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# (Ferrocenylthiophosphonato-κ*S*)-(triphenylphosphane-κ*P*)gold(I) dichloromethane monosolvate

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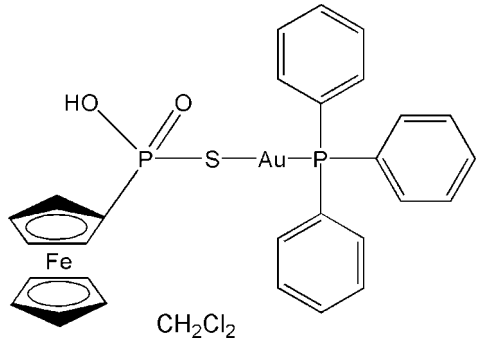
Key indicators: single-crystal X-ray study; $T$ = 173 K; mean $\sigma$(C–C) = 0.012 Å; disorder in solvent or counterion; $R$ factor = 0.051; $wR$ factor = 0.153; data-to-parameter ratio = 13.1.

In the title compound, $[AuFe(C_5H_5)(C_5H_5O_2PS)(C_{18}H_{15}P)]\cdot CH_2Cl_2$, the two-coordinate gold(I) atom shows a slightly distorted linear arrangement, with a P—Au—S bond angle of 176.81 (6)°. The difference in P=O and P—O(H) bond lengths, which are 1.503 (6) and 1.541 (5) Å, respectively, implies there is apparently no delocalization between the P—O bonds, and the proton appears to be localized on one O atom only. In the crystal structure, intermolecular O—H···O hydrogen bonds link dinuclear molecules into chains propagated in the [010] direction. The dichloromethane solvent molecule was disordered between two positions in a 0.63 (3):0.37 (3) ratio.

## Related literature

For information on dithiophosphonate complexes of Group 11 metals, see: Van Zyl (2010). For the synthesis of dithiophosphonate salt derivatives, see: Van Zyl & Fackler (2000). For gold complexes with thiophosphoryl-based ligands, see: Crespo *et al.* (2004). For gold complexes with dithiophosphate phosphine gold(I) complexes, see: Preisenberger *et al.* (1998). For the synthesis of ferrocenyl (Fc) dimers of the type $[PS_2(Fc)]_2$, see: Foreman *et al.* (1996). For general background, see: Allen (2002).




## Experimental

### Crystal data

$[AuFe(C_5H_5)(C_5H_5O_2PS)(C_{18}H_{15}P)]\cdot CH_2Cl_2$
$M_r$ = 825.22
Monoclinic, $P2_1/c$
$a$ = 15.122 (3) Å
$b$ = 9.3157 (18) Å
$c$ = 22.581 (3) Å
$\beta$ = 112.831 (10)°
$V$ = 2931.8 (9) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 5.88 mm$^{-1}$
$T$ = 173 K
0.20 × 0.18 × 0.16 mm

### Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
$T_{min}$ = 0.386, $T_{max}$ = 0.453
13808 measured reflections
4924 independent reflections
4348 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.112

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.153
$S$ = 1.10
4924 reflections
375 parameters
18 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 2.14 e Å$^{-3}$
$\Delta\rho_{min}$ = −1.63 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O2—H1···O1$^{i}$ | 1.11 (11) | 1.33 (11) | 2.432 (7) | 173 (9) |

Symmetry code: (i) $-x, y+\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *APEX2* (Bruker 2006); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2003); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Berndt, 2001); 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: CV2770).

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# supplementary materials

*Acta Cryst.* (2010). E**66**, m1364-m1365 [ **doi:10.1107/S1600536810039255** ]

# (Ferrocenylthiophosphonato-$\kappa S$)(triphenylphosphane-$\kappa P$)gold(I) dichloromethane monosolvate

**H. van der Walt, A. Muller, R. J. Staples and W. E. Van Zyl**

## Comment

The dithiophosphonato dianion salt, $(NH_4)_2[S_2P(Fc)OCH_2CH_2O(Fc)PS_2]$ (Fc = ferrocenyl) was used in this study. It was obtained from the reaction between the dimer $[PS_2(Fc)]_2$ (Foreman *et al.*, 1996) and ethanediol, which formed a diacid. The diacid could be readily deprotonated by ammonia gas. The salt was then reacted with $[Au(PPh_3)Cl]$ to yield the complex $[(PPh_3)AuS_2(Fc)P(OC_2H_4O)P(Fc)S_2Au(PPh_3)]$ (I). The complex was fully characterized spectroscopically (see Experimental). During work-up for crystal growth, however, the ligand became oxidized, presumably as a result of water present in the solvent dichloromethane. This resulted in substitution of a terminal P═S bond with a P═O bond, a reaction that can readily occur with oxophilic phosphorus(V) in the presence of moisture. Additionally, it resulted in C—O bond cleavage to be replaced with H—O bond formation. A precise mechanism for the reaction is not proposed. The title compound I (see Fig 1) crystalizes in the $P2_1/c$ space group with molecules lying on general positions in the unit cell. All geometrical data for the compound are within the normal limits (Allen, 2002). In the crystal packing, there is a hydrogen-bonding network along the *b* axis between the P═O···H—O—P moieties (see Fig 2 and Table 1).

