Crystal structure of [μ_2-1,1′-bis(diphenylphosphanyl)ferrocene-κ^2P:$P′$]bis[(pyrrolidine-1-carbodithioato-κS)gold(I)]

Yee Seng Tan[a] and Edward R. T. Tiekink[a,b]*

[a]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia, and [b]Centre for Chemical Crystallography, Faculty of Science and Technology, Sunway University, 47500 Bandar Sunway, Selangor Darul Ehsan, Malaysia. *Correspondence e-mail: edward.tiekink@gmail.com

The asymmetric unit of the title compound, {($C_{34}H_{28}FeP_2$)[Au($C_5H_8NS_2$)]$_2$}, comprises half a molecule, with the full molecule being generated by the application of a centre of inversion. The independent Au^I atom is coordinated by thiolate S and phosphane P atoms that define an approximate linear geometry [S—Au—P = 169.35 (3)°]. The deviation from the ideal linear is traced to the close approach of the (intramolecular) non-coordinating thione S atom [Au$\cdots$S = 3.1538 (8) Å]. Supramolecular layers parallel to (100) feature in the crystal packing, being sustained by phenyl–thione C—H$\cdots$S interactions, with the non-coordinating thione S atom in the role of a dual acceptor. Layers stack with no specific interactions between them.
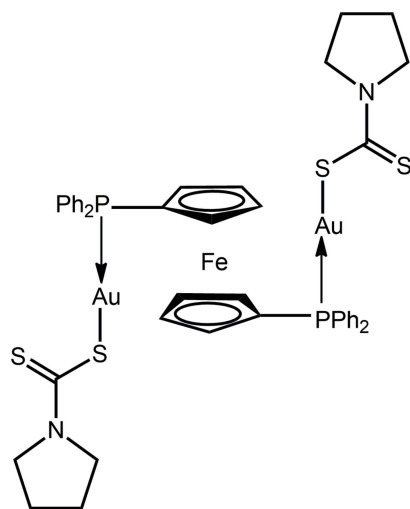
1. Chemical context

Investigations into the potential anti-cancer activity of phosphanegold(I) dithiocarbamates, R_3PAu(S_2CN$R′_2$), date back over a decade (de Vos *et al.*, 2004; Vergara *et al.*, 2007; Jamaludin *et al.*, 2013). These investigations are complemented by the recently reported impressive anti-microbial activity for this class of compound (Sim *et al.*, 2014) whereby R_3PAu[S_2CN(^{i}Pr)CH_2CH_2OH], R = Ph and Cy, exhibited specific activity against Gram-positive bacteria while the R = Et derivative displayed broad-range activity against both Gram-positive and Gram-negative bacteria. Motivated by observations that 1,1′-bis(diphenylphosphanyl)ferrocene (dppf) derivatives also possess biological activity (Ornelas, 2011; Braga & Silva, 2013), it was thought of interest to couple dppf with Au^I dithiocarbamates. This led to the isolation of the broadly insoluble title compound, dppf{Au[S_2CN(CH_2)$_4$]}$_2$, (I), which was subjected to a crystal structure determination. The results of this study are reported herein along with a comparison to related species.

2. Structural commentary

The Fe^{II} atom in dppf{Au[S_2CN(CH_2)$_4$]}$_2$, (I), is located on a centre of inversion, Fig. 1. The Au^I central atom exists in the anticipated linear geometry defined by thiolate-S and phosphane-P atoms. The Au—S1 bond length is considerably longer than the Au—P1 bond, *i.e.* 2.3378 (8) *cf.* 2.2580 (8) Å. The dithiocarbamate ligand is orientated to place the S2 atom in close proximity to the Au^I atom. However, the resulting intramolecular Au$\cdots$S2 interaction is long at 3.1538 (8) Å, consistent with a monodentate mode of coordination for the dithiocarbamate ligand. The pattern of C1—S1, S2 bond
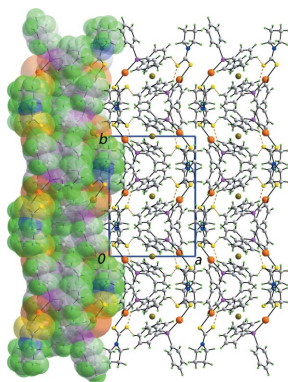
lengths supports this conclusion in that the strongly bound S1 atom forms a longer, *i.e.* weaker, C1—S1 bond [1.757 (3) Å] *cf.* with C1—S2 of 1.689 (3) Å. Nevertheless, the close approach of the S2 atom to the Au^I central atom is correlated with the deviation from the ideal linear geometry, *i.e.* S1—Au—P1 is 169.35 (3)°.

