Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Crystal structure of chlorido[1-(4-nitrophenyl)-thiourea-κS]bis(triphenylphosphane-κP)silver(I)

Arunpatcha Nimthong-Roldán,[a] Paramee Sripa[b] and Yupa Wattanakanjana[b]*

[a]Department of Chemistry, Boston University, Boston, Massachusetts 02215, University, and [b]Department of Chemistry, Faculty of Science, Prince of Songkla University, Hat Yai, Songkhla 90112 , Thailand. *Correspondence e-mail: yupa.t@psu.ac.th

Received 14 March 2017
Accepted 28 April 2017

Edited by A. J. Lough, University of Toronto, Canada

Keywords: crystal structure; N—H···Cl hydrogen bonding; intramolecular hydrogen bonding; intermolecular hydrogen bonding.

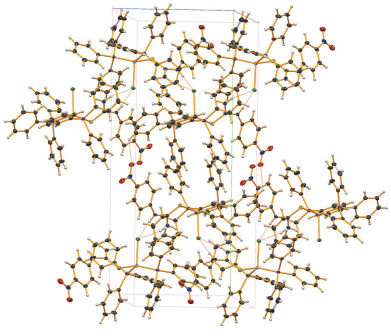
CCDC reference: 1546767

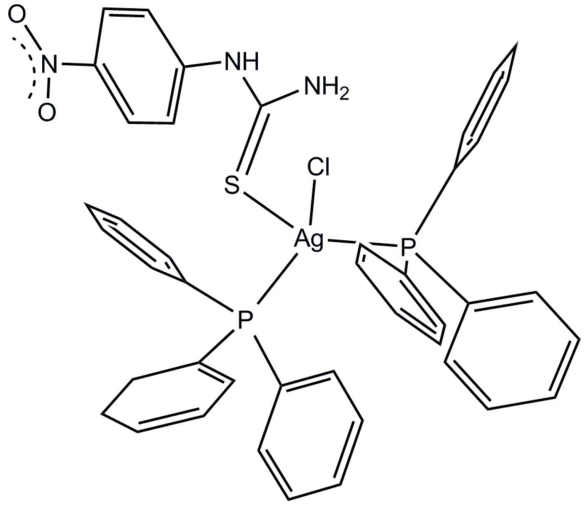
Supporting information: this article has supporting information at journals.iucr.org/e

In the title compound, $[AgCl(C_7H_7N_3O_2S)(C_{18}H_{15}P)_2]$, the Ag^I ion is in a distorted tetrahedral coordination environment formed by P atoms from two triphenylphosphane ligands, one terminal S atom from the 1-(4-nitrophenyl)-thiourea ligand and a chloride ion. In the crystal, bifurcated (N—H)2···Cl hydrogen bonds [with graph-set motif $R_2^1(6)$] connect complex molecules, forming zigzag chains along [001]. These chains are linked *via* weak C—H···O hydrogen bonds, forming a two-dimensional network parallel to (100). An intramolecular N—H···Cl hydrogen bond forming an $S(6)$ ring is also observed.

1. Chemical context

Studies of thiourea and thiourea derivatives have recently attracted considerable attention because of their variety of biological properties such as increasing technologies for plasma membrane proteomics (Cordwell & Thingholm, 2010), antimicrobial and cytotoxic activity (Bielenica *et al.*, 2015) and significant antifungal and anti-viral activity of curative rates (Wu *et al.*, 2012). Silver(I) complexes containing triphenylphosphane as precursors have been studied extensively for the preparation of mixed ligands with thiourea derivatives (Mekarat *et al.*, 2014; Wattanakanjana *et al.*, 2014). Recently, we reported a complex that was prepared by reacting copper(I) chloride containing triphenylphosphane and 1-(4-nitrophenyl)thiourea ligands (Nimthong-Roldán *et al.*, 2017). Herein, we report the crystal structure of the compound formed using silver(I) instead of copper(I) under the same conditions, $[AgCl(C_7H_7N_3O_2S)(C_{18}H_{15}P)_2]$ (I).





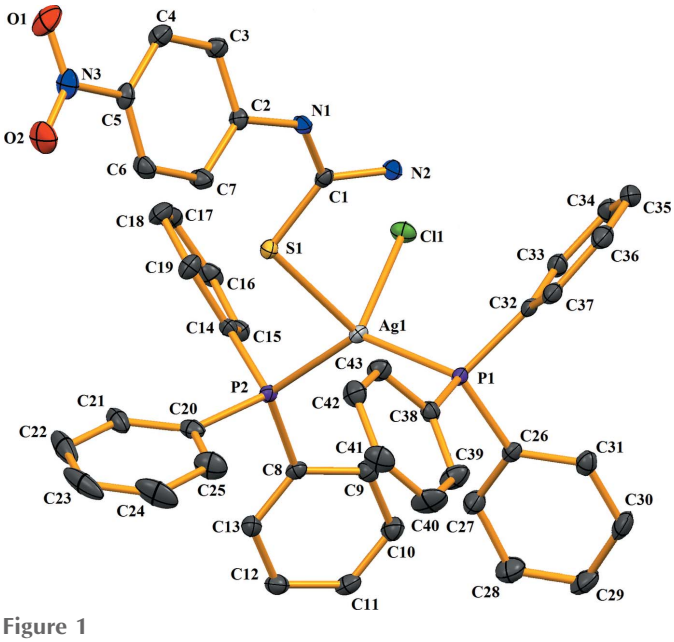


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level. All H atoms have been omitted for clarity.