## Experimental

The complex (I) was obtained as an oxidized product. The initial reaction was between the $(NH_4)_2[S_2P(Fc)OCH_2CH_2O(Fc)PS_2]$ salt (Fc = ferrocenyl) (0.122 g, 0.206 mmol) and $[Au(PPh_3)Cl]$ (0.200 g, 0.413 mmol) in a THF solution. The $NH_4Cl$ was filtered off and the filtrate solvent removed under reduced pressure yielding a yellow colored powder. Yield: 0.127 g (41%). *M*.p.: 128°C. $^1$H-NMR ($CDCl_3$) δ: 7.54 – 7.45 (m, 30H, $PPh_3$); 4.65 – 4.22 (d, 4H, Fc); 4.53 – 4.50 (d, 4H, Fc); 4.30 (s, 10H, Fc); 4.36 – 4.31 (t, 2H, $CH_2$); 3.99 – 3.93 (m, 2H, $CH_2$). $^{31}$P-NMR δ: 106.14 and 106.03 (d, 2P, P—S); 37.82 (s, 2P, $PPh_3$). ESI-MS: m/*z* 1539 (95%) for $[(PPh_3)AuS_2(Fc)P(OC_2H_4O)P(Fc)S_2Au(PPh_3)]$.

## Refinement

The aromatic H atoms were placed in geometrically idealized positions (C—H = 0.95 Å) and constrained to ride on their parent atoms with $U_{iso}(H) = 1.2U_{eq}(C)$. The hydroxyl H was located in a Fourier difference map and refined isotropically. The disorder of the solvent molecule was refined over two positions that adds to unity. The final refinement shows a ratio of 37:63 for the two components. The neccesary bond and $U^{ij}$ restraints were applied to keep the refinement stable.

## Figures

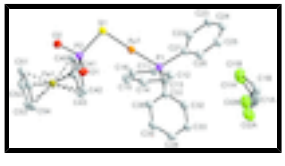

Fig. 1. View of (I) (30% probability displacement ellipsoids). Hydrogen atoms omitted for clarity.

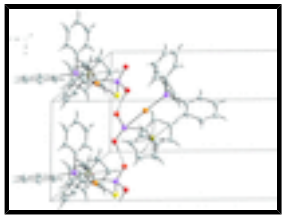

Fig. 2. Packing diagram of (I) showing the infinite hydrogen bonding interactions (dashed lines) along the *b* axis. Solvent molecules omitted for clarity.

**(Ferrocenylthiophosphonato-κ*S*)(triphenylphosphane-κ*P*)gold(I) dichloromethane monosolvate**

*Crystal data*

| | |
|---|---|
| $[AuFe(C_5H_5)(C_5H_5O_2PS)(C_{18}H_{15}P)]\cdot CH_2Cl_2$ | $F(000) = 1608$ |
| $M_r = 825.22$ | $D_x = 1.87$ Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Hall symbol: -P 2ybc | Cell parameters from 9574 reflections |
| $a = 15.122$ (3) Å | $\theta = 2.2$–$27.9°$ |
| $b = 9.3157$ (18) Å | $\mu = 5.88$ mm$^{-1}$ |
| $c = 22.581$ (3) Å | $T = 173$ K |
| $\beta = 112.831$ (10)° | Block, orange-brown |
| $V = 2931.8$ (9) Å$^3$ | 0.2 × 0.18 × 0.16 mm |
| $Z = 4$ | |

*Data collection*

| | |
|---|---|
| Bruker CCD diffractometer | 4348 reflections with $I > 2\sigma(I)$ |
| graphite | $R_{int} = 0.112$ |
| φ scans | $\theta_{max} = 25°$, $\theta_{min} = 1.5°$ |
| Absorption correction: multi-scan (*SADABS*; Bruker, 1998) | $h = -16\rightarrow17$ |
| $T_{min} = 0.386$, $T_{max} = 0.453$ | $k = -11\rightarrow11$ |
| 13808 measured reflections | $l = -26\rightarrow20$ |
| 4924 independent reflections | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.051$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.153$ | H atoms treated by a mixture of independent and constrained refinement |
| $S = 1.10$ | $w = 1/[\sigma^2(F_o^2) + (0.095P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 4924 reflections | $(\Delta/\sigma)_{max} = 0.001$ |
| 375 parameters | $\Delta\rho_{max} = 2.14$ e Å$^{-3}$ |

18 restraints $\Delta\rho_{min} = -1.63$ e Å$^{-3}$

*Special details*

**Experimental**. The intensity data was collected on a Bruker CCD based diffractometer equipped with an Oxford Cryostream low-temperature apparatus operating at 173 K diffractometer using an exposure time of 30 s/frame. A total of 1276 frames were collected with a frame width of 0.5° covering up to θ = 28.63% with 86.5% completeness accomplished. Due to decomposition and the weak diffracting nature of the title compound, refinement of data was restricted to θ = 25.0% to reach a completeness of 95.5%.