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

D—H···A	D—H	H···A	D···A	D—H···A
C13—H13···S2^i	0.95	2.86	3.680 (3)	144
C20—H20···S2^{ii}	0.95	2.84	3.628 (3)	141

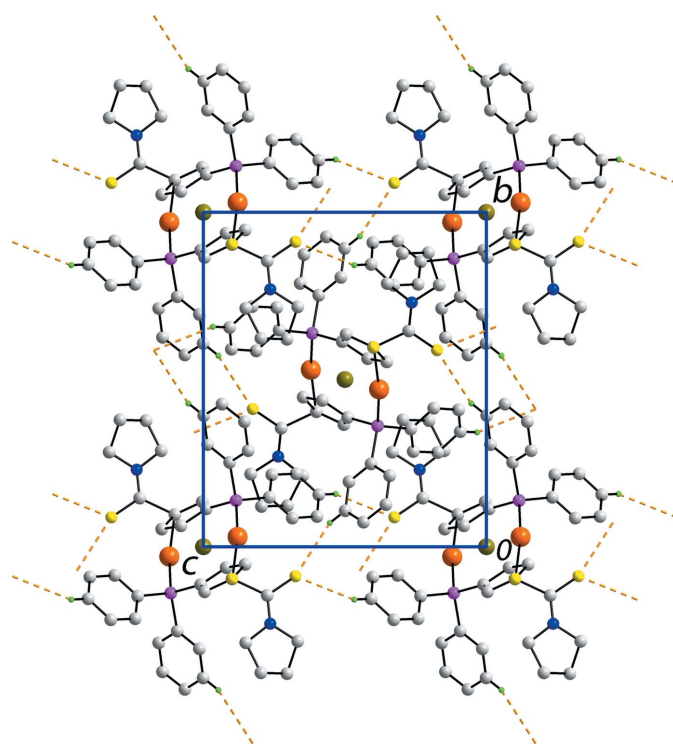Symmetry codes: (i) $x, -y + \frac{1}{2}, z + \frac{1}{2}$; (ii) $-x + 1, -y, -z + 1$.

symmetry operation (i): $1 - x, -y, 2 - z$. Again, from symmetry, the Cp rings have a staggered relationship.

3. Supramolecular features

In the crystal packing, the most prominent interactions are of the type C—H···S. Data for the phenyl-C—H···S(thione) interactions are collected in Table 1. These interactions, involving the dual acceptor S2 atom, serve to assemble molecules into supramolecular layers in the *bc* plane, Fig. 2. The thickness of each layer corresponds to the length of the *a* axis, *i.e.* 10.9635 (4) Å, and the layers stack along this axis with no directional interactions between them, Fig. 3.

4. Database survey

It has been approximately 40 years since the first report of a structure related to (I), *i.e.* Ph$_3$PAu(S$_2$CNEt$_2$), by Wijnhoven *et al.* (1972). This serves as the archetype for approximately 20 other neutral phosphanegold(I) dithiocarbamate structures in

Similar features are noted in related structures as outlined below in the *Database survey*. The pyrrolidine ring is twisted about the C2—C3 bond. Owing to being located on a centre of inversion, the Fe^{II} atom is equidistant from the ring centroids of the Cp rings [Fe···Cg, Cg^i = 1.6566 (13) Å] and the Cg—Fe—Cg^i angle is constrained by symmetry to be 180°;
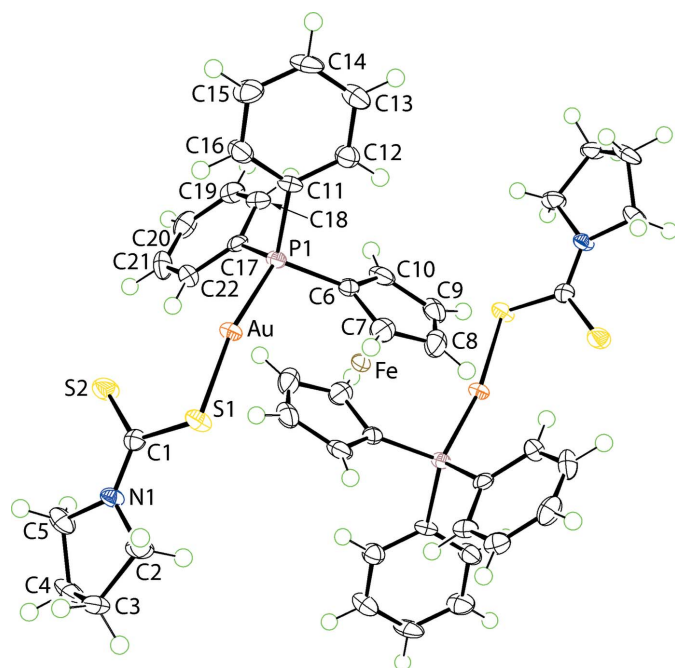
Figure 1
The molecular structure of (I), showing the atom-labelling scheme and displacement ellipsoids at the 70% probability level. Unlabelled atoms are related by the symmetry operation $(-x + 1, -y, -z + 2)$.

Figure 2
A view of the supramolecular layer in the *bc* plane sustained by phenyl–thione C—H···S interactions, shown as orange dashed lines. H atoms not involved in intermolecular interactions have been omitted for clarity.

