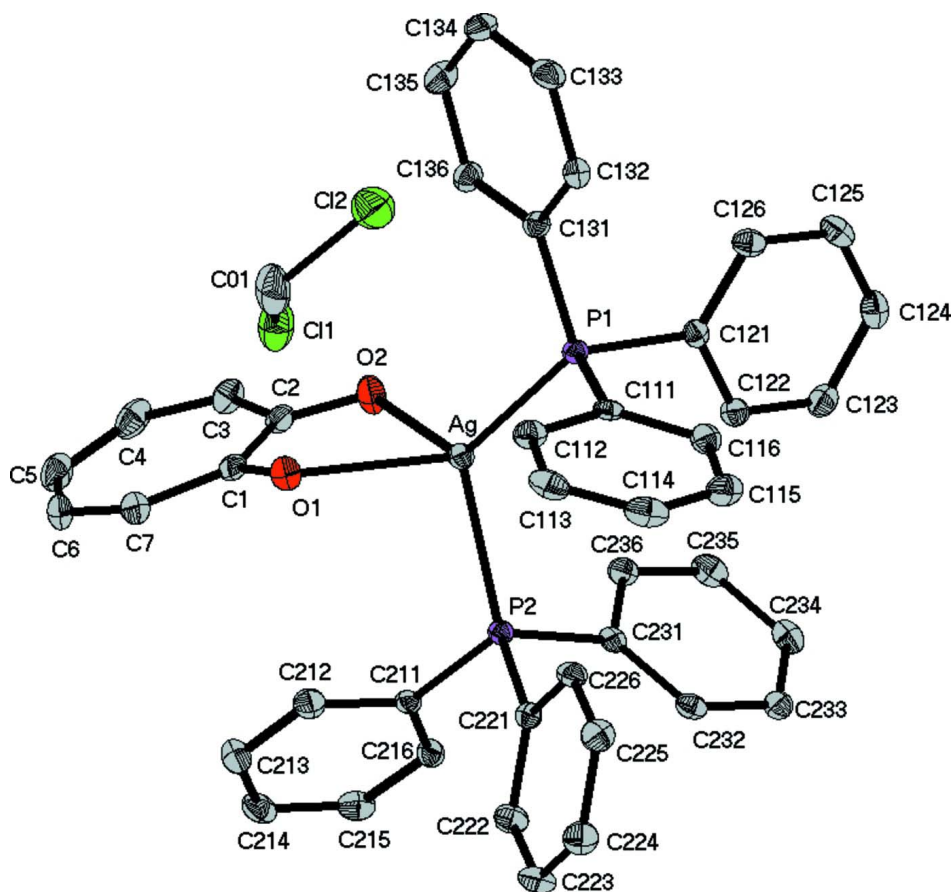
The Ag—O and Ag—P bond distances differ significantly from each other (Table 1). The distortion of these bonds are also reflected in the interplanar angle between the two planes defined by P—Ag—P and O—Ag—O in the order of 84.90 (4)°. A similar oxalate structure with two Ag centres has been reported (Dean *et al.*, 2001). In this complex the P—Ag—P and O—Ag—O bond angles are 126.35 and 71.28 °, respectively. These values compared to the title compound are significantly larger, indicating the structural effect of the tropolonate moiety on the complexes. In previous studies it has been shown that the tropolonate moiety can act as either a bidentate or a monodentate ligand in its coordination behaviour (Steyl, 2006). Effectively, the tropolonate moiety assumes an optimal coordination mode to pack as efficiently as possible. No classical hydrogen bonds are observed in the solid-state structure of the title compound, although weak interactions do exist between the dichloromethane solvate and the silver complex.

### S2. Experimental

The title complex was synthesized by the addition of sodium salt of tropolone (0.083 g, 0.57 mmol) to a dichloromethane solution (10 ml) of [Cu(PPh<sub>3</sub>)<sub>2</sub>]NO<sub>3</sub> (0.374 g, 0.287 mmol). On slow evaporation of the solvent, crystals suitable for X-ray crystallography were obtained (yield 85%).

### S3. Refinement

H atoms were positioned geometrically and refined using a riding model, with C—H = 0.95 (CH) and 0.99 (CH<sub>2</sub>) Å and with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .



**Figure 1**

Molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity.

**Bis(triphenylphosphine- $\kappa P$ )(tropolonato- $\kappa^2 O, O'$ )silver(I) dichloromethane solvate**

*Crystal data*

$[\text{Ag}(\text{C}_7\text{H}_5\text{O}_2)(\text{C}_{18}\text{H}_{15}\text{P})_2] \cdot \text{CH}_2\text{Cl}_2$

$M_r = 838.45$

Triclinic,  $P\bar{1}$

Hall symbol:  $-P\ 1$

$a = 12.0175\ (3)\ \text{\AA}$

$b = 12.9925\ (4)\ \text{\AA}$

$c = 13.8394\ (7)\ \text{\AA}$

$\alpha = 100.487\ (2)^\circ$

$\beta = 93.760\ (2)^\circ$

$\gamma = 116.809\ (1)^\circ$

$V = 1869.33\ (12)\ \text{\AA}^3$

$Z = 2$

$F(000) = 856$

$D_x = 1.490\ \text{Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 9106 reflections

$\theta = 2.3\text{--}28.3^\circ$

$\mu = 0.81\ \text{mm}^{-1}$

$T = 100\ \text{K}$

Cuboid, yellow

$0.19 \times 0.11 \times 0.08\ \text{mm}$

*Data collection*

Bruker APEXII CCD  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan  
(*SADABS*; Bruker, 2001)