**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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Au1 | 0.227778 (18) | 0.21410 (3) | 0.709726 (13) | 0.04416 (16) | |
| Fe1 | −0.15433 (8) | 0.30798 (11) | 0.60212 (6) | 0.0463 (3) | |
| S1 | 0.17770 (13) | 0.35983 (19) | 0.77340 (10) | 0.0499 (4) | |
| P1 | 0.28030 (12) | 0.08231 (19) | 0.64580 (9) | 0.0407 (4) | |
| P2 | 0.03846 (15) | 0.29088 (17) | 0.74410 (11) | 0.0443 (5) | |
| O1 | 0.0279 (4) | 0.1480 (6) | 0.7716 (3) | 0.0528 (13) | |
| O2 | −0.0137 (4) | 0.4034 (6) | 0.7688 (3) | 0.0502 (12) | |
| C11 | 0.2290 (5) | 0.1421 (7) | 0.5625 (3) | 0.0439 (15) | |
| C12 | 0.2703 (5) | 0.1110 (8) | 0.5188 (4) | 0.0515 (17) | |
| H12 | 0.3305 | 0.0634 | 0.5326 | 0.062* | |
| C13 | 0.2237 (6) | 0.1493 (8) | 0.4552 (4) | 0.0544 (18) | |
| H13 | 0.2521 | 0.1274 | 0.4254 | 0.065* | |
| C14 | 0.1361 (7) | 0.2190 (8) | 0.4340 (5) | 0.058 (2) | |
| H14 | 0.1054 | 0.2476 | 0.3904 | 0.069* | |
| C15 | 0.0933 (7) | 0.2469 (10) | 0.4779 (4) | 0.058 (2) | |
| H15 | 0.0317 | 0.2903 | 0.4639 | 0.069* | |
| C16 | 0.1411 (6) | 0.2112 (7) | 0.5408 (4) | 0.0514 (19) | |
| H16 | 0.1132 | 0.2344 | 0.5707 | 0.062* | |
| C21 | 0.4100 (5) | 0.0891 (7) | 0.6701 (3) | 0.0435 (15) | |
| C22 | 0.4526 (6) | 0.2000 (8) | 0.6510 (4) | 0.0516 (19) | |
| H22 | 0.4133 | 0.2703 | 0.6224 | 0.062* | |
| C23 | 0.5507 (6) | 0.2118 (7) | 0.6723 (5) | 0.055 (2) | |
| H23 | 0.579 | 0.2881 | 0.6579 | 0.067* | |
| C24 | 0.6073 (5) | 0.1112 (9) | 0.7150 (4) | 0.0542 (18) | |
| H24 | 0.6751 | 0.118 | 0.7295 | 0.065* | |
| C25 | 0.5671 (5) | 0.0007 (9) | 0.7369 (4) | 0.063 (2) | |
| H25 | 0.6069 | −0.0661 | 0.7674 | 0.075* | |