Table 2
Geometric details (Å, °) for (I) and related literature structures.

| Structure	Au—S	Au—P	S—Au—P	Au···S2	CSD Refcode[a]	Reference
(I)	2.3378 (8)	2.2580 (8)	169.35 (3)	3.1538 (8)	–	This work
(II)	2.3333 (11)	2.2447 (10)	173.82 (4)	3.0440 (10)	AYIYAI	Ho & Tiekink (2004)
(III)	2.3256 (16)	2.2547 (15)	176.55 (5)	3.1067 (17)	XUMRIG	Ho & Tiekink (2002)
(IV)	2.3365 (11)	2.2495 (10)	171.98 (3)	3.0472 (10)	GICZAV	Štěpnička & Císařová (2012)
	2.3559 (8)	2.2459 (8)	172.12 (3)	2.9178 (12)		

Reference: (a) Groom & Allen (2014).

the crystallographic literature (Groom & Allen, 2014), each having a more or less linear P—Au—S arrangement. There are two structures containing the pyrrolinedithiocarbamate ligand, as in (I), but with phosphane ligands Ph₃P [(II); Ho & Tiekink, 2004] and Cy₃P [(III); Ho & Tiekink, 2002]. From the data collated for (I)–(III) in Table 2, it is evident that the basic structural features in all three compounds are similar. There is also a closely related dppf-type structure whereby a methylene bridge has been inserted between one P atom and the Cp ring, *i.e.* (Ph₂PCH₂C₅H₄FeC₅H₄PPh₂)[Au(S₂CNEt₂)]₂·2CHCl₃, [(IV); Štěpnička & Císařová, 2012]. In this analogue of (I), the Fe^{II} atom is in a general position. While the Au₂P₂ entity in

(IV) remains approximately co-planar, as is crystallographically imposed in (I), *i.e.* the Au—P···P—Au pseudo torsion angle is 161.82 (5)°, the Au^{I} atoms lie approximately to the same side of the molecule as opposed to the strictly *anti* conformation found in (I). As seen in Table 2, the selected geometric parameters in (I) and (IV) are comparable. Despite having the shortest intramolecular Au···S2 contact in (IV), the deviation of the S—Au—P angle from linearity is not the greatest in this structure.

5. Synthesis and crystallization

Two solutions were prepared. Firstly, a solution of the sodium salt of pyrrolidine dithiocarbamate (Aldrich, 1.6 mmol) was prepared by dissolving this (0.2628 g) in methanol (25 ml). A second solution containing [1,1′-bis(diphenylphosphanyl)-
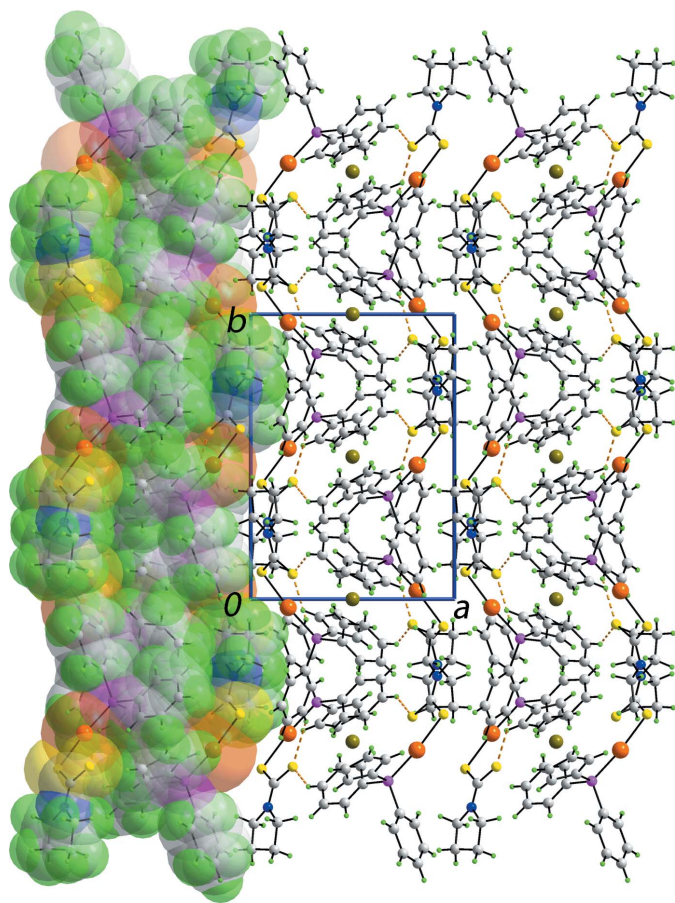
Figure 3
Unit-cell contents shown in projection down the *c* axis, showing the stacking of supramolecular layers. The phenyl–thione C—H···S interactions are shown as orange dashed lines. One layer is shown in space-filling mode.

Table 3
Experimental details.