2. Structural commentary

In compound (I), triphenylphosphane, PPh_3, and a 1-(4-nitrophenyl)thiourea ligand, NPTU, as co-ligands coordinate the Ag^I ion with two P atoms from two PPh_3 ligands, one terminal S atom from the NPTU ligand and one chloride ion, resulting in a distorted tetrahedral environment (Fig. 1). The Ag—S bond length of 2.6316 (5) is similar to that of 2.603 (4) Å found in $[Ag_2Cl_2(CH_5N_3S)_2(C_{18}H_{15}P)_2]$, (Wattanakanjana *et al.*, 2012). An intramolecular N2—H2*B*···Cl1 hydrogen bond with graph-set motif *S*(6) is observed (Table 1).

3. Supramolecular features

In the crystal, N2—H2*A*···Cl1 and N1—H1···Cl1 hydrogen bonds link the molecules, forming a zigzag chain along [001]. These chains are linked by weak C12—H12···O2 hydrogen bonds, leading to the formation of a two-dimensional network parallel to (100) (Fig. 2 and Table 1).

4. Database survey

A search of the Cambridge Structural Database (Version 5.37, Feb 2016 with two updates; Groom *et al.*, 2016) revealed no complexes with the 1-(4-nitrophenyl)thiourea ligand, and only the crystal structure of the ligand itself has been reported (LONSEN; Xian *et al.*, 2008). A search for phenylthiourea ligands with substitutions on the phenyl ring yielded 34 hits. Of these, four hits were Ag^I complexes, namely TUYZAQ (Wattanakanjana *et al.*, 2015), SUFDUU (Nimthong-Roldán *et al.*, 2015*b*), WUFBIK (Nimthong-Roldán *et al.*, 2015*a*), and XOFDED (Mekarat *et al.*, 2014)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···Cl1^{i}	0.88	2.41	3.2454 (17)	159
N2—H2*A*···Cl1^{i}	0.88 (2)	2.39 (2)	3.2257 (18)	160 (2)
N2—H2*B*···Cl1	0.87 (2)	2.50 (2)	3.3247 (18)	159 (2)
C12—H12···O2^{ii}	0.95	2.60	3.272 (3)	129

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

5. Synthesis and crystallization

Triphenylphosphane, PPh_3 (0.16 g, 0.51 mmol) was dissolved in 30 ml of acetonitrile at 340 K and then silver(I) chloride, AgCl (0.04 g, 0.25 mmol) , was added. The mixture was stirred for 3 h and then 1-(4-nitrophenyl)-2- thiourea, NPTU (0.05 g, 0.25 mmol), was added. The resulting reaction mixture was heated under reflux for 3 h during which the precipitate gradually disappeared. The resulting clear solution was filtered and left to evaporate at room temperature. The crystalline complex, which deposited upon standing for a couple of days, was filtered off and dried *in vacuo* (0.16 g, 66% yield). M.p. 465–467 K. IR bands (KBr, cm^{-1}): 3259 (*w*), 3134 (*w*), 3051(*w*), 2366 (*w*), 2345 (*w*), 1584 (*w*), 1509 (*w*), 1498 (*w*), 1458 (*w*), 1433 (*w*), 1399 (*w*), 1334 (*s*), 1297 (*w*), 1259 (*w*), 1181 (*w*), 1157 (*w*), 1110 (*w*), 1095 (*w*), 1027 (*w*), 998 (*w*), 890 (*w*), 851 (*w*), 746 (*m*), 720 (*w*), 694 (*s*), 670 (*w*), 594 (*w*), 515 (*m*), 501 (*m*), 491 (*m*).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All H atoms attached to carbon atoms and atom H1 attached to nitrogen atom N1 were

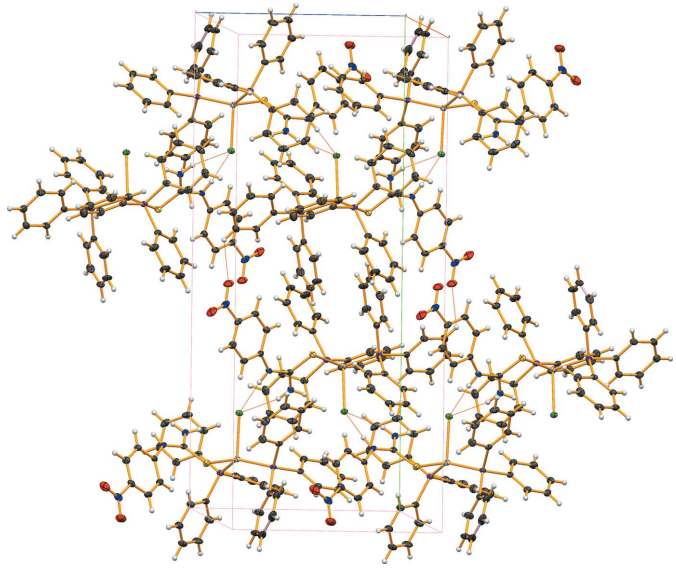


Figure 2
Part of the crystal structure of (I), showing the two-dimensional network formed by intermolecular N—H···Cl and C—H···O hydrogen bonds (shown as dashed lines) parallel to (100).

positioned geometrically and constrained to ride on their parent atoms, with C—H = 0.95 Å and N1—H1 = 0.88 Å. The other nitrogen-bound H atoms were located in difference-Fourier maps and were refined with an N—H distance restraint of 0.88 (2) Å. U_{iso}(H) values were set to 1.2U_{eq}(C/N). Reflections 1 1 0, $\overline{1}$ 1 1, 0 2 0, 1 2 0, 0 4 0, $\overline{1}$ 2 1, 0 2 1, 0 1 1, 1 0 0, −5 11, 13 8 1, 6 15 10, 12 10 4, $\overline{5}$ 15 13, $\overline{12}$ 20 2, 0 22 11, 12 1 5, $\overline{3}$ 23 11, $\overline{2}$ 26 10, 4 9 12, 10 14 6, $\overline{10}$ 20 9, 7 22 7, $\overline{14}$ 8 5, 10 10 7, 0 5 14, 7 5 10, $\overline{14}$ 8 4, $\overline{5}$ 25 10, $\overline{2}$ 20 12 and $\overline{12}$ 14 9 were affected by the beam stop and were omitted from the refinement.