| | | | | | |
|---|---|---|---|---|---|
| C26 | 0.4683 (5) | −0.0116 (8) | 0.7141 (4) | 0.0522 (17) | |
| H26 | 0.44 | −0.0884 | 0.7282 | 0.063* | |
| C31 | 0.2488 (5) | −0.1068 (7) | 0.6423 (3) | 0.0428 (15) | |
| C32 | 0.2881 (6) | −0.2077 (7) | 0.6148 (4) | 0.0499 (19) | |
| H32 | 0.3353 | −0.18 | 0.599 | 0.06* | |
| C33 | 0.2582 (6) | −0.3508 (8) | 0.6105 (4) | 0.0540 (18) | |
| H33 | 0.2868 | −0.4213 | 0.5931 | 0.065* | |
| C34 | 0.1871 (5) | −0.3890 (9) | 0.6314 (4) | 0.0559 (19) | |
| H34 | 0.1657 | −0.4858 | 0.6274 | 0.067* | |
| C35 | 0.1471 (6) | −0.2889 (8) | 0.6581 (5) | 0.058 (2) | |
| H35 | 0.0991 | −0.3167 | 0.6732 | 0.07* | |
| C36 | 0.1766 (5) | −0.1480 (8) | 0.6628 (4) | 0.0505 (17) | |
| H36 | 0.1476 | −0.0784 | 0.6803 | 0.061* | |
| C41 | −0.0125 (5) | 0.2862 (7) | 0.6583 (4) | 0.0452 (18) | |
| C42 | −0.0462 (5) | 0.1626 (8) | 0.6188 (4) | 0.0476 (17) | |
| H42 | −0.0448 | 0.0669 | 0.6336 | 0.057* | |
| C43 | −0.0826 (6) | 0.2068 (8) | 0.5530 (4) | 0.0490 (18) | |
| H43 | −0.1094 | 0.1461 | 0.5166 | 0.059* | |
| C44 | −0.0713 (5) | 0.3575 (8) | 0.5522 (4) | 0.0504 (17) | |
| H44 | −0.0896 | 0.4162 | 0.5149 | 0.061* | |
| C45 | −0.0278 (5) | 0.4060 (8) | 0.6169 (4) | 0.0467 (16) | |
| H45 | −0.0117 | 0.5027 | 0.6302 | 0.056* | |
| C51 | −0.2328 (6) | 0.4488 (10) | 0.6316 (5) | 0.070 (2) | |
| H51 | −0.2125 | 0.5385 | 0.6524 | 0.083* | |
| C52 | −0.2765 (6) | 0.4242 (12) | 0.5630 (5) | 0.074 (3) | |
| H52 | −0.2884 | 0.4942 | 0.5302 | 0.089* | |
| C53 | −0.2981 (6) | 0.2771 (10) | 0.5542 (5) | 0.069 (3) | |
| H53 | −0.329 | 0.2308 | 0.5139 | 0.082* | |
| C54 | −0.2672 (7) | 0.2106 (10) | 0.6135 (6) | 0.066 (3) | |
| H54 | −0.2727 | 0.1112 | 0.6207 | 0.08* | |
| C55 | −0.2263 (6) | 0.3149 (10) | 0.6614 (5) | 0.060 (2) | |
| H55 | −0.1988 | 0.2976 | 0.7064 | 0.072* | |
| H1 | −0.020 (7) | 0.512 (12) | 0.747 (4) | 0.09 (3)* | |
| C1A | 0.539 (6) | −0.310 (3) | 0.556 (4) | 0.18 (4) | 0.37 (3) |
| H1A1 | 0.5102 | −0.3393 | 0.51 | 0.217* | 0.37 (3) |
| H1A2 | 0.6083 | −0.3287 | 0.5704 | 0.217* | 0.37 (3) |
| Cl1A | 0.528 (3) | −0.134 (3) | 0.5549 (15) | 0.094 (5) | 0.37 (3) |
| Cl2A | 0.4991 (7) | −0.427 (2) | 0.5939 (16) | 0.117 (8) | 0.37 (3) |
| C1B | 0.5793 (14) | −0.284 (2) | 0.6005 (13) | 0.093 (8) | 0.63 (3) |
| H1B1 | 0.5783 | −0.3424 | 0.5636 | 0.112* | 0.63 (3) |
| H1B2 | 0.6472 | −0.2746 | 0.6303 | 0.112* | 0.63 (3) |
| Cl1B | 0.5391 (15) | −0.1218 (16) | 0.5733 (8) | 0.081 (3) | 0.63 (3) |
| Cl2B | 0.5235 (9) | −0.3745 (11) | 0.6373 (9) | 0.106 (5) | 0.63 (3) |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Au1 | 0.0426 (2) | 0.0476 (2) | 0.0452 (2) | −0.00013 (9) | 0.02019 (16) | −0.00134 (11) |