Crystal data	
Chemical formula	[Au₂Fe(C₅H₈NS₂)₂(C₃₄H₂₈P₂)]
M_r	1240.77
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	100
a, b, c (Å)	10.9635 (4), 14.9720 (5), 13.0087 (4)
β (°)	102.977 (3)
V (Å³)	2080.78 (12)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	7.69
Crystal size (mm)	0.20 × 0.20 × 0.20
Data collection	
Diffractometer	Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction	Multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min}, T_{max}	0.294, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	24384, 4777, 4363
R_{int}	0.048
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.650
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.022, 0.051, 1.06
No. of reflections	4777
No. of parameters	250
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	1.57, −1.11

Computer programs: *CrysAlis PRO* (Agilent, 2014), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012), *DIAMOND* (Brandenburg, 2006) and *publCIF* (Westrip, 2010).

ferrocene]bis[chloridogold(I)] (synthesized by the reduction of $KAuCl_4$ by Na_2SO_3 followed by the addition of a stoichiometric amount of 1,1′-bis(diphenylphosphanyl)ferrocene; 0.8154 g, 0.8 mmol) was prepared by dissolution in dichloromethane (75 ml). The solution containing the dithiocarbamate salt was added to the gold precursor solution. The resulting mixture was stirred for 3 h at room condition and then filtered. After a week of slow evaporation in a refrigerator, some dark-yellow blocks appeared that were characterized crystallographically. M. p. 378–379 K. IR (cm^{-1}): 1435 s ν(C—N); 1152 m, 996 m ν(C—S).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. Carbon-bound H-atoms were placed in calculated positions (C—H = 0.95–0.99 Å) and were included in the refinement in the riding-model approximation, with U_{iso}(H) set to $1.2U_{eq}$(C). The maximum and minimum residual electron density peaks of 1.57 and 1.11 e Å^{-3}, respectively, were located 0.92 and 0.79 Å from the Au atom.

Acknowledgements

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Crystal structure of [*μ*₂-1,1′-bis(diphenylphosphanyl)ferrocene-*κ*²*P*:*P*′]bis-[(pyrrolidine-1-carbodithioato-*κS*)gold(I)]

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Computing details

Data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO* (Agilent, 2014); data reduction: *CrysAlis PRO* (Agilent, 2014); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

[*μ*₂-1,1′-Bis(diphenylphosphanyl)ferrocene-*κ*²*P*:*P*′]bis[(pyrrolidine-1-carbodithioato-*κS*)gold(I)]

Crystal data

$[Au_2Fe(C_5H_8NS_2)_2(C_{34}H_{28}P_2)]$	$F(000) = 1200$
$M_r = 1240.77$	$D_x = 1.980$ Mg m⁻³
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 10.9635$ (4) Å	Cell parameters from 10685 reflections
$b = 14.9720$ (5) Å	$\theta = 3.5$–30.2°
$c = 13.0087$ (4) Å	$\mu = 7.69$ mm⁻¹
$\beta = 102.977$ (3)°	$T = 100$ K
$V = 2080.78$ (12) Å³	Block, dark-yellow
$Z = 2$	$0.20 \times 0.20 \times 0.20$ mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector	$T_{min} = 0.294$, $T_{max} = 1.000$
	24384 measured reflections
Radiation source: SuperNova (Mo) X-ray Source	4777 independent reflections
	4363 reflections with $I > 2\sigma(I)$
Mirror monochromator	$R_{int} = 0.048$
Detector resolution: 10.4041 pixels mm⁻¹	$\theta_{max} = 27.5°$, $\theta_{min} = 3.1°$
ω scan	$h = -14 \rightarrow 14$
Absorption correction: multi-scan	$k = -19 \rightarrow 19$
(*CrysAlis PRO*; Agilent, 2014)	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.022$	$w = 1/[\sigma^2(F_o^2) + (0.0208P)^2 + 0.4001P]$
$wR(F^2) = 0.051$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} = 0.002$
4777 reflections	$\Delta\rho_{max} = 1.57$ e Å⁻³
250 parameters	$\Delta\rho_{min} = -1.11$ e Å⁻³
0 restraints	