Acknowledgements

Financial support from the Department of Chemistry, Faculty of Science, Prince of Songkla University, is gratefully acknowledged. We would like to thank Dr Matthias Zeller and Purdue University for assistance with the X-ray structure determination and use of structure refinement programs based on funding by the National Science Foundation of the United States (CHE-1625543).

Table 2
Experimental details.

Crystal data	
Chemical formula	$[AgCl(C_7H_7N_3O_2S)(C_{18}H_{15}P)_2]$
M_r	865.07
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	100
a, b, c (Å)	11.8581 (2), 28.5087 (4), 12.0272 (2)
β (°)	104.9338 (17)
V (Å^3)	3928.57 (11)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	6.33
Crystal size (mm)	0.25 × 0.23 × 0.18
Data collection	
Diffractometer	Rigaku RAPID II curved image plate diffractometer
Absorption correction	Multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min}, T_{max}	0.253, 0.395
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	73254, 7590, 7495
R_{int}	0.051
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.618
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.027, 0.068, 1.08
No. of reflections	7590
No. of parameters	485
No. of restraints	2
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.43, −0.37

Computer programs: *CrystalClear-SM Expert* (Rigaku, 2014), *HKL-3000* (Otwinowski & Minor, 1997), *SHELXS97* and *SHELXL97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *SHELXLE* (Hübschle *et al.*, 2011), *Mercury* (Macrae *et al.*, 2008) and *publCIF* (Westrip, 2010).

References

Bielenica, A., Stefańska, J., Stępień, K., Napiórkowska, A., Augustynowicz-Kopeć, E., Sanna, G., Madeddu, S., Boi, S., Giliberti, G., Wrzosek, M. & Struga, M. (2015). *Eur. J. Med. Chem.* **101**, 111–125.
Cordwell, S. C. & Thingholm, T. E. (2010). *Proteomics*, **10**, 611–627.
Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.
Hübschle, C. B., Sheldrick, G. M. & Dittrich, B. (2011). *J. Appl. Cryst.* **44**, 1281–1284.
Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
Mekarat, S., Pakawatchai, C. & Saithong, S. (2014). *Acta Cryst.* E**70**, m281–m282.
Nimthong-Roldán, A., Promsuwhan, N., Puetpaiboon, W. & Wattanakanjana, Y. (2017). *Acta Cryst.* E**73**, 41–44.
Nimthong-Roldán, A., Ratthiwal, J. & Wattanakanjana, Y. (2015*a*). *Acta Cryst.* E**71**, m133–m134.
Nimthong-Roldán, A., Wattanakanjana, Y. & Rodkeaw, J. (2015*b*). *Acta Cryst.* E**71**, m89–m90.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Rigaku (2014). *CrystalClear-SM Expert*. Rigaku Corporation, The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Wattanakanjana, Y., Nimthong, A. & Darasuriyong, C. (2014). *Acta Cryst.* E**70**, m337–m338.
Wattanakanjana, Y., Nimthong-Roldán, A., Palavat, S. & Puetpaiboon, W. (2015). *Acta Cryst.* E**71**, m187–m188.
Wattanakanjana, Y., Pakawatchai, C., Kowittheeraphong, S. & Nimthong, R. (2012). *Acta Cryst.* E**68**, m1572–m1573.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
Wu, J., Shi, Q., Chen, Z., He, M., Jin, L. & Hu, D. (2012). *Molecules*, **17**, 5139–5150.
Xian, L., Wang, Y. & Zhang, B. (2008). *Z. Kristallogr. New Cryst. Struct.* **223**, 411–412.

supporting information

Acta Cryst. (2017). E**73**, 829-831 [https://doi.org/10.1107/S2056989017006405]

Crystal structure of chlorido[1-(4-nitrophenyl)thiourea-κ*S*]bis(triphenylphosphane-κ*P*)silver(I)

Arunpatcha Nimthong-Roldán, Paramee Sripa and Yupa Wattanakanjana

Computing details

Data collection: *CrystalClear-SM Expert* (Rigaku, 2014); cell refinement: *HKL-3000* (Otwinowski & Minor, 1997); data reduction: *HKL-3000* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015) and *SHELXLE* (Hübschle *et al.*, 2011); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008) and *publCIF* (Westrip, 2010).

Chlorido[1-(4-nitrophenyl)thiourea-κ*S*]bis(triphenylphosphane-κ*P*)silver(I)

Crystal data

$[AgCl(C_7H_7N_3O_2S)(C_{18}H_{15}P)_2]$
M_r = 865.07
Monoclinic, $P2_1/c$
a = 11.8581 (2) Å
b = 28.5087 (4) Å
c = 12.0272 (2) Å
β = 104.9338 (17)°
V = 3928.57 (11) Å^3
Z = 4
$F(000)$ = 1768
D_x = 1.463 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 73254 reflections
θ = 6.0–72.3°
μ = 6.33 mm^{-1}
T = 100 K
Fragment, colourless
0.25 × 0.23 × 0.18 mm

Data collection

Rigaku RAPID II curved image plate diffractometer
Radiation source: microfocus X-ray tube
Laterally graded multilayer (Goebel) mirror monochromator
ω scans
Absorption correction: multi-scan (SCALEPACK; Otwinowski & Minor, 1997)
T_{min} = 0.253, T_{max} = 0.395
73254 measured reflections
7590 independent reflections
7495 reflections with $I > 2\sigma(I)$
R_{int} = 0.051
θ_{max} = 72.3°, θ_{min} = 6.0°
h = −13→14
k = −34→34
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.068
S = 1.08
7590 reflections
485 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0286P)^2 + 4.5052P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.37$ e Å^{-3}
Extinction correction: SHELXL2014 (Sheldrick, 2015), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00032 (3)

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.