| | | | | | | |
|---|---|---|---|---|---|---|
| Fe1 | 0.0409 (6) | 0.0511 (5) | 0.0486 (7) | −0.0013 (4) | 0.0191 (5) | −0.0046 (5) |
| S1 | 0.0459 (9) | 0.0530 (10) | 0.0529 (11) | −0.0054 (7) | 0.0215 (8) | −0.0097 (9) |
| P1 | 0.0396 (9) | 0.0443 (8) | 0.0418 (10) | −0.0003 (7) | 0.0196 (7) | 0.0001 (8) |
| P2 | 0.0471 (11) | 0.0430 (10) | 0.0443 (11) | −0.0016 (7) | 0.0192 (9) | −0.0016 (8) |
| O1 | 0.072 (3) | 0.042 (3) | 0.048 (3) | −0.005 (2) | 0.028 (3) | 0.001 (2) |
| O2 | 0.056 (3) | 0.050 (3) | 0.054 (3) | 0.000 (2) | 0.031 (3) | −0.002 (2) |
| C11 | 0.045 (4) | 0.047 (4) | 0.041 (4) | −0.003 (3) | 0.018 (3) | −0.001 (3) |
| C12 | 0.049 (4) | 0.054 (4) | 0.051 (4) | 0.003 (3) | 0.019 (3) | 0.000 (4) |
| C13 | 0.065 (5) | 0.057 (4) | 0.046 (4) | 0.003 (4) | 0.027 (4) | 0.008 (4) |
| C14 | 0.060 (5) | 0.059 (5) | 0.047 (5) | −0.001 (3) | 0.013 (4) | 0.009 (4) |
| C15 | 0.060 (5) | 0.064 (4) | 0.050 (5) | 0.013 (4) | 0.022 (4) | 0.010 (4) |
| C16 | 0.042 (4) | 0.061 (5) | 0.059 (5) | 0.005 (3) | 0.028 (4) | 0.008 (4) |
| C21 | 0.036 (3) | 0.048 (3) | 0.047 (4) | −0.005 (3) | 0.017 (3) | −0.009 (3) |
| C22 | 0.053 (5) | 0.049 (4) | 0.053 (5) | 0.003 (3) | 0.021 (4) | 0.002 (3) |
| C23 | 0.046 (4) | 0.056 (5) | 0.070 (6) | −0.006 (3) | 0.028 (4) | −0.004 (4) |
| C24 | 0.045 (4) | 0.065 (4) | 0.054 (5) | −0.003 (3) | 0.020 (3) | −0.007 (4) |
| C25 | 0.046 (4) | 0.068 (5) | 0.071 (5) | 0.009 (4) | 0.019 (4) | 0.007 (4) |
| C26 | 0.051 (4) | 0.056 (4) | 0.053 (5) | −0.003 (3) | 0.023 (3) | 0.006 (4) |
| C31 | 0.044 (4) | 0.043 (3) | 0.044 (4) | 0.003 (3) | 0.020 (3) | 0.002 (3) |
| C32 | 0.056 (5) | 0.053 (4) | 0.050 (5) | −0.004 (3) | 0.031 (4) | 0.000 (3) |
| C33 | 0.056 (4) | 0.047 (4) | 0.059 (5) | 0.000 (3) | 0.022 (4) | −0.002 (4) |
| C34 | 0.062 (5) | 0.053 (4) | 0.054 (5) | −0.014 (3) | 0.024 (4) | −0.002 (4) |
| C35 | 0.050 (5) | 0.061 (5) | 0.069 (6) | −0.009 (3) | 0.028 (4) | 0.001 (4) |
| C36 | 0.050 (4) | 0.050 (4) | 0.054 (5) | −0.002 (3) | 0.024 (3) | 0.001 (4) |
| C41 | 0.040 (4) | 0.051 (4) | 0.049 (5) | 0.001 (3) | 0.023 (3) | −0.004 (3) |
| C42 | 0.044 (4) | 0.049 (4) | 0.050 (4) | −0.003 (3) | 0.019 (3) | −0.005 (4) |
| C43 | 0.045 (4) | 0.060 (5) | 0.042 (4) | 0.000 (3) | 0.017 (3) | −0.007 (3) |
| C44 | 0.049 (4) | 0.060 (4) | 0.045 (4) | 0.003 (3) | 0.021 (3) | 0.009 (4) |
| C45 | 0.047 (4) | 0.047 (4) | 0.052 (4) | −0.003 (3) | 0.026 (3) | 0.000 (3) |
| C51 | 0.048 (4) | 0.072 (5) | 0.092 (7) | 0.004 (4) | 0.032 (5) | −0.020 (5) |
| C52 | 0.047 (4) | 0.090 (6) | 0.081 (7) | 0.017 (4) | 0.021 (4) | 0.000 (6) |
| C53 | 0.039 (5) | 0.094 (7) | 0.072 (7) | −0.013 (4) | 0.020 (4) | −0.030 (5) |
| C54 | 0.053 (5) | 0.075 (6) | 0.082 (7) | −0.009 (4) | 0.039 (5) | −0.008 (5) |
| C55 | 0.054 (5) | 0.082 (5) | 0.055 (5) | −0.006 (4) | 0.032 (4) | −0.006 (5) |
| C1A | 0.19 (7) | 0.101 (13) | 0.34 (11) | 0.00 (4) | 0.20 (8) | 0.00 (4) |
| Cl1A | 0.070 (6) | 0.100 (7) | 0.098 (13) | −0.005 (6) | 0.016 (10) | 0.011 (8) |
| Cl2A | 0.077 (5) | 0.108 (9) | 0.147 (17) | 0.009 (5) | 0.020 (7) | 0.040 (12) |
| C1B | 0.051 (10) | 0.097 (12) | 0.13 (2) | 0.014 (8) | 0.029 (11) | 0.029 (13) |
| Cl1B | 0.076 (6) | 0.079 (3) | 0.084 (7) | 0.002 (3) | 0.026 (6) | −0.008 (4) |
| Cl2B | 0.098 (5) | 0.090 (4) | 0.149 (11) | 0.016 (4) | 0.068 (7) | 0.034 (5) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| Au1—P1 | 2.2609 (18) | C32—H32 | 0.95 |
| Au1—S1 | 2.3084 (19) | C33—C34 | 1.379 (11) |
| S1—P2 | 2.050 (3) | C33—H33 | 0.95 |
| P1—C31 | 1.819 (7) | C34—C35 | 1.372 (12) |
| P1—C21 | 1.821 (6) | C34—H34 | 0.95 |
| P1—C11 | 1.822 (7) | C35—C36 | 1.377 (11) |