$T_{\text{min}} = 0.862$ ,  $T_{\text{max}} = 0.938$

33544 measured reflections

8178 independent reflections

6931 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.046$   
 $\theta_{\text{max}} = 27.0^\circ$ ,  $\theta_{\text{min}} = 1.8^\circ$

$h = -13 \rightarrow 15$   
 $k = -16 \rightarrow 16$   
 $l = -17 \rightarrow 17$

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.030$   
 $wR(F^2) = 0.070$   
 $S = 1.05$   
 8178 reflections  
 460 parameters  
 0 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  
 $w = 1/[\sigma^2(F_o^2) + (0.0283P)^2 + 0.6452P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.002$   
 $\Delta\rho_{\text{max}} = 0.51 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.41 \text{ e } \text{\AA}^{-3}$

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

|      | <i>x</i>      | <i>y</i>      | <i>z</i>      | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|---------------|---------------|---------------|----------------------------------|
| Ag   | 0.743570 (16) | 0.720243 (14) | 0.744274 (12) | 0.01303 (5)                      |
| P2   | 0.86325 (5)   | 0.62927 (5)   | 0.65456 (4)   | 0.01241 (12)                     |
| P1   | 0.71316 (5)   | 0.87540 (5)   | 0.69503 (4)   | 0.01207 (12)                     |
| O1   | 0.79674 (15)  | 0.70564 (14)  | 0.90673 (11)  | 0.0184 (4)                       |
| C236 | 0.6628 (2)    | 0.49295 (19)  | 0.49875 (17)  | 0.0156 (5)                       |
| H236 | 0.6130        | 0.4770        | 0.5501        | 0.019*                           |
| O2   | 0.57859 (15)  | 0.56103 (15)  | 0.78664 (12)  | 0.0223 (4)                       |
| C115 | 1.0246 (2)    | 1.1352 (2)    | 0.61838 (19)  | 0.0231 (6)                       |
| H115 | 1.0505        | 1.1622        | 0.5603        | 0.028*                           |
| C211 | 0.8758 (2)    | 0.50995 (19)  | 0.69797 (16)  | 0.0132 (5)                       |
| C131 | 0.6271 (2)    | 0.93724 (19)  | 0.76747 (16)  | 0.0134 (5)                       |
| C116 | 0.9035 (2)    | 1.0442 (2)    | 0.61294 (17)  | 0.0174 (5)                       |
| H116 | 0.8465        | 1.0099        | 0.5514        | 0.021*                           |
| C111 | 0.8651 (2)    | 1.00316 (19)  | 0.69726 (16)  | 0.0131 (5)                       |
| C132 | 0.6443 (2)    | 1.0510 (2)    | 0.77170 (16)  | 0.0160 (5)                       |
| H132 | 0.7057        | 1.1010        | 0.7383        | 0.019*                           |
| C231 | 0.7939 (2)    | 0.56327 (18)  | 0.52340 (16)  | 0.0119 (4)                       |
| C233 | 0.8062 (2)    | 0.5380 (2)    | 0.34768 (17)  | 0.0164 (5)                       |
| H233 | 0.8555        | 0.5529        | 0.2960        | 0.020*                           |
| C232 | 0.8649 (2)    | 0.58525 (19)  | 0.44675 (16)  | 0.0145 (5)                       |
| H232 | 0.9542        | 0.6329        | 0.4624        | 0.017*                           |
| C122 | 0.6516 (2)    | 0.7563 (2)    | 0.49517 (16)  | 0.0165 (5)                       |
| H122 | 0.7103        | 0.7299        | 0.5131        | 0.020*                           |
| C2   | 0.6043 (2)    | 0.5279 (2)    | 0.86135 (16)  | 0.0163 (5)                       |
| C121 | 0.6314 (2)    | 0.83422 (19)  | 0.56701 (16)  | 0.0130 (4)                       |
| C216 | 0.8462 (2)    | 0.40129 (19)  | 0.63496 (17)  | 0.0149 (5)                       |
| H216 | 0.8168        | 0.3862        | 0.5659        | 0.018*                           |
| C221 | 1.0266 (2)    | 0.73307 (19)  | 0.65092 (15)  | 0.0124 (4)                       |
| C133 | 0.5721 (2)    | 1.0925 (2)    | 0.82465 (17)  | 0.0211 (5)                       |
| H133 | 0.5848        | 1.1708        | 0.8280        | 0.025*                           |
| C235 | 0.6052 (2)    | 0.4466 (2)    | 0.40016 (18)  | 0.0187 (5)                       |