Refinement. NH2 hydrogen positions were refined with an N-H distance restraint of 0.88 (2) Angstrom.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.82775 (17)	0.17354 (7)	0.72805 (16)	0.0133 (4)
C2	0.96312 (17)	0.14219 (7)	0.61654 (16)	0.0139 (4)
C3	1.03535 (18)	0.16067 (7)	0.55233 (18)	0.0165 (4)
H3	1.0332	0.1933	0.5361	0.020*
C4	1.10993 (18)	0.13194 (7)	0.51217 (18)	0.0190 (4)
H4	1.1578	0.1444	0.4670	0.023*
C5	1.11381 (18)	0.08464 (7)	0.53871 (17)	0.0174 (4)
C6	1.04603 (19)	0.06567 (7)	0.60585 (18)	0.0194 (4)
H6	1.0514	0.0333	0.6249	0.023*
C7	0.97043 (19)	0.09454 (7)	0.64472 (18)	0.0187 (4)
H7	0.9235	0.0819	0.6906	0.022*
C8	0.74747 (17)	0.13044 (7)	1.26846 (16)	0.0138 (4)
C9	0.65144 (19)	0.15917 (7)	1.26283 (18)	0.0179 (4)
H9	0.6270	0.1806	1.2007	0.022*
C10	0.5909 (2)	0.15680 (7)	1.3476 (2)	0.0210 (5)
H10	0.5264	0.1770	1.3442	0.025*
C11	0.62485 (18)	0.12492 (7)	1.43677 (18)	0.0189 (4)
H11	0.5831	0.1230	1.4943	0.023*
C12	0.71971 (18)	0.09583 (7)	1.44220 (17)	0.0190 (4)
H12	0.7422	0.0737	1.5030	0.023*
C13	0.78211 (18)	0.09879 (7)	1.35929 (17)	0.0166 (4)
H13	0.8482	0.0793	1.3644	0.020*
C14	0.93770 (17)	0.17768 (6)	1.20401 (16)	0.0136 (4)
C15	0.93999 (18)	0.20815 (7)	1.29504 (17)	0.0164 (4)
H15	0.8819	0.2059	1.3364	0.020*
C16	1.02734 (19)	0.24204 (7)	1.32554 (18)	0.0205 (4)
H16	1.0283	0.2629	1.3873	0.025*
C17	1.11251 (19)	0.24539 (7)	1.26628 (19)	0.0225 (5)
H17	1.1731	0.2680	1.2886	0.027*
C18	1.10938 (19)	0.21572 (8)	1.17397 (19)	0.0235 (5)
H18	1.1675	0.2182	1.1326	0.028*
C19	1.02183 (18)	0.18265 (8)	1.14223 (18)	0.0192 (4)

H19	1.0188	0.1631	1.0776	0.023*
C20	0.89340 (19)	0.07855 (7)	1.15416 (17)	0.0181 (4)
C21	1.0099 (2)	0.06968 (8)	1.21129 (18)	0.0233 (5)
H21	1.0565	0.0936	1.2558	0.028*
C22	1.0570 (3)	0.02535 (9)	1.2023 (2)	0.0361 (6)
H22	1.1365	0.0193	1.2399	0.043*
C23	0.9895 (3)	−0.00974 (9)	1.1395 (2)	0.0416 (7)
H23	1.0225	−0.0397	1.1336	0.050*
C24	0.8739 (3)	−0.00123 (8)	1.0852 (2)	0.0399 (7)
H24	0.8268	−0.0256	1.0435	0.048*
C25	0.8263 (2)	0.04271 (8)	1.0911 (2)	0.0294 (5)
H25	0.7472	0.0485	1.0518	0.035*
C26	0.40317 (17)	0.12920 (7)	0.91747 (17)	0.0144 (4)
C27	0.43678 (19)	0.11373 (8)	1.03101 (18)	0.0211 (4)
H27	0.5168	0.1076	1.0661	0.025*
C28	0.3548 (2)	0.10713 (8)	1.0934 (2)	0.0258 (5)
H28	0.3784	0.0962	1.1705	0.031*
C29	0.2380 (2)	0.11659 (8)	1.0429 (2)	0.0251 (5)
H29	0.1817	0.1122	1.0855	0.030*
C30	0.20359 (19)	0.13244 (8)	0.9305 (2)	0.0236 (5)
H30	0.1237	0.1393	0.8966	0.028*
C31	0.28542 (18)	0.13847 (7)	0.86665 (19)	0.0189 (4)
H31	0.2613	0.1488	0.7891	0.023*
C32	0.45452 (17)	0.17405 (7)	0.72094 (17)	0.0145 (4)
C33	0.45617 (18)	0.22199 (7)	0.74552 (19)	0.0190 (4)
H33	0.4862	0.2326	0.8224	0.023*
C34	0.41417 (19)	0.25422 (7)	0.6582 (2)	0.0229 (5)
H34	0.4137	0.2867	0.6758	0.028*
C35	0.37279 (19)	0.23917 (8)	0.5452 (2)	0.0237 (5)
H35	0.3453	0.2613	0.4853	0.028*
C36	0.37170 (19)	0.19174 (8)	0.52005 (19)	0.0225 (4)
H36	0.3438	0.1814	0.4427	0.027*
C37	0.41126 (18)	0.15910 (7)	0.60767 (18)	0.0178 (4)
H37	0.4087	0.1266	0.5901	0.021*
C38	0.51237 (17)	0.07648 (7)	0.77110 (17)	0.0151 (4)
C39	0.4272 (2)	0.04295 (8)	0.7711 (2)	0.0248 (5)
H39	0.3669	0.0495	0.8077	0.030*
C40	0.4301 (2)	−0.00014 (8)	0.7180 (2)	0.0308 (5)
H40	0.3719	−0.0229	0.7187	0.037*
C41	0.5173 (2)	−0.01007 (7)	0.66374 (19)	0.0252 (5)
H41	0.5186	−0.0395	0.6270	0.030*
C42	0.60271 (19)	0.02319 (7)	0.66335 (18)	0.0212 (4)
H42	0.6624	0.0166	0.6259	0.025*
C43	0.60110 (19)	0.06598 (7)	0.71745 (18)	0.0186 (4)
H43	0.6606	0.0884	0.7182	0.022*
N1	0.88013 (15)	0.17306 (6)	0.64028 (14)	0.0148 (3)
H1	0.8585	0.1958	0.5899	0.018*
N2	0.74178 (15)	0.20477 (6)	0.71595 (15)	0.0162 (3)