| | | | |
|---|---|---|---|
| P2—O1 | 1.503 (6) | C35—H35 | 0.95 |
| P2—O2 | 1.541 (5) | C36—H36 | 0.95 |
| P2—C41 | 1.786 (9) | C41—C45 | 1.416 (10) |
| O2—H1 | 1.11 (11) | C41—C42 | 1.424 (11) |
| C11—C16 | 1.384 (10) | C42—C43 | 1.428 (11) |
| C11—C12 | 1.387 (10) | C42—H42 | 0.95 |
| C12—C13 | 1.379 (11) | C43—C44 | 1.416 (11) |
| C12—H12 | 0.95 | C43—H43 | 0.95 |
| C13—C14 | 1.383 (12) | C44—C45 | 1.424 (11) |
| C13—H13 | 0.95 | C44—H44 | 0.95 |
| C14—C15 | 1.402 (13) | C45—H45 | 0.95 |
| C14—H14 | 0.95 | C51—C55 | 1.403 (13) |
| C15—C16 | 1.361 (12) | C51—C52 | 1.446 (13) |
| C15—H15 | 0.95 | C51—H51 | 0.95 |
| C16—H16 | 0.95 | C52—C53 | 1.405 (14) |
| C21—C22 | 1.372 (10) | C52—H52 | 0.95 |
| C21—C26 | 1.402 (10) | C53—C54 | 1.382 (15) |
| C22—C23 | 1.375 (12) | C53—H53 | 0.95 |
| C22—H22 | 0.95 | C54—C55 | 1.406 (13) |
| C23—C24 | 1.378 (12) | C54—H54 | 0.95 |
| C23—H23 | 0.95 | C55—H55 | 0.95 |
| C24—C25 | 1.381 (11) | C1A—Cl2A | 1.642 (17) |
| C24—H24 | 0.95 | C1A—Cl1A | 1.645 (17) |
| C25—C26 | 1.383 (10) | C1A—H1A1 | 0.99 |
| C25—H25 | 0.95 | C1A—H1A2 | 0.99 |
| C26—H26 | 0.95 | C1B—Cl2B | 1.629 (14) |
| C31—C32 | 1.381 (10) | C1B—Cl1B | 1.656 (14) |
| C31—C36 | 1.396 (10) | C1B—H1B1 | 0.99 |
| C32—C33 | 1.399 (11) | C1B—H1B2 | 0.99 |
| P1—Au1—S1 | 176.81 (6) | C33—C32—H32 | 120.1 |
| P2—S1—Au1 | 99.14 (9) | C34—C33—C32 | 119.7 (8) |
| C31—P1—C21 | 106.3 (3) | C34—C33—H33 | 120.2 |
| C31—P1—C11 | 104.6 (3) | C32—C33—H33 | 120.2 |
| C21—P1—C11 | 106.1 (3) | C35—C34—C33 | 120.7 (7) |
| C31—P1—Au1 | 113.8 (2) | C35—C34—H34 | 119.6 |
| C21—P1—Au1 | 113.1 (2) | C33—C34—H34 | 119.6 |
| C11—P1—Au1 | 112.3 (2) | C34—C35—C36 | 119.8 (8) |
| O1—P2—O2 | 107.6 (3) | C34—C35—H35 | 120.1 |
| O1—P2—C41 | 110.8 (3) | C36—C35—H35 | 120.1 |
| O2—P2—C41 | 110.1 (3) | C35—C36—C31 | 120.5 (7) |
| O1—P2—S1 | 113.9 (3) | C35—C36—H36 | 119.7 |
| O2—P2—S1 | 106.0 (2) | C31—C36—H36 | 119.7 |
| C41—P2—S1 | 108.3 (3) | C45—C41—C42 | 107.2 (7) |
| P2—O2—H1 | 115 (5) | C45—C41—P2 | 126.0 (5) |
| C16—C11—C12 | 118.7 (7) | C42—C41—P2 | 126.8 (6) |
| C16—C11—P1 | 118.3 (6) | C41—C42—C43 | 108.6 (7) |
| C12—C11—P1 | 122.8 (5) | C41—C42—H42 | 125.7 |
| C13—C12—C11 | 119.9 (7) | C43—C42—H42 | 125.7 |
| C13—C12—H12 | 120.1 | C44—C43—C42 | 107.5 (7) |