---

|      |            |            |              |            |
|------|------------|------------|--------------|------------|
| H235 | 0.5160     | 0.3985     | 0.3842       | 0.022*     |
| C123 | 0.5874 (2) | 0.7175 (2) | 0.39882 (17) | 0.0188 (5) |
| H123 | 0.6026     | 0.6652     | 0.3507       | 0.023*     |
| C226 | 1.0571 (2) | 0.8503 (2) | 0.65170 (16) | 0.0155 (5) |
| H226 | 0.9939     | 0.8749     | 0.6565       | 0.019*     |
| C124 | 0.5006 (2) | 0.7547 (2) | 0.37165 (17) | 0.0200 (5) |
| H124 | 0.4555     | 0.7270     | 0.3054       | 0.024*     |
| C136 | 0.5374 (2) | 0.8647 (2) | 0.81740 (16) | 0.0170 (5) |
| H136 | 0.5262     | 0.7873     | 0.8164       | 0.020*     |
| C222 | 1.1212 (2) | 0.6993 (2) | 0.64488 (17) | 0.0182 (5) |
| H222 | 1.1019     | 0.6200     | 0.6452       | 0.022*     |
| C215 | 0.8596 (2) | 0.3150 (2) | 0.67277 (18) | 0.0191 (5) |
| H215 | 0.8393     | 0.2410     | 0.6294       | 0.023*     |
| C126 | 0.5451 (2) | 0.8718 (2) | 0.53879 (17) | 0.0169 (5) |
| H126 | 0.5305     | 0.9250     | 0.5864       | 0.020*     |
| C114 | 1.1078 (2) | 1.1868 (2) | 0.7076 (2)   | 0.0233 (6) |
| H114 | 1.1902     | 1.2499     | 0.7111       | 0.028*     |
| C224 | 1.2722 (2) | 0.8965 (2) | 0.63878 (18) | 0.0203 (5) |
| H224 | 1.3557     | 0.9522     | 0.6344       | 0.024*     |
| C112 | 0.9504 (2) | 1.0542 (2) | 0.78683 (17) | 0.0183 (5) |
| H112 | 0.9258     | 1.0258     | 0.8446       | 0.022*     |
| C113 | 1.0710 (2) | 1.1464 (2) | 0.79192 (19) | 0.0227 (5) |
| H113 | 1.1282     | 1.1818     | 0.8533       | 0.027*     |
| C223 | 1.2432 (2) | 0.7803 (2) | 0.63844 (18) | 0.0203 (5) |
| H223 | 1.3068     | 0.7561     | 0.6338       | 0.024*     |
| C1   | 0.7289 (2) | 0.6063 (2) | 0.92667 (16) | 0.0152 (5) |
| C134 | 0.4822 (2) | 1.0193 (2) | 0.87219 (17) | 0.0227 (6) |
| H134 | 0.4322     | 1.0470     | 0.9077       | 0.027*     |
| C125 | 0.4804 (2) | 0.8322 (2) | 0.44196 (18) | 0.0198 (5) |
| H125 | 0.4218     | 0.8585     | 0.4236       | 0.024*     |
| C234 | 0.6762 (2) | 0.4693 (2) | 0.32393 (17) | 0.0178 (5) |
| H234 | 0.6358     | 0.4381     | 0.2562       | 0.021*     |
| C3   | 0.5113 (2) | 0.4195 (2) | 0.87918 (17) | 0.0200 (5) |
| H3   | 0.4331     | 0.3847     | 0.8350       | 0.024*     |
| C7   | 0.7775 (2) | 0.5754 (2) | 1.00616 (17) | 0.0207 (5) |
| H7   | 0.8607     | 0.6334     | 1.0379       | 0.025*     |
| C213 | 0.9307 (2) | 0.4435 (2) | 0.83629 (18) | 0.0224 (5) |
| H213 | 0.9596     | 0.4579     | 0.9054       | 0.027*     |
| C225 | 1.1789 (2) | 0.9311 (2) | 0.64553 (17) | 0.0191 (5) |
| H225 | 1.1987     | 1.0107     | 0.6459       | 0.023*     |
| C135 | 0.4643 (2) | 0.9060 (2) | 0.86865 (18) | 0.0239 (6) |
| H135 | 0.4018     | 0.8560     | 0.9014       | 0.029*     |
| C214 | 0.9023 (2) | 0.3358 (2) | 0.77313 (18) | 0.0208 (5) |
| H214 | 0.9122     | 0.2767     | 0.7987       | 0.025*     |
| C212 | 0.9173 (2) | 0.5296 (2) | 0.79969 (17) | 0.0192 (5) |
| H212 | 0.9363     | 0.6028     | 0.8437       | 0.023*     |
| C6   | 0.7276 (3) | 0.4775 (2) | 1.04662 (18) | 0.0258 (6) |
| H6   | 0.7831     | 0.4780     | 1.0991       | 0.031*     |

|      |             |             |              |              |
|------|-------------|-------------|--------------|--------------|
| C4   | 0.5130 (3)  | 0.3559 (2)  | 0.94783 (18) | 0.0244 (6)   |
| H4   | 0.4364      | 0.2854      | 0.9435       | 0.029*       |
| C5   | 0.6098 (3)  | 0.3790 (2)  | 1.02256 (19) | 0.0281 (6)   |
| H5   | 0.5933      | 0.3215      | 1.0608       | 0.034*       |
| Cl1  | 0.82465 (6) | 0.96237 (6) | 0.01672 (5)  | 0.03396 (16) |
| C01  | 0.9005 (3)  | 1.1161 (3)  | 0.0739 (2)   | 0.0347 (7)   |
| H01A | 0.8877      | 1.1276      | 0.1442       | 0.042*       |
| H01B | 0.9926      | 1.1495      | 0.0736       | 0.042*       |
| Cl2  | 0.84113 (7) | 1.19298 (7) | 0.01169 (5)  | 0.03632 (17) |