H2A	0.727 (2)	0.2227 (8)	0.6547 (17)	0.019*
H2B	0.721 (2)	0.2127 (8)	0.7774 (17)	0.019*
N3	1.18951 (16)	0.05374 (6)	0.49253 (16)	0.0226 (4)
O1	1.25615 (14)	0.07175 (6)	0.44157 (15)	0.0309 (4)
O2	1.18095 (15)	0.01114 (6)	0.50467 (15)	0.0314 (4)
S1	0.86821 (4)	0.13848 (2)	0.84576 (4)	0.01445 (10)
Cl1	0.73410 (4)	0.24665 (2)	0.97189 (4)	0.01787 (10)
Ag1	0.70884 (2)	0.15453 (2)	0.95816 (2)	0.01254 (6)
P1	0.51614 (4)	0.13383 (2)	0.83979 (4)	0.01185 (10)
P2	0.82115 (4)	0.13508 (2)	1.15318 (4)	0.01158 (10)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0103 (9)	0.0120 (9)	0.0179 (9)	−0.0021 (7)	0.0045 (7)	−0.0016 (7)
C2	0.0143 (10)	0.0136 (9)	0.0140 (9)	0.0017 (7)	0.0042 (7)	−0.0015 (7)
C3	0.0171 (11)	0.0123 (9)	0.0214 (10)	−0.0012 (7)	0.0072 (8)	−0.0011 (7)
C4	0.0163 (11)	0.0192 (10)	0.0233 (10)	−0.0021 (8)	0.0082 (8)	−0.0030 (8)
C5	0.0146 (10)	0.0176 (10)	0.0202 (10)	0.0040 (8)	0.0049 (8)	−0.0038 (8)
C6	0.0235 (11)	0.0126 (9)	0.0230 (10)	0.0048 (8)	0.0076 (9)	0.0022 (8)
C7	0.0222 (11)	0.0156 (10)	0.0214 (10)	0.0022 (8)	0.0113 (8)	0.0022 (8)
C8	0.0136 (10)	0.0127 (9)	0.0157 (9)	−0.0030 (7)	0.0047 (7)	−0.0008 (7)
C9	0.0188 (11)	0.0146 (10)	0.0213 (10)	0.0012 (8)	0.0068 (8)	0.0018 (8)
C10	0.0192 (12)	0.0178 (10)	0.0293 (12)	0.0029 (8)	0.0118 (9)	−0.0001 (8)
C11	0.0185 (11)	0.0180 (10)	0.0234 (10)	−0.0045 (8)	0.0113 (8)	−0.0033 (8)
C12	0.0207 (11)	0.0178 (10)	0.0192 (10)	−0.0020 (8)	0.0064 (8)	0.0031 (8)
C13	0.0140 (10)	0.0152 (9)	0.0208 (10)	0.0011 (7)	0.0052 (8)	0.0019 (8)
C14	0.0120 (10)	0.0103 (9)	0.0179 (9)	0.0008 (7)	0.0028 (7)	0.0028 (7)
C15	0.0173 (10)	0.0130 (9)	0.0192 (10)	0.0013 (7)	0.0056 (8)	0.0005 (7)
C16	0.0251 (12)	0.0116 (9)	0.0219 (10)	−0.0014 (8)	0.0009 (9)	−0.0014 (8)
C17	0.0177 (11)	0.0165 (10)	0.0289 (11)	−0.0052 (8)	−0.0020 (9)	0.0039 (8)
C18	0.0150 (11)	0.0290 (12)	0.0278 (11)	−0.0047 (9)	0.0077 (9)	0.0037 (9)
C19	0.0136 (10)	0.0227 (11)	0.0215 (10)	−0.0018 (8)	0.0050 (8)	−0.0021 (8)
C20	0.0256 (11)	0.0145 (10)	0.0177 (9)	0.0016 (8)	0.0116 (8)	0.0019 (8)
C21	0.0290 (12)	0.0211 (11)	0.0218 (10)	0.0117 (9)	0.0105 (9)	0.0064 (8)
C22	0.0482 (16)	0.0373 (14)	0.0283 (12)	0.0303 (12)	0.0197 (11)	0.0134 (11)
C23	0.085 (2)	0.0183 (12)	0.0312 (13)	0.0258 (13)	0.0327 (14)	0.0094 (10)
C24	0.076 (2)	0.0108 (11)	0.0357 (14)	0.0014 (12)	0.0192 (14)	−0.0020 (9)
C25	0.0399 (15)	0.0176 (11)	0.0316 (12)	−0.0019 (10)	0.0110 (11)	−0.0044 (9)
C26	0.0130 (10)	0.0102 (9)	0.0215 (10)	−0.0009 (7)	0.0072 (8)	−0.0034 (7)
C27	0.0159 (11)	0.0244 (11)	0.0234 (10)	0.0003 (8)	0.0061 (8)	0.0003 (8)
C28	0.0268 (12)	0.0283 (12)	0.0262 (11)	−0.0029 (9)	0.0137 (9)	0.0009 (9)
C29	0.0224 (12)	0.0209 (11)	0.0390 (13)	−0.0035 (9)	0.0208 (10)	−0.0038 (9)
C30	0.0125 (11)	0.0210 (11)	0.0400 (13)	−0.0005 (8)	0.0115 (9)	−0.0033 (9)
C31	0.0152 (10)	0.0157 (10)	0.0255 (10)	0.0003 (8)	0.0049 (8)	−0.0028 (8)
C32	0.0082 (9)	0.0146 (9)	0.0218 (10)	−0.0002 (7)	0.0056 (7)	0.0013 (7)
C33	0.0131 (10)	0.0168 (10)	0.0262 (10)	0.0000 (8)	0.0034 (8)	0.0000 (8)
C34	0.0152 (11)	0.0150 (10)	0.0380 (12)	0.0000 (8)	0.0059 (9)	0.0027 (9)