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
Au	0.81601 (2)	0.02934 (2)	0.87927 (2)	0.01301 (5)
Fe	0.5000	0.0000	1.0000	0.01445 (13)
S1	0.94626 (7)	−0.09602 (5)	0.89801 (6)	0.01689 (16)
S2	0.79416 (8)	−0.08799 (5)	0.67459 (6)	0.02172 (18)
P1	0.67514 (7)	0.13829 (5)	0.88458 (6)	0.01219 (15)
N1	0.9156 (2)	−0.23029 (16)	0.76400 (18)	0.0145 (5)
C1	0.8858 (3)	−0.1454 (2)	0.7746 (2)	0.0139 (6)
C2	0.9848 (3)	−0.2871 (2)	0.8506 (2)	0.0203 (7)
H2A	0.9382	−0.2934	0.9072	0.024*
H2B	1.0686	−0.2619	0.8812	0.024*
C3	0.9954 (3)	−0.3769 (2)	0.7971 (3)	0.0232 (7)
H3A	1.0746	−0.3806	0.7727	0.028*
H3B	0.9924	−0.4270	0.8461	0.028*
C4	0.8842 (3)	−0.3795 (2)	0.7050 (3)	0.0245 (7)
H4A	0.8084	−0.4004	0.7273	0.029*
H4B	0.9001	−0.4194	0.6488	0.029*
C5	0.8690 (3)	−0.2827 (2)	0.6669 (2)	0.0206 (7)
H5A	0.9193	−0.2708	0.6140	0.025*
H5B	0.7802	−0.2688	0.6356	0.025*
C6	0.5997 (3)	0.1135 (2)	0.9904 (2)	0.0141 (6)
C7	0.6606 (3)	0.0625 (2)	1.0801 (2)	0.0211 (7)
H7	0.7432	0.0391	1.0924	0.025*
C8	0.5766 (4)	0.0528 (2)	1.1475 (2)	0.0268 (8)
H8	0.5932	0.0220	1.2130	0.032*
C9	0.4649 (3)	0.0963 (2)	1.1010 (3)	0.0254 (8)
H9	0.3926	0.0996	1.1296	0.030*
C10	0.4771 (3)	0.1346 (2)	1.0040 (2)	0.0202 (7)
H10	0.4152	0.1682	0.9568	0.024*
C11	0.7323 (3)	0.25218 (19)	0.9075 (2)	0.0131 (6)
C12	0.7027 (3)	0.3055 (2)	0.9856 (2)	0.0187 (7)
H12	0.6518	0.2826	1.0298	0.022*
C13	0.7478 (3)	0.3923 (2)	0.9990 (3)	0.0231 (7)
H13	0.7283	0.4286	1.0531	0.028*
C14	0.8207 (3)	0.4265 (2)	0.9344 (3)	0.0241 (7)
H14	0.8513	0.4860	0.9442	0.029*
C15	0.8494 (3)	0.3739 (2)	0.8554 (3)	0.0245 (7)
H15	0.8993	0.3972	0.8107	0.029*
C16	0.8048 (3)	0.2874 (2)	0.8419 (3)	0.0205 (7)
H16	0.8238	0.2515	0.7874	0.025*

C17	0.5506 (3)	0.1478 (2)	0.7674 (2)	0.0133 (6)
C18	0.4616 (3)	0.2152 (2)	0.7586 (2)	0.0170 (6)
H18	0.4699	0.2595	0.8120	0.020*
C19	0.3606 (3)	0.2183 (2)	0.6723 (2)	0.0191 (7)
H19	0.2980	0.2629	0.6680	0.023*
C20	0.3520 (3)	0.1557 (2)	0.5924 (2)	0.0214 (7)
H20	0.2837	0.1579	0.5329	0.026*
C21	0.4414 (3)	0.0906 (2)	0.5986 (2)	0.0222 (7)
H21	0.4355	0.0488	0.5428	0.027*
C22	0.5405 (3)	0.0857 (2)	0.6862 (2)	0.0175 (6)
H22	0.6015	0.0399	0.6907	0.021*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Au	0.01259 (7)	0.00937 (7)	0.01715 (7)	0.00067 (4)	0.00346 (5)	−0.00134 (4)
Fe	0.0201 (3)	0.0098 (3)	0.0150 (3)	−0.0028 (3)	0.0070 (2)	−0.0025 (2)
S1	0.0171 (4)	0.0131 (4)	0.0180 (4)	0.0029 (3)	−0.0011 (3)	−0.0037 (3)
S2	0.0292 (5)	0.0162 (4)	0.0168 (4)	0.0070 (3)	−0.0012 (3)	0.0001 (3)
P1	0.0129 (4)	0.0093 (4)	0.0148 (3)	0.0002 (3)	0.0038 (3)	−0.0006 (3)
N1	0.0164 (13)	0.0101 (13)	0.0159 (12)	−0.0008 (10)	0.0011 (10)	−0.0027 (10)
C1	0.0117 (15)	0.0142 (16)	0.0160 (14)	−0.0017 (12)	0.0040 (11)	0.0003 (12)
C2	0.0243 (18)	0.0133 (16)	0.0220 (16)	0.0060 (14)	0.0025 (13)	0.0023 (13)
C3	0.032 (2)	0.0107 (16)	0.0297 (17)	0.0047 (14)	0.0126 (14)	0.0027 (13)
C4	0.0259 (19)	0.0113 (16)	0.0380 (19)	−0.0026 (14)	0.0111 (15)	−0.0110 (14)
C5	0.0192 (17)	0.0177 (17)	0.0247 (16)	−0.0033 (14)	0.0043 (13)	−0.0101 (13)
C6	0.0175 (16)	0.0097 (15)	0.0159 (14)	−0.0034 (12)	0.0056 (12)	−0.0016 (11)
C7	0.0257 (19)	0.0187 (17)	0.0166 (15)	−0.0066 (14)	−0.0001 (13)	−0.0019 (13)
C8	0.041 (2)	0.0240 (18)	0.0164 (16)	−0.0154 (17)	0.0084 (15)	−0.0058 (14)
C9	0.035 (2)	0.0177 (18)	0.0297 (17)	−0.0134 (15)	0.0205 (15)	−0.0107 (14)
C10	0.0226 (17)	0.0105 (16)	0.0306 (17)	−0.0002 (13)	0.0124 (14)	−0.0038 (13)
C11	0.0111 (15)	0.0078 (14)	0.0189 (14)	0.0008 (12)	0.0002 (11)	−0.0008 (11)
C12	0.0199 (17)	0.0158 (17)	0.0211 (15)	−0.0014 (13)	0.0061 (13)	−0.0016 (12)
C13	0.0233 (18)	0.0165 (17)	0.0302 (17)	0.0005 (14)	0.0076 (14)	−0.0072 (14)
C14	0.0231 (18)	0.0093 (16)	0.041 (2)	−0.0039 (14)	0.0095 (15)	−0.0031 (14)
C15	0.0226 (18)	0.0179 (18)	0.0370 (19)	−0.0027 (14)	0.0153 (15)	0.0029 (14)
C16	0.0222 (18)	0.0153 (17)	0.0259 (16)	0.0003 (14)	0.0095 (13)	−0.0039 (13)
C17	0.0150 (15)	0.0108 (15)	0.0151 (13)	−0.0017 (12)	0.0054 (11)	0.0033 (11)
C18	0.0177 (16)	0.0116 (16)	0.0217 (15)	−0.0010 (12)	0.0045 (12)	0.0021 (12)
C19	0.0177 (17)	0.0170 (17)	0.0230 (16)	0.0002 (13)	0.0055 (13)	0.0038 (13)
C20	0.0174 (17)	0.0299 (19)	0.0155 (14)	−0.0050 (15)	0.0007 (12)	0.0041 (13)
C21	0.0252 (18)	0.0285 (19)	0.0137 (14)	−0.0022 (15)	0.0058 (13)	−0.0049 (13)
C22	0.0206 (17)	0.0159 (16)	0.0175 (14)	0.0019 (13)	0.0074 (12)	−0.0018 (12)