| | | | |
|---|---|---|---|
| C11—C12—H12 | 120.1 | C44—C43—H43 | 126.3 |
| C12—C13—C14 | 121.0 (8) | C42—C43—H43 | 126.3 |
| C12—C13—H13 | 119.5 | C43—C44—C45 | 108.1 (7) |
| C14—C13—H13 | 119.5 | C43—C44—H44 | 125.9 |
| C13—C14—C15 | 119.0 (8) | C45—C44—H44 | 125.9 |
| C13—C14—H14 | 120.5 | C41—C45—C44 | 108.6 (6) |
| C15—C14—H14 | 120.5 | C41—C45—H45 | 125.7 |
| C16—C15—C14 | 119.2 (8) | C44—C45—H45 | 125.7 |
| C16—C15—H15 | 120.4 | C55—C51—C52 | 106.9 (8) |
| C14—C15—H15 | 120.4 | C55—C51—H51 | 126.5 |
| C15—C16—C11 | 122.1 (8) | C52—C51—H51 | 126.5 |
| C15—C16—H16 | 119 | C53—C52—C51 | 106.9 (10) |
| C11—C16—H16 | 119 | C53—C52—H52 | 126.5 |
| C22—C21—C26 | 118.9 (6) | C51—C52—H52 | 126.5 |
| C22—C21—P1 | 120.9 (6) | C54—C53—C52 | 109.1 (9) |
| C26—C21—P1 | 119.9 (5) | C54—C53—H53 | 125.5 |
| C21—C22—C23 | 121.7 (8) | C52—C53—H53 | 125.5 |
| C21—C22—H22 | 119.2 | C53—C54—C55 | 108.6 (8) |
| C23—C22—H22 | 119.2 | C53—C54—H54 | 125.7 |
| C22—C23—C24 | 118.9 (7) | C55—C54—H54 | 125.7 |
| C22—C23—H23 | 120.6 | C51—C55—C54 | 108.5 (9) |
| C24—C23—H23 | 120.6 | C51—C55—H55 | 125.8 |
| C23—C24—C25 | 121.1 (7) | C54—C55—H55 | 125.8 |
| C23—C24—H24 | 119.4 | Cl2A—C1A—Cl1A | 128 (3) |
| C25—C24—H24 | 119.4 | Cl2A—C1A—H1A1 | 105.3 |
| C24—C25—C26 | 119.4 (8) | Cl1A—C1A—H1A1 | 105.3 |
| C24—C25—H25 | 120.3 | Cl2A—C1A—H1A2 | 105.3 |
| C26—C25—H25 | 120.3 | Cl1A—C1A—H1A2 | 105.3 |
| C25—C26—C21 | 120.0 (7) | H1A1—C1A—H1A2 | 106 |
| C25—C26—H26 | 120 | Cl2B—C1B—Cl1B | 118.3 (16) |
| C21—C26—H26 | 120 | Cl2B—C1B—H1B1 | 107.7 |
| C32—C31—C36 | 119.4 (7) | Cl1B—C1B—H1B1 | 107.7 |
| C32—C31—P1 | 121.9 (5) | Cl2B—C1B—H1B2 | 107.7 |
| C36—C31—P1 | 118.5 (5) | Cl1B—C1B—H1B2 | 107.7 |
| C31—C32—C33 | 119.8 (7) | H1B1—C1B—H1B2 | 107.1 |
| C31—C32—H32 | 120.1 | | |
| Au1—S1—P2—O1 | 76.2 (3) | C11—P1—C31—C32 | 68.3 (7) |
| Au1—S1—P2—O2 | −165.7 (2) | Au1—P1—C31—C32 | −168.8 (6) |
| Au1—S1—P2—C41 | −47.6 (2) | C21—P1—C31—C36 | 142.1 (6) |
| C31—P1—C11—C16 | 97.8 (6) | C11—P1—C31—C36 | −105.9 (6) |
| C21—P1—C11—C16 | −150.0 (6) | Au1—P1—C31—C36 | 17.0 (7) |
| Au1—P1—C11—C16 | −26.0 (6) | C36—C31—C32—C33 | −2.8 (12) |
| C31—P1—C11—C12 | −76.7 (7) | P1—C31—C32—C33 | −176.9 (6) |
| C21—P1—C11—C12 | 35.4 (7) | C31—C32—C33—C34 | 2.4 (13) |
| Au1—P1—C11—C12 | 159.5 (5) | C32—C33—C34—C35 | −1.7 (13) |
| C16—C11—C12—C13 | 0.2 (11) | C33—C34—C35—C36 | 1.3 (14) |
| P1—C11—C12—C13 | 174.7 (6) | C34—C35—C36—C31 | −1.6 (13) |
| C11—C12—C13—C14 | 0.2 (12) | C32—C31—C36—C35 | 2.4 (12) |
| C12—C13—C14—C15 | −1.9 (12) | P1—C31—C36—C35 | 176.8 (7) |