C35	0.0147 (11)	0.0254 (11)	0.0299 (11)	0.0012 (8)	0.0041 (9)	0.0124 (9)
C36	0.0159 (11)	0.0321 (12)	0.0201 (10)	−0.0002 (9)	0.0054 (8)	0.0043 (9)
C37	0.0121 (10)	0.0200 (10)	0.0224 (10)	−0.0012 (7)	0.0064 (8)	−0.0004 (8)
C38	0.0147 (10)	0.0120 (9)	0.0177 (9)	0.0017 (7)	0.0027 (8)	0.0005 (7)
C39	0.0241 (12)	0.0201 (11)	0.0333 (12)	−0.0063 (9)	0.0131 (10)	−0.0074 (9)
C40	0.0374 (14)	0.0161 (11)	0.0423 (14)	−0.0132 (10)	0.0165 (11)	−0.0091 (10)
C41	0.0345 (13)	0.0134 (10)	0.0283 (11)	−0.0011 (9)	0.0089 (10)	−0.0050 (8)
C42	0.0228 (11)	0.0169 (10)	0.0252 (10)	0.0034 (8)	0.0088 (9)	−0.0027 (8)
C43	0.0185 (11)	0.0142 (10)	0.0242 (10)	−0.0020 (8)	0.0073 (8)	−0.0027 (8)
N1	0.0176 (9)	0.0113 (8)	0.0181 (8)	0.0044 (6)	0.0091 (7)	0.0038 (6)
N2	0.0171 (9)	0.0159 (8)	0.0181 (8)	0.0049 (6)	0.0090 (7)	0.0037 (7)
N3	0.0205 (10)	0.0211 (9)	0.0262 (9)	0.0052 (7)	0.0063 (8)	−0.0058 (7)
O1	0.0248 (9)	0.0320 (9)	0.0425 (10)	0.0001 (7)	0.0205 (8)	−0.0101 (7)
O2	0.0366 (10)	0.0196 (8)	0.0410 (10)	0.0112 (7)	0.0158 (8)	−0.0010 (7)
S1	0.0120 (2)	0.0160 (2)	0.0164 (2)	0.00335 (17)	0.00569 (17)	0.00330 (17)
Cl1	0.0236 (3)	0.0121 (2)	0.0184 (2)	−0.00173 (17)	0.00615 (18)	−0.00192 (16)
Ag1	0.00999 (9)	0.01314 (8)	0.01461 (8)	−0.00147 (4)	0.00338 (6)	0.00063 (5)
P1	0.0085 (2)	0.0110 (2)	0.0165 (2)	−0.00051 (16)	0.00401 (18)	−0.00144 (17)
P2	0.0108 (2)	0.0098 (2)	0.0145 (2)	−0.00040 (17)	0.00404 (18)	0.00016 (17)

Geometric parameters (Å, º)

C1—N2	1.333 (3)	C24—C25	1.384 (3)
C1—N1	1.356 (2)	C24—H24	0.9500
C1—S1	1.698 (2)	C25—H25	0.9500
C2—C3	1.396 (3)	C26—C27	1.392 (3)
C2—C7	1.397 (3)	C26—C31	1.398 (3)
C2—N1	1.403 (2)	C26—P1	1.825 (2)
C3—C4	1.381 (3)	C27—C28	1.386 (3)
C3—H3	0.9500	C27—H27	0.9500
C4—C5	1.384 (3)	C28—C29	1.387 (3)
C4—H4	0.9500	C28—H28	0.9500
C5—C6	1.388 (3)	C29—C30	1.384 (3)
C5—N3	1.465 (3)	C29—H29	0.9500
C6—C7	1.384 (3)	C30—C31	1.394 (3)
C6—H6	0.9500	C30—H30	0.9500
C7—H7	0.9500	C31—H31	0.9500
C8—C9	1.390 (3)	C32—C37	1.393 (3)
C8—C13	1.395 (3)	C32—C33	1.398 (3)
C8—P2	1.824 (2)	C32—P1	1.831 (2)
C9—C10	1.391 (3)	C33—C34	1.388 (3)
C9—H9	0.9500	C33—H33	0.9500
C10—C11	1.385 (3)	C34—C35	1.389 (3)
C10—H10	0.9500	C34—H34	0.9500
C11—C12	1.385 (3)	C35—C36	1.385 (3)
C11—H11	0.9500	C35—H35	0.9500
C12—C13	1.389 (3)	C36—C37	1.393 (3)
C12—H12	0.9500	C36—H36	0.9500