Geometric parameters (Å, º)

Au—P1	2.2580 (8)	C6—C7	1.430 (4)
Au—S1	2.3378 (8)	C6—C10	1.430 (4)

Fe—C10[i]	2.033 (3)	C7—C8	1.414 (5)
Fe—C10	2.033 (3)	C7—H7	0.9500
Fe—C6[i]	2.039 (3)	C8—C9	1.399 (5)
Fe—C6	2.039 (3)	C8—H8	0.9500
Fe—C9	2.044 (3)	C9—C10	1.420 (4)
Fe—C9[i]	2.044 (3)	C9—H9	0.9500
Fe—C7[i]	2.059 (3)	C10—H10	0.9500
Fe—C7	2.059 (3)	C11—C12	1.386 (4)
Fe—C8	2.071 (3)	C11—C16	1.395 (4)
Fe—C8[i]	2.071 (3)	C12—C13	1.388 (4)
S1—C1	1.757 (3)	C12—H12	0.9500
S2—C1	1.689 (3)	C13—C14	1.382 (5)
P1—C6	1.796 (3)	C13—H13	0.9500
P1—C17	1.809 (3)	C14—C15	1.386 (5)
P1—C11	1.818 (3)	C14—H14	0.9500
N1—C1	1.327 (4)	C15—C16	1.381 (5)
N1—C2	1.478 (4)	C15—H15	0.9500
N1—C5	1.478 (4)	C16—H16	0.9500
C2—C3	1.531 (4)	C17—C18	1.390 (4)
C2—H2A	0.9900	C17—C22	1.394 (4)
C2—H2B	0.9900	C18—C19	1.389 (4)
C3—C4	1.506 (5)	C18—H18	0.9500
C3—H3A	0.9900	C19—C20	1.387 (4)
C3—H3B	0.9900	C19—H19	0.9500
C4—C5	1.528 (5)	C20—C21	1.371 (5)
C4—H4A	0.9900	C20—H20	0.9500
C4—H4B	0.9900	C21—C22	1.388 (4)
C5—H5A	0.9900	C21—H21	0.9500
C5—H5B	0.9900	C22—H22	0.9500
P1—Au—S1	169.35 (3)	C5—C4—H4B	110.9
C10[i]—Fe—C10	180.00 (19)	H4A—C4—H4B	109.0
C10[i]—Fe—C6[i]	41.11 (12)	N1—C5—C4	103.6 (2)
C10—Fe—C6[i]	138.89 (12)	N1—C5—H5A	111.0
C10[i]—Fe—C6	138.89 (12)	C4—C5—H5A	111.0
C10—Fe—C6	41.11 (12)	N1—C5—H5B	111.0
C6[i]—Fe—C6	180.0	C4—C5—H5B	111.0
C10[i]—Fe—C9	139.24 (12)	H5A—C5—H5B	109.0
C10—Fe—C9	40.76 (12)	C7—C6—C10	107.3 (3)
C6[i]—Fe—C9	111.54 (12)	C7—C6—P1	121.7 (2)
C6—Fe—C9	68.46 (12)	C10—C6—P1	131.0 (2)
C10[i]—Fe—C9[i]	40.76 (12)	C7—C6—Fe	70.32 (18)
C10—Fe—C9[i]	139.24 (13)	C10—C6—Fe	69.22 (17)
C6[i]—Fe—C9[i]	68.46 (12)	P1—C6—Fe	124.38 (15)
C6—Fe—C9[i]	111.54 (12)	C8—C7—C6	108.1 (3)
C9—Fe—C9[i]	180.0	C8—C7—Fe	70.42 (19)
C10[i]—Fe—C7[i]	68.50 (14)	C6—C7—Fe	68.85 (17)
C10—Fe—C7[i]	111.50 (13)	C8—C7—H7	125.9