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

|      | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$    | $U^{13}$     | $U^{23}$    |
|------|-------------|-------------|-------------|-------------|--------------|-------------|
| Ag   | 0.01416 (9) | 0.01288 (9) | 0.01448 (9) | 0.00751 (7) | 0.00373 (7)  | 0.00533 (6) |
| P2   | 0.0128 (3)  | 0.0121 (3)  | 0.0143 (3)  | 0.0071 (2)  | 0.0035 (2)   | 0.0041 (2)  |
| P1   | 0.0126 (3)  | 0.0120 (3)  | 0.0140 (3)  | 0.0069 (2)  | 0.0042 (2)   | 0.0046 (2)  |
| O1   | 0.0174 (9)  | 0.0154 (8)  | 0.0183 (8)  | 0.0046 (7)  | 0.0003 (7)   | 0.0043 (7)  |
| C236 | 0.0139 (12) | 0.0145 (12) | 0.0195 (12) | 0.0066 (10) | 0.0044 (9)   | 0.0067 (9)  |
| O2   | 0.0160 (9)  | 0.0265 (10) | 0.0211 (9)  | 0.0052 (8)  | 0.0008 (7)   | 0.0123 (7)  |
| C115 | 0.0213 (14) | 0.0205 (13) | 0.0322 (14) | 0.0099 (11) | 0.0124 (11)  | 0.0135 (11) |
| C211 | 0.0109 (11) | 0.0157 (12) | 0.0168 (11) | 0.0077 (10) | 0.0054 (9)   | 0.0081 (9)  |
| C131 | 0.0132 (11) | 0.0142 (11) | 0.0121 (11) | 0.0068 (10) | 0.0015 (9)   | 0.0014 (9)  |
| C116 | 0.0196 (13) | 0.0154 (12) | 0.0200 (12) | 0.0096 (10) | 0.0049 (10)  | 0.0070 (10) |
| C111 | 0.0123 (11) | 0.0113 (11) | 0.0192 (11) | 0.0083 (9)  | 0.0043 (9)   | 0.0039 (9)  |
| C132 | 0.0171 (12) | 0.0176 (12) | 0.0141 (11) | 0.0085 (10) | 0.0021 (9)   | 0.0045 (9)  |
| C231 | 0.0153 (11) | 0.0078 (10) | 0.0147 (11) | 0.0072 (9)  | 0.0017 (9)   | 0.0027 (8)  |
| C233 | 0.0185 (12) | 0.0166 (12) | 0.0179 (11) | 0.0104 (10) | 0.0049 (9)   | 0.0063 (9)  |
| C232 | 0.0120 (11) | 0.0120 (11) | 0.0210 (12) | 0.0062 (10) | 0.0030 (9)   | 0.0056 (9)  |
| C122 | 0.0170 (12) | 0.0182 (12) | 0.0180 (12) | 0.0099 (10) | 0.0054 (9)   | 0.0077 (9)  |
| C2   | 0.0186 (12) | 0.0181 (12) | 0.0149 (11) | 0.0102 (10) | 0.0066 (9)   | 0.0049 (9)  |
| C121 | 0.0111 (11) | 0.0124 (11) | 0.0145 (11) | 0.0038 (9)  | 0.0025 (9)   | 0.0057 (9)  |
| C216 | 0.0141 (12) | 0.0159 (12) | 0.0167 (11) | 0.0073 (10) | 0.0039 (9)   | 0.0071 (9)  |
| C221 | 0.0128 (11) | 0.0117 (11) | 0.0117 (10) | 0.0056 (9)  | 0.0012 (9)   | 0.0012 (8)  |
| C133 | 0.0264 (14) | 0.0221 (13) | 0.0207 (12) | 0.0179 (12) | 0.0012 (10)  | 0.0018 (10) |
| C235 | 0.0118 (12) | 0.0137 (12) | 0.0262 (13) | 0.0028 (10) | -0.0008 (10) | 0.0048 (10) |
| C123 | 0.0219 (13) | 0.0179 (12) | 0.0177 (12) | 0.0093 (11) | 0.0072 (10)  | 0.0056 (10) |
| C226 | 0.0162 (12) | 0.0159 (12) | 0.0184 (11) | 0.0105 (10) | 0.0041 (9)   | 0.0049 (9)  |
| C124 | 0.0206 (13) | 0.0200 (13) | 0.0155 (12) | 0.0063 (11) | -0.0003 (10) | 0.0054 (10) |
| C136 | 0.0189 (12) | 0.0171 (12) | 0.0175 (11) | 0.0091 (10) | 0.0067 (10)  | 0.0068 (9)  |
| C222 | 0.0179 (12) | 0.0139 (12) | 0.0236 (12) | 0.0088 (10) | 0.0027 (10)  | 0.0034 (10) |
| C215 | 0.0166 (12) | 0.0131 (12) | 0.0274 (13) | 0.0068 (10) | 0.0053 (10)  | 0.0042 (10) |
| C126 | 0.0161 (12) | 0.0144 (12) | 0.0211 (12) | 0.0081 (10) | 0.0037 (10)  | 0.0036 (9)  |
| C114 | 0.0138 (13) | 0.0137 (12) | 0.0420 (16) | 0.0049 (10) | 0.0077 (11)  | 0.0092 (11) |
| C224 | 0.0138 (12) | 0.0197 (13) | 0.0233 (13) | 0.0045 (10) | 0.0028 (10)  | 0.0051 (10) |
| C112 | 0.0179 (12) | 0.0173 (12) | 0.0216 (12) | 0.0095 (11) | 0.0053 (10)  | 0.0050 (10) |
| C113 | 0.0193 (13) | 0.0171 (13) | 0.0306 (14) | 0.0102 (11) | -0.0020 (11) | 0.0008 (10) |
| C223 | 0.0152 (12) | 0.0211 (13) | 0.0278 (13) | 0.0113 (11) | 0.0031 (10)  | 0.0056 (10) |
| C1   | 0.0202 (12) | 0.0164 (12) | 0.0128 (11) | 0.0118 (10) | 0.0055 (9)   | 0.0026 (9)  |