C13—H13	0.9500	C37—H37	0.9500
C14—C15	1.392 (3)	C38—C39	1.391 (3)
C14—C19	1.396 (3)	C38—C43	1.401 (3)
C14—P2	1.823 (2)	C38—P1	1.827 (2)
C15—C16	1.395 (3)	C39—C40	1.389 (3)
C15—H15	0.9500	C39—H39	0.9500
C16—C17	1.381 (3)	C40—C41	1.387 (3)
C16—H16	0.9500	C40—H40	0.9500
C17—C18	1.389 (3)	C41—C42	1.388 (3)
C17—H17	0.9500	C41—H41	0.9500
C18—C19	1.381 (3)	C42—C43	1.385 (3)
C18—H18	0.9500	C42—H42	0.9500
C19—H19	0.9500	C43—H43	0.9500
C20—C25	1.394 (3)	N1—H1	0.8800
C20—C21	1.398 (3)	N2—H2A	0.877 (16)
C20—P2	1.824 (2)	N2—H2B	0.870 (16)
C21—C22	1.397 (3)	N3—O1	1.230 (3)
C21—H21	0.9500	N3—O2	1.231 (2)
C22—C23	1.379 (4)	S1—Ag1	2.6316 (5)
C22—H22	0.9500	Cl1—Ag1	2.6435 (5)
C23—C24	1.379 (4)	Ag1—P1	2.4330 (5)
C23—H23	0.9500	Ag1—P2	2.4440 (5)

N2—C1—N1	114.37 (17)	C28—C27—H27	119.6
N2—C1—S1	121.88 (15)	C26—C27—H27	119.6
N1—C1—S1	123.74 (15)	C27—C28—C29	119.8 (2)
C3—C2—C7	119.50 (18)	C27—C28—H28	120.1
C3—C2—N1	116.00 (17)	C29—C28—H28	120.1
C7—C2—N1	124.28 (18)	C30—C29—C28	120.1 (2)
C4—C3—C2	120.63 (19)	C30—C29—H29	120.0
C4—C3—H3	119.7	C28—C29—H29	120.0
C2—C3—H3	119.7	C29—C30—C31	120.4 (2)
C3—C4—C5	118.92 (19)	C29—C30—H30	119.8
C3—C4—H4	120.5	C31—C30—H30	119.8
C5—C4—H4	120.5	C30—C31—C26	119.6 (2)
C4—C5—C6	121.62 (19)	C30—C31—H31	120.2
C4—C5—N3	119.05 (19)	C26—C31—H31	120.2
C6—C5—N3	119.31 (18)	C37—C32—C33	119.18 (19)
C7—C6—C5	119.18 (19)	C37—C32—P1	122.97 (15)
C7—C6—H6	120.4	C33—C32—P1	117.79 (15)
C5—C6—H6	120.4	C34—C33—C32	120.3 (2)
C6—C7—C2	120.08 (19)	C34—C33—H33	119.8
C6—C7—H7	120.0	C32—C33—H33	119.8
C2—C7—H7	120.0	C33—C34—C35	120.2 (2)
C9—C8—C13	119.44 (18)	C33—C34—H34	119.9
C9—C8—P2	117.84 (15)	C35—C34—H34	119.9
C13—C8—P2	122.70 (15)	C36—C35—C34	119.7 (2)
C8—C9—C10	120.44 (19)	C36—C35—H35	120.1

C8—C9—H9	119.8	C34—C35—H35	120.1
C10—C9—H9	119.8	C35—C36—C37	120.3 (2)
C11—C10—C9	119.8 (2)	C35—C36—H36	119.8
C11—C10—H10	120.1	C37—C36—H36	119.8
C9—C10—H10	120.1	C32—C37—C36	120.2 (2)
C10—C11—C12	120.00 (19)	C32—C37—H37	119.9
C10—C11—H11	120.0	C36—C37—H37	119.9
C12—C11—H11	120.0	C39—C38—C43	118.99 (18)
C11—C12—C13	120.40 (19)	C39—C38—P1	123.24 (16)
C11—C12—H12	119.8	C43—C38—P1	117.76 (15)
C13—C12—H12	119.8	C40—C39—C38	120.3 (2)
C12—C13—C8	119.86 (19)	C40—C39—H39	119.9
C12—C13—H13	120.1	C38—C39—H39	119.9
C8—C13—H13	120.1	C41—C40—C39	120.4 (2)
C15—C14—C19	118.90 (18)	C41—C40—H40	119.8
C15—C14—P2	122.60 (15)	C39—C40—H40	119.8
C19—C14—P2	118.24 (15)	C40—C41—C42	119.7 (2)
C14—C15—C16	120.11 (19)	C40—C41—H41	120.2
C14—C15—H15	119.9	C42—C41—H41	120.2
C16—C15—H15	119.9	C43—C42—C41	120.1 (2)
C17—C16—C15	120.3 (2)	C43—C42—H42	119.9
C17—C16—H16	119.9	C41—C42—H42	119.9
C15—C16—H16	119.9	C42—C43—C38	120.50 (19)
C16—C17—C18	119.86 (19)	C42—C43—H43	119.7
C16—C17—H17	120.1	C38—C43—H43	119.7
C18—C17—H17	120.1	C1—N1—C2	130.59 (17)
C19—C18—C17	120.0 (2)	C1—N1—H1	114.7
C19—C18—H18	120.0	C2—N1—H1	114.7
C17—C18—H18	120.0	C1—N2—H2A	117.7 (16)
C18—C19—C14	120.8 (2)	C1—N2—H2B	117.4 (16)
C18—C19—H19	119.6	H2A—N2—H2B	122 (2)
C14—C19—H19	119.6	O1—N3—O2	123.66 (18)
C25—C20—C21	119.2 (2)	O1—N3—C5	118.22 (18)
C25—C20—P2	116.29 (17)	O2—N3—C5	118.10 (18)
C21—C20—P2	124.46 (17)	C1—S1—Ag1	103.88 (7)
C22—C21—C20	119.3 (2)	P1—Ag1—P2	134.741 (17)
C22—C21—H21	120.3	P1—Ag1—S1	110.349 (16)
C20—C21—H21	120.3	P2—Ag1—S1	99.654 (16)
C23—C22—C21	120.8 (3)	P1—Ag1—Cl1	110.591 (16)
C23—C22—H22	119.6	P2—Ag1—Cl1	98.075 (15)
C21—C22—H22	119.6	S1—Ag1—Cl1	96.894 (15)
C22—C23—C24	119.8 (2)	C26—P1—C38	103.36 (9)
C22—C23—H23	120.1	C26—P1—C32	104.31 (9)
C24—C23—H23	120.1	C38—P1—C32	104.41 (9)
C23—C24—C25	120.3 (3)	C26—P1—Ag1	114.88 (7)
C23—C24—H24	119.9	C38—P1—Ag1	113.04 (7)
C25—C24—H24	119.9	C32—P1—Ag1	115.51 (6)
C24—C25—C20	120.5 (3)	C14—P2—C20	105.80 (9)