C6ⁱ—Fe—C7ⁱ	40.83 (12)	C6—C7—H7	125.9
C6—Fe—C7ⁱ	139.17 (12)	Fe—C7—H7	126.4
C9—Fe—C7ⁱ	112.49 (14)	C9—C8—C7	108.2 (3)
C9ⁱ—Fe—C7ⁱ	67.51 (14)	C9—C8—Fe	69.09 (18)
C10ⁱ—Fe—C7	111.50 (13)	C7—C8—Fe	69.53 (18)
C10—Fe—C7	68.50 (14)	C9—C8—H8	125.9
C6ⁱ—Fe—C7	139.17 (12)	C7—C8—H8	125.9
C6—Fe—C7	40.83 (12)	Fe—C8—H8	127.1
C9—Fe—C7	67.51 (14)	C8—C9—C10	109.0 (3)
C9ⁱ—Fe—C7	112.49 (14)	C8—C9—Fe	71.15 (19)
C7ⁱ—Fe—C7	180.0	C10—C9—Fe	69.22 (17)
C10ⁱ—Fe—C8	112.02 (14)	C8—C9—H9	125.5
C10—Fe—C8	67.98 (14)	C10—C9—H9	125.5
C6ⁱ—Fe—C8	111.87 (12)	Fe—C9—H9	125.7
C6—Fe—C8	68.13 (12)	C9—C10—C6	107.4 (3)
C9—Fe—C8	39.76 (15)	C9—C10—Fe	70.02 (18)
C9ⁱ—Fe—C8	140.24 (15)	C6—C10—Fe	69.67 (18)
C7ⁱ—Fe—C8	139.95 (13)	C9—C10—H10	126.3
C7—Fe—C8	40.05 (13)	C6—C10—H10	126.3
C10ⁱ—Fe—C8ⁱ	67.98 (14)	Fe—C10—H10	125.6
C10—Fe—C8ⁱ	112.02 (14)	C12—C11—C16	119.4 (3)
C6ⁱ—Fe—C8ⁱ	68.13 (12)	C12—C11—P1	122.1 (2)
C6—Fe—C8ⁱ	111.87 (12)	C16—C11—P1	118.5 (2)
C9—Fe—C8ⁱ	140.24 (15)	C11—C12—C13	119.7 (3)
C9ⁱ—Fe—C8ⁱ	39.76 (15)	C11—C12—H12	120.1
C7ⁱ—Fe—C8ⁱ	40.05 (13)	C13—C12—H12	120.1
C7—Fe—C8ⁱ	139.95 (13)	C14—C13—C12	120.6 (3)
C8—Fe—C8ⁱ	180.0	C14—C13—H13	119.7
C1—S1—Au	98.40 (10)	C12—C13—H13	119.7
C6—P1—C17	105.75 (14)	C13—C14—C15	119.9 (3)
C6—P1—C11	105.61 (14)	C13—C14—H14	120.0
C17—P1—C11	103.37 (13)	C15—C14—H14	120.0
C6—P1—Au	108.05 (10)	C16—C15—C14	119.6 (3)
C17—P1—Au	115.04 (10)	C16—C15—H15	120.2
C11—P1—Au	118.03 (10)	C14—C15—H15	120.2
C1—N1—C2	124.6 (2)	C15—C16—C11	120.7 (3)
C1—N1—C5	123.5 (2)	C15—C16—H16	119.6
C2—N1—C5	111.5 (2)	C11—C16—H16	119.6
N1—C1—S2	121.7 (2)	C18—C17—C22	119.1 (3)
N1—C1—S1	116.5 (2)	C18—C17—P1	120.7 (2)
S2—C1—S1	121.79 (18)	C22—C17—P1	120.2 (2)
N1—C2—C3	103.7 (2)	C19—C18—C17	120.6 (3)
N1—C2—H2A	111.0	C19—C18—H18	119.7
C3—C2—H2A	111.0	C17—C18—H18	119.7
N1—C2—H2B	111.0	C20—C19—C18	119.3 (3)
C3—C2—H2B	111.0	C20—C19—H19	120.3
H2A—C2—H2B	109.0	C18—C19—H19	120.3
C4—C3—C2	104.7 (3)	C21—C20—C19	120.6 (3)