|      |             |             |             |             |              |             |
|------|-------------|-------------|-------------|-------------|--------------|-------------|
| C134 | 0.0240 (14) | 0.0336 (15) | 0.0175 (12) | 0.0202 (12) | 0.0061 (10)  | 0.0033 (11) |
| C125 | 0.0161 (12) | 0.0200 (13) | 0.0241 (13) | 0.0092 (11) | -0.0009 (10) | 0.0065 (10) |
| C234 | 0.0209 (13) | 0.0132 (12) | 0.0178 (12) | 0.0085 (10) | -0.0028 (10) | 0.0008 (9)  |
| C3   | 0.0211 (13) | 0.0193 (13) | 0.0161 (12) | 0.0071 (11) | 0.0045 (10)  | 0.0025 (10) |
| C7   | 0.0210 (13) | 0.0271 (14) | 0.0165 (12) | 0.0131 (11) | 0.0029 (10)  | 0.0063 (10) |
| C213 | 0.0232 (14) | 0.0280 (14) | 0.0205 (12) | 0.0142 (12) | 0.0031 (10)  | 0.0100 (11) |
| C225 | 0.0223 (13) | 0.0136 (12) | 0.0224 (12) | 0.0087 (11) | 0.0055 (10)  | 0.0052 (10) |
| C135 | 0.0216 (14) | 0.0314 (15) | 0.0218 (13) | 0.0133 (12) | 0.0095 (11)  | 0.0092 (11) |
| C214 | 0.0173 (13) | 0.0207 (13) | 0.0306 (14) | 0.0107 (11) | 0.0069 (11)  | 0.0146 (11) |
| C212 | 0.0222 (13) | 0.0202 (13) | 0.0182 (12) | 0.0130 (11) | 0.0039 (10)  | 0.0034 (10) |
| C6   | 0.0353 (16) | 0.0388 (16) | 0.0164 (12) | 0.0257 (14) | 0.0078 (11)  | 0.0134 (11) |
| C4   | 0.0320 (15) | 0.0166 (13) | 0.0232 (13) | 0.0085 (12) | 0.0139 (12)  | 0.0065 (10) |
| C5   | 0.0455 (18) | 0.0265 (15) | 0.0265 (14) | 0.0234 (14) | 0.0185 (13)  | 0.0173 (12) |
| C11  | 0.0284 (4)  | 0.0349 (4)  | 0.0261 (3)  | 0.0032 (3)  | 0.0014 (3)   | 0.0117 (3)  |
| C01  | 0.0299 (16) | 0.0410 (17) | 0.0203 (14) | 0.0081 (14) | -0.0073 (11) | 0.0050 (12) |
| C12  | 0.0302 (4)  | 0.0453 (4)  | 0.0321 (4)  | 0.0184 (3)  | 0.0015 (3)   | 0.0057 (3)  |

*Geometric parameters (Å, °)*

|           |             |           |           |
|-----------|-------------|-----------|-----------|
| Ag—O1     | 2.3612 (16) | C123—H123 | 0.9500    |
| Ag—O2     | 2.3342 (16) | C226—C225 | 1.384 (3) |
| Ag—P1     | 2.4070 (6)  | C226—H226 | 0.9500    |
| Ag—P2     | 2.4981 (5)  | C124—C125 | 1.384 (3) |
| P2—C231   | 1.821 (2)   | C124—H124 | 0.9500    |
| P2—C221   | 1.823 (2)   | C136—C135 | 1.389 (3) |
| P2—C211   | 1.824 (2)   | C136—H136 | 0.9500    |
| P1—C131   | 1.820 (2)   | C222—C223 | 1.387 (3) |
| P1—C111   | 1.825 (2)   | C222—H222 | 0.9500    |
| P1—C121   | 1.829 (2)   | C215—C214 | 1.382 (3) |
| O1—C1     | 1.271 (3)   | C215—H215 | 0.9500    |
| C236—C235 | 1.380 (3)   | C126—C125 | 1.385 (3) |
| C236—C231 | 1.397 (3)   | C126—H126 | 0.9500    |
| C236—H236 | 0.9500      | C114—C113 | 1.383 (3) |
| O2—C2     | 1.261 (3)   | C114—H114 | 0.9500    |
| C115—C114 | 1.381 (4)   | C224—C225 | 1.385 (3) |
| C115—C116 | 1.386 (3)   | C224—C223 | 1.388 (3) |
| C115—H115 | 0.9500      | C224—H224 | 0.9500    |
| C211—C216 | 1.392 (3)   | C112—C113 | 1.390 (3) |
| C211—C212 | 1.399 (3)   | C112—H112 | 0.9500    |
| C131—C132 | 1.386 (3)   | C113—H113 | 0.9500    |
| C131—C136 | 1.395 (3)   | C223—H223 | 0.9500    |
| C116—C111 | 1.391 (3)   | C1—C7     | 1.414 (3) |
| C116—H116 | 0.9500      | C134—C135 | 1.379 (3) |
| C111—C112 | 1.395 (3)   | C134—H134 | 0.9500    |
| C132—C133 | 1.393 (3)   | C125—H125 | 0.9500    |
| C132—H132 | 0.9500      | C234—H234 | 0.9500    |
| C231—C232 | 1.395 (3)   | C3—C4     | 1.373 (3) |
| C233—C234 | 1.383 (3)   | C3—H3     | 0.9500    |