| | | | |
|---|---|---|---|
| C13—C14—C15—C16 | 3.2 (12) | O1—P2—C41—C45 | 170.2 (6) |
| C14—C15—C16—C11 | −2.9 (13) | O2—P2—C41—C45 | 51.3 (7) |
| C12—C11—C16—C15 | 1.2 (11) | S1—P2—C41—C45 | −64.2 (7) |
| P1—C11—C16—C15 | −173.5 (7) | O1—P2—C41—C42 | −9.0 (8) |
| C31—P1—C21—C22 | 150.7 (6) | O2—P2—C41—C42 | −127.9 (6) |
| C11—P1—C21—C22 | 39.8 (7) | S1—P2—C41—C42 | 116.6 (6) |
| Au1—P1—C21—C22 | −83.8 (7) | C45—C41—C42—C43 | −0.2 (8) |
| C31—P1—C21—C26 | −35.8 (7) | P2—C41—C42—C43 | 179.1 (6) |
| C11—P1—C21—C26 | −146.7 (6) | C41—C42—C43—C44 | −0.1 (8) |
| Au1—P1—C21—C26 | 89.7 (6) | C42—C43—C44—C45 | 0.3 (8) |
| C26—C21—C22—C23 | 2.1 (12) | C42—C41—C45—C44 | 0.4 (8) |
| P1—C21—C22—C23 | 175.7 (7) | P2—C41—C45—C44 | −178.9 (5) |
| C21—C22—C23—C24 | −1.4 (13) | C43—C44—C45—C41 | −0.4 (8) |
| C22—C23—C24—C25 | −0.7 (13) | C55—C51—C52—C53 | 2.3 (10) |
| C23—C24—C25—C26 | 2.0 (13) | C51—C52—C53—C54 | −1.9 (10) |
| C24—C25—C26—C21 | −1.3 (12) | C52—C53—C54—C55 | 0.7 (10) |
| C22—C21—C26—C25 | −0.8 (11) | C52—C51—C55—C54 | −1.9 (9) |
| P1—C21—C26—C25 | −174.4 (6) | C53—C54—C55—C51 | 0.7 (10) |
| C21—P1—C31—C32 | −43.7 (8) | | |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O2—H1···O1$^{i}$ | 1.11 (11) | 1.33 (11) | 2.432 (7) | 173 (9) |

Symmetry codes: (i) −*x*, *y*+1/2, −*z*+3/2.

**Fig. 1**

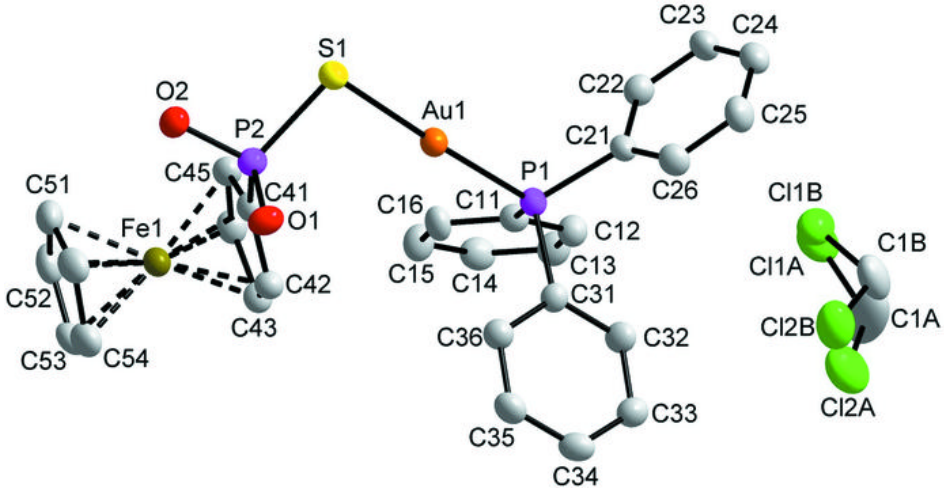
C23
C24
S1
C22
O2
Au1
C25
P2
C21
C45
P1
C26
C51
C41
C16
C11
Cl1B
Fe1
O1
C12
C1B
C15
C14
C13
Cl1A
C52
C42
C31
C1A
Cl2B
C43
C36
C32
C53
C54
C33
C35
Cl2A
C34

**Fig. 2**