C4—C3—H3A	110.8	C21—C20—H20	119.7
C2—C3—H3A	110.8	C19—C20—H20	119.7
C4—C3—H3B	110.8	C20—C21—C22	120.2 (3)
C2—C3—H3B	110.8	C20—C21—H21	119.9
H3A—C3—H3B	108.9	C22—C21—H21	119.9
C3—C4—C5	104.1 (3)	C21—C22—C17	120.0 (3)
C3—C4—H4A	110.9	C21—C22—H22	120.0
C5—C4—H4A	110.9	C17—C22—H22	120.0
C3—C4—H4B	110.9		
C2—N1—C1—S2	−173.8 (2)	C8—C9—C10—Fe	60.3 (2)
C5—N1—C1—S2	−2.2 (4)	C7—C6—C10—C9	−0.2 (3)
C2—N1—C1—S1	6.1 (4)	P1—C6—C10—C9	178.2 (2)
C5—N1—C1—S1	177.7 (2)	Fe—C6—C10—C9	60.1 (2)
Au—S1—C1—N1	−164.2 (2)	C7—C6—C10—Fe	−60.3 (2)
Au—S1—C1—S2	15.7 (2)	P1—C6—C10—Fe	118.1 (3)
C1—N1—C2—C3	−179.5 (3)	C6—P1—C11—C12	−7.3 (3)
C5—N1—C2—C3	8.0 (3)	C17—P1—C11—C12	103.5 (3)
N1—C2—C3—C4	−27.0 (3)	Au—P1—C11—C12	−128.2 (2)
C2—C3—C4—C5	35.8 (3)	C6—P1—C11—C16	174.7 (2)
C1—N1—C5—C4	−158.8 (3)	C17—P1—C11—C16	−74.4 (3)
C2—N1—C5—C4	13.8 (3)	Au—P1—C11—C16	53.8 (3)
C3—C4—C5—N1	−30.3 (3)	C16—C11—C12—C13	−1.4 (5)
C17—P1—C6—C7	150.6 (2)	P1—C11—C12—C13	−179.3 (2)
C11—P1—C6—C7	−100.2 (3)	C11—C12—C13—C14	0.7 (5)
Au—P1—C6—C7	27.0 (3)	C12—C13—C14—C15	0.1 (5)
C17—P1—C6—C10	−27.5 (3)	C13—C14—C15—C16	−0.2 (5)
C11—P1—C6—C10	81.6 (3)	C14—C15—C16—C11	−0.5 (5)
Au—P1—C6—C10	−151.2 (3)	C12—C11—C16—C15	1.3 (5)
C17—P1—C6—Fe	63.9 (2)	P1—C11—C16—C15	179.3 (2)
C11—P1—C6—Fe	173.08 (17)	C6—P1—C17—C18	64.2 (3)
Au—P1—C6—Fe	−59.75 (19)	C11—P1—C17—C18	−46.6 (3)
C10—C6—C7—C8	−0.1 (4)	Au—P1—C17—C18	−176.7 (2)
P1—C6—C7—C8	−178.6 (2)	C6—P1—C17—C22	−113.7 (3)
Fe—C6—C7—C8	−59.7 (2)	C11—P1—C17—C22	135.5 (2)
C10—C6—C7—Fe	59.6 (2)	Au—P1—C17—C22	5.4 (3)
P1—C6—C7—Fe	−118.9 (2)	C22—C17—C18—C19	3.0 (4)
C6—C7—C8—C9	0.3 (4)	P1—C17—C18—C19	−175.0 (2)
Fe—C7—C8—C9	−58.4 (2)	C17—C18—C19—C20	−2.8 (5)
C6—C7—C8—Fe	58.7 (2)	C18—C19—C20—C21	0.7 (5)
C7—C8—C9—C10	−0.4 (4)	C19—C20—C21—C22	1.2 (5)
Fe—C8—C9—C10	−59.1 (2)	C20—C21—C22—C17	−1.0 (5)
C7—C8—C9—Fe	58.7 (2)	C18—C17—C22—C21	−1.0 (4)
C8—C9—C10—C6	0.4 (4)	P1—C17—C22—C21	176.9 (2)
Fe—C9—C10—C6	−59.9 (2)		

Symmetry code: (i) −x+1, −y, −z+2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C13—H13···S2[ii]	0.95	2.86	3.680 (3)	144
C20—H20···S2[iii]	0.95	2.84	3.628 (3)	141

Symmetry codes: (ii) *x*, −*y*+1/2, *z*+1/2; (iii) −*x*+1, −*y*, −*z*+1.