|                |              |                |           |
|----------------|--------------|----------------|-----------|
| C233—C232      | 1.390 (3)    | C7—C6          | 1.382 (3) |
| C233—H233      | 0.9500       | C7—H7          | 0.9500    |
| C232—H232      | 0.9500       | C213—C212      | 1.376 (3) |
| C122—C123      | 1.377 (3)    | C213—C214      | 1.387 (3) |
| C122—C121      | 1.400 (3)    | C213—H213      | 0.9500    |
| C122—H122      | 0.9500       | C225—H225      | 0.9500    |
| C2—C3          | 1.427 (3)    | C135—H135      | 0.9500    |
| C2—C1          | 1.486 (3)    | C214—H214      | 0.9500    |
| C121—C126      | 1.391 (3)    | C212—H212      | 0.9500    |
| C216—C215      | 1.387 (3)    | C6—C5          | 1.379 (4) |
| C216—H216      | 0.9500       | C6—H6          | 0.9500    |
| C221—C222      | 1.393 (3)    | C4—C5          | 1.388 (4) |
| C221—C226      | 1.394 (3)    | C4—H4          | 0.9500    |
| C133—C134      | 1.378 (3)    | C5—H5          | 0.9500    |
| C133—H133      | 0.9500       | C11—C01        | 1.770 (3) |
| C235—C234      | 1.390 (3)    | C01—C12        | 1.768 (3) |
| C235—H235      | 0.9500       | C01—H01A       | 0.9900    |
| C123—C124      | 1.390 (3)    | C01—H01B       | 0.9900    |
| O2—Ag—O1       | 68.67 (6)    | C125—C124—H124 | 120.3     |
| O2—Ag—P1       | 121.43 (4)   | C123—C124—H124 | 120.3     |
| O1—Ag—P1       | 128.35 (4)   | C135—C136—C131 | 119.8 (2) |
| O2—Ag—P2       | 103.97 (4)   | C135—C136—H136 | 120.1     |
| O1—Ag—P2       | 98.81 (4)    | C131—C136—H136 | 120.1     |
| P1—Ag—P2       | 122.391 (19) | C223—C222—C221 | 120.7 (2) |
| C231—P2—C221   | 103.35 (10)  | C223—C222—H222 | 119.6     |
| C231—P2—C211   | 103.57 (10)  | C221—C222—H222 | 119.6     |
| C221—P2—C211   | 103.62 (10)  | C214—C215—C216 | 120.5 (2) |
| C231—P2—Ag     | 111.93 (7)   | C214—C215—H215 | 119.8     |
| C221—P2—Ag     | 114.95 (7)   | C216—C215—H215 | 119.8     |
| C211—P2—Ag     | 117.80 (7)   | C125—C126—C121 | 120.6 (2) |
| C131—P1—C111   | 104.60 (10)  | C125—C126—H126 | 119.7     |
| C131—P1—C121   | 103.57 (10)  | C121—C126—H126 | 119.7     |
| C111—P1—C121   | 104.03 (10)  | C115—C114—C113 | 119.9 (2) |
| C131—P1—Ag     | 118.60 (7)   | C115—C114—H114 | 120.1     |
| C111—P1—Ag     | 110.63 (7)   | C113—C114—H114 | 120.1     |
| C121—P1—Ag     | 114.02 (7)   | C225—C224—C223 | 119.7 (2) |
| C1—O1—Ag       | 115.51 (14)  | C225—C224—H224 | 120.1     |
| C235—C236—C231 | 120.3 (2)    | C223—C224—H224 | 120.1     |
| C235—C236—H236 | 119.8        | C113—C112—C111 | 120.3 (2) |
| C231—C236—H236 | 119.8        | C113—C112—H112 | 119.8     |
| C2—O2—Ag       | 117.18 (14)  | C111—C112—H112 | 119.8     |
| C114—C115—C116 | 120.4 (2)    | C114—C113—C112 | 120.0 (2) |
| C114—C115—H115 | 119.8        | C114—C113—H113 | 120.0     |
| C116—C115—H115 | 119.8        | C112—C113—H113 | 120.0     |
| C216—C211—C212 | 119.0 (2)    | C222—C223—C224 | 119.9 (2) |
| C216—C211—P2   | 123.18 (17)  | C222—C223—H223 | 120.0     |
| C212—C211—P2   | 117.83 (17)  | C224—C223—H223 | 120.0     |



|                |             |                |             |
|----------------|-------------|----------------|-------------|
| C132—C131—C136 | 119.41 (19) | O1—C1—C7       | 118.7 (2)   |
| C132—C131—P1   | 122.58 (16) | O1—C1—C2       | 117.0 (2)   |
| C136—C131—P1   | 117.97 (16) | C7—C1—C2       | 124.2 (2)   |
| C115—C116—C111 | 120.2 (2)   | C133—C134—C135 | 120.5 (2)   |
| C115—C116—H116 | 119.9       | C133—C134—H134 | 119.7       |
| C111—C116—H116 | 119.9       | C135—C134—H134 | 119.7       |
| C116—C111—C112 | 119.1 (2)   | C124—C125—C126 | 120.4 (2)   |
| C116—C111—P1   | 123.35 (18) | C124—C125—H125 | 119.8       |
| C112—C111—P1   | 117.30 (17) | C126—C125—H125 | 119.8       |
| C131—C132—C133 | 120.4 (2)   | C233—C234—C235 | 119.3 (2)   |
| C131—C132—H132 | 119.8       | C233—C234—H234 | 120.3       |
| C133—C132—H132 | 119.8       | C235—C234—H234 | 120.3       |
| C232—C231—C236 | 118.8 (2)   | C4—C3—C2       | 132.4 (2)   |
| C232—C231—P2   | 122.87 (17) | C4—C3—H3       | 113.8       |
| C236—C231—P2   | 118.21 (16) | C2—C3—H3       | 113.8       |
| C234—C233—C232 | 120.3 (2)   | C6—C7—C1       | 132.6 (2)   |
| C234—C233—H233 | 119.8       | C6—C7—H7       | 113.7       |
| C232—C233—H233 | 119.8       | C1—C7—H7       | 113.7       |
| C233—C232—C231 | 120.5 (2)   | C212—C213—C214 | 120.5 (2)   |
| C233—C232—H232 | 119.8       | C212—C213—H213 | 119.7       |
| C231—C232—H232 | 119.8       | C214—C213—H213 | 119.7       |
| C123—C122—C121 | 120.7 (2)   | C226—C225—C224 | 120.4 (2)   |
| C123—C122—H122 | 119.6       | C226—C225—H225 | 119.8       |
| C121—C122—H122 | 119.6       | C224—C225—H225 | 119.8       |
| O2—C2—C3       | 118.2 (2)   | C134—C135—C136 | 120.2 (2)   |
| O2—C2—C1       | 117.6 (2)   | C134—C135—H135 | 119.9       |
| C3—C2—C1       | 124.2 (2)   | C136—C135—H135 | 119.9       |
| C126—C121—C122 | 118.5 (2)   | C215—C214—C213 | 119.5 (2)   |
| C126—C121—P1   | 123.10 (16) | C215—C214—H214 | 120.2       |
| C122—C121—P1   | 118.31 (17) | C213—C214—H214 | 120.2       |
| C215—C216—C211 | 120.2 (2)   | C213—C212—C211 | 120.3 (2)   |
| C215—C216—H216 | 119.9       | C213—C212—H212 | 119.8       |
| C211—C216—H216 | 119.9       | C211—C212—H212 | 119.8       |
| C222—C221—C226 | 118.8 (2)   | C5—C6—C7       | 130.0 (3)   |
| C222—C221—P2   | 122.69 (17) | C5—C6—H6       | 115.0       |
| C226—C221—P2   | 118.54 (16) | C7—C6—H6       | 115.0       |
| C134—C133—C132 | 119.6 (2)   | C3—C4—C5       | 129.8 (2)   |
| C134—C133—H133 | 120.2       | C3—C4—H4       | 115.1       |
| C132—C133—H133 | 120.2       | C5—C4—H4       | 115.1       |
| C236—C235—C234 | 120.8 (2)   | C6—C5—C4       | 126.3 (2)   |
| C236—C235—H235 | 119.6       | C6—C5—H5       | 116.9       |
| C234—C235—H235 | 119.6       | C4—C5—H5       | 116.9       |
| C122—C123—C124 | 120.3 (2)   | C12—C01—C11    | 111.60 (15) |
| C122—C123—H123 | 119.8       | C12—C01—H01A   | 109.3       |
| C124—C123—H123 | 119.8       | C11—C01—H01A   | 109.3       |
| C225—C226—C221 | 120.5 (2)   | C12—C01—H01B   | 109.3       |
| C225—C226—H226 | 119.7       | C11—C01—H01B   | 109.3       |
| C221—C226—H226 | 119.7       | H01A—C01—H01B  | 108.0       |

|                     |              |                     |              |
|---------------------|--------------|---------------------|--------------|
| C125—C124—C123      | 119.4 (2)    |                     |              |
| O2—Ag—P2—C231       | 83.92 (9)    | C131—P1—C121—C126   | 13.0 (2)     |
| O1—Ag—P2—C231       | 153.99 (8)   | C111—P1—C121—C126   | -96.14 (19)  |
| P1—Ag—P2—C231       | -58.66 (8)   | Ag—P1—C121—C126     | 143.27 (17)  |
| O2—Ag—P2—C221       | -158.57 (9)  | C131—P1—C121—C122   | -163.60 (17) |
| O1—Ag—P2—C221       | -88.49 (9)   | C111—P1—C121—C122   | 87.28 (18)   |
| P1—Ag—P2—C221       | 58.85 (8)    | Ag—P1—C121—C122     | -33.31 (19)  |
| O2—Ag—P2—C211       | -35.96 (10)  | C212—C211—C216—C215 | -0.9 (3)     |
| O1—Ag—P2—C211       | 34.11 (10)   | P2—C211—C216—C215   | 179.11 (16)  |
| P1—Ag—P2—C211       | -178.54 (9)  | C231—P2—C221—C222   | -86.0 (2)    |
| O2—Ag—P1—C131       | 44.96 (10)   | C211—P2—C221—C222   | 21.8 (2)     |
| O1—Ag—P1—C131       | -41.59 (10)  | Ag—P2—C221—C222     | 151.70 (16)  |
| P2—Ag—P1—C131       | -178.76 (8)  | C231—P2—C221—C226   | 92.32 (18)   |
| O2—Ag—P1—C111       | 165.75 (9)   | C211—P2—C221—C226   | -159.88 (17) |
| O1—Ag—P1—C111       | 79.20 (9)    | Ag—P2—C221—C226     | -29.94 (19)  |
| P2—Ag—P1—C111       | -57.97 (8)   | C131—C132—C133—C134 | -0.7 (4)     |
| O2—Ag—P1—C121       | -77.42 (9)   | C231—C236—C235—C234 | -0.3 (3)     |
| O1—Ag—P1—C121       | -163.97 (8)  | C121—C122—C123—C124 | -0.5 (3)     |
| P2—Ag—P1—C121       | 58.86 (8)    | C222—C221—C226—C225 | 0.7 (3)      |
| O2—Ag—O1—C1         | 17.65 (14)   | P2—C221—C226—C225   | -177.75 (17) |
| P1—Ag—O1—C1         | 131.53 (13)  | C122—C123—C124—C125 | 0.9 (3)      |
| P2—Ag—O1—C1         | -83.99 (14)  | C132—C131—C136—C135 | 1.5 (3)      |
| O1—Ag—O2—C2         | -15.03 (15)  | P1—C131—C136—C135   | -176.21 (19) |
| P1—Ag—O2—C2         | -137.85 (14) | C226—C221—C222—C223 | -0.8 (3)     |
| P2—Ag—O2—C2         | 79.12 (15)   | P2—C221—C222—C223   | 177.51 (18)  |
| C231—P2—C211—C216   | 5.0 (2)      | C211—C216—C215—C214 | 0.0 (3)      |
| C221—P2—C211—C216   | -102.68 (19) | C122—C121—C126—C125 | 0.4 (3)      |
| Ag—P2—C211—C216     | 129.12 (16)  | P1—C121—C126—C125   | -176.20 (18) |
| C231—P2—C211—C212   | -175.02 (17) | C116—C115—C114—C113 | 1.0 (4)      |
| C221—P2—C211—C212   | 77.34 (18)   | C116—C111—C112—C113 | 1.3 (3)      |
| Ag—P2—C211—C212     | -50.86 (19)  | P1—C111—C112—C113   | 175.55 (17)  |
| C111—P1—C131—C132   | 30.9 (2)     | C115—C114—C113—C112 | -0.1 (3)     |
| C121—P1—C131—C132   | -77.8 (2)    | C111—C112—C113—C114 | -1.1 (3)     |
| Ag—P1—C131—C132     | 154.70 (17)  | C221—C222—C223—C224 | 0.5 (4)      |
| C111—P1—C131—C136   | -151.45 (18) | C225—C224—C223—C222 | 0.0 (4)      |
| C121—P1—C131—C136   | 99.85 (19)   | Ag—O1—C1—C7         | 158.97 (15)  |
| Ag—P1—C131—C136     | -27.6 (2)    | Ag—O1—C1—C2         | -18.7 (2)    |
| C114—C115—C116—C111 | -0.7 (3)     | O2—C2—C1—O1         | 5.2 (3)      |
| C115—C116—C111—C112 | -0.4 (3)     | C3—C2—C1—O1         | -173.26 (19) |
| C115—C116—C111—P1   | -174.29 (17) | O2—C2—C1—C7         | -172.4 (2)   |
| C131—P1—C111—C116   | -113.66 (19) | C3—C2—C1—C7         | 9.2 (3)      |
| C121—P1—C111—C116   | -5.3 (2)     | C132—C133—C134—C135 | 0.8 (4)      |
| Ag—P1—C111—C116     | 117.54 (17)  | C123—C124—C125—C126 | -0.6 (4)     |
| C131—P1—C111—C112   | 72.36 (18)   | C121—C126—C125—C124 | 0.0 (4)      |
| C121—P1—C111—C112   | -179.28 (16) | C232—C233—C234—C235 | -1.0 (3)     |
| Ag—P1—C111—C112     | -56.44 (17)  | C236—C235—C234—C233 | 0.9 (3)      |
| C136—C131—C132—C133 | -0.5 (3)     | O2—C2—C3—C4         | 174.6 (2)    |

---

|                     |              |                     |              |
|---------------------|--------------|---------------------|--------------|
| P1—C131—C132—C133   | 177.16 (18)  | C1—C2—C3—C4         | -7.0 (4)     |
| C235—C236—C231—C232 | -0.2 (3)     | O1—C1—C7—C6         | 178.7 (2)    |
| C235—C236—C231—P2   | 176.33 (17)  | C2—C1—C7—C6         | -3.8 (4)     |
| C221—P2—C231—C232   | 6.6 (2)      | C221—C226—C225—C224 | -0.2 (3)     |
| C211—P2—C231—C232   | -101.18 (19) | C223—C224—C225—C226 | -0.1 (4)     |
| Ag—P2—C231—C232     | 130.91 (16)  | C133—C134—C135—C136 | 0.3 (4)      |
| C221—P2—C231—C236   | -169.69 (17) | C131—C136—C135—C134 | -1.5 (4)     |
| C211—P2—C231—C236   | 82.48 (18)   | C216—C215—C214—C213 | 0.6 (3)      |
| Ag—P2—C231—C236     | -45.43 (19)  | C212—C213—C214—C215 | -0.4 (3)     |
| C234—C233—C232—C231 | 0.6 (3)      | C214—C213—C212—C211 | -0.5 (3)     |
| C236—C231—C232—C233 | 0.0 (3)      | C216—C211—C212—C213 | 1.2 (3)      |
| P2—C231—C232—C233   | -176.29 (17) | P2—C211—C212—C213   | -178.86 (17) |
| Ag—O2—C2—C3         | -169.93 (14) | C1—C7—C6—C5         | -2.5 (4)     |
| Ag—O2—C2—C1         | 11.5 (2)     | C2—C3—C4—C5         | -0.4 (4)     |
| C123—C122—C121—C126 | -0.1 (3)     | C7—C6—C5—C4         | 1.3 (4)      |
| C123—C122—C121—P1   | 176.62 (17)  | C3—C4—C5—C6         | 2.8 (4)      |

---