C24—C25—H25	119.7	C14—P2—C8	105.29 (9)
C20—C25—H25	119.7	C20—P2—C8	104.31 (9)
C27—C26—C31	119.35 (19)	C14—P2—Ag1	110.44 (6)
C27—C26—P1	117.57 (15)	C20—P2—Ag1	110.21 (7)
C31—C26—P1	123.02 (16)	C8—P2—Ag1	119.78 (7)
C28—C27—C26	120.7 (2)		

C7—C2—C3—C4	2.9 (3)	C38—C39—C40—C41	−0.4 (4)
N1—C2—C3—C4	−171.96 (18)	C39—C40—C41—C42	0.4 (4)
C2—C3—C4—C5	−1.4 (3)	C40—C41—C42—C43	0.4 (3)
C3—C4—C5—C6	−1.0 (3)	C41—C42—C43—C38	−1.2 (3)
C3—C4—C5—N3	177.66 (18)	C39—C38—C43—C42	1.2 (3)
C4—C5—C6—C7	1.7 (3)	P1—C38—C43—C42	−179.65 (16)
N3—C5—C6—C7	−176.90 (19)	N2—C1—N1—C2	−171.97 (19)
C5—C6—C7—C2	−0.1 (3)	S1—C1—N1—C2	9.2 (3)
C3—C2—C7—C6	−2.1 (3)	C3—C2—N1—C1	−153.6 (2)
N1—C2—C7—C6	172.30 (19)	C7—C2—N1—C1	31.8 (3)
C13—C8—C9—C10	0.6 (3)	C4—C5—N3—O1	7.4 (3)
P2—C8—C9—C10	179.24 (16)	C6—C5—N3—O1	−173.91 (19)
C8—C9—C10—C11	−1.4 (3)	C4—C5—N3—O2	−171.0 (2)
C9—C10—C11—C12	0.7 (3)	C6—C5—N3—O2	7.7 (3)
C10—C11—C12—C13	0.8 (3)	N2—C1—S1—Ag1	7.68 (17)
C11—C12—C13—C8	−1.5 (3)	N1—C1—S1—Ag1	−173.58 (15)
C9—C8—C13—C12	0.8 (3)	C27—C26—P1—C38	91.38 (17)
P2—C8—C13—C12	−177.71 (15)	C31—C26—P1—C38	−85.81 (18)
C19—C14—C15—C16	−1.9 (3)	C27—C26—P1—C32	−159.69 (16)
P2—C14—C15—C16	−175.92 (15)	C31—C26—P1—C32	23.13 (19)
C14—C15—C16—C17	−0.5 (3)	C27—C26—P1—Ag1	−32.24 (18)
C15—C16—C17—C18	1.7 (3)	C31—C26—P1—Ag1	150.58 (15)
C16—C17—C18—C19	−0.6 (3)	C39—C38—P1—C26	8.1 (2)
C17—C18—C19—C14	−1.8 (3)	C43—C38—P1—C26	−170.92 (16)
C15—C14—C19—C18	3.1 (3)	C39—C38—P1—C32	−100.71 (19)
P2—C14—C19—C18	177.33 (17)	C43—C38—P1—C32	80.22 (17)
C25—C20—C21—C22	1.1 (3)	C39—C38—P1—Ag1	132.96 (17)
P2—C20—C21—C22	−177.62 (17)	C43—C38—P1—Ag1	−46.11 (17)
C20—C21—C22—C23	−1.0 (3)	C37—C32—P1—C26	−104.98 (18)
C21—C22—C23—C24	−0.3 (4)	C33—C32—P1—C26	77.84 (17)
C22—C23—C24—C25	1.6 (4)	C37—C32—P1—C38	3.18 (19)
C23—C24—C25—C20	−1.6 (4)	C33—C32—P1—C38	−174.00 (16)
C21—C20—C25—C24	0.2 (3)	C37—C32—P1—Ag1	127.96 (16)
P2—C20—C25—C24	179.03 (19)	C33—C32—P1—Ag1	−49.22 (17)
C31—C26—C27—C28	0.5 (3)	C15—C14—P2—C20	−126.80 (16)
P1—C26—C27—C28	−176.77 (17)	C19—C14—P2—C20	59.16 (17)
C26—C27—C28—C29	−0.8 (3)	C15—C14—P2—C8	−16.70 (18)
C27—C28—C29—C30	0.2 (3)	C19—C14—P2—C8	169.26 (16)
C28—C29—C30—C31	0.8 (3)	C15—C14—P2—Ag1	113.94 (15)
C29—C30—C31—C26	−1.1 (3)	C19—C14—P2—Ag1	−60.09 (16)
C27—C26—C31—C30	0.5 (3)	C25—C20—P2—C14	−163.63 (17)

P1—C26—C31—C30	177.59 (16)	C21—C20—P2—C14	15.1 (2)
C37—C32—C33—C34	0.6 (3)	C25—C20—P2—C8	85.58 (18)
P1—C32—C33—C34	177.89 (16)	C21—C20—P2—C8	−95.71 (19)
C32—C33—C34—C35	−1.6 (3)	C25—C20—P2—Ag1	−44.22 (18)
C33—C34—C35—C36	1.1 (3)	C21—C20—P2—Ag1	134.49 (16)
C34—C35—C36—C37	0.4 (3)	C9—C8—P2—C14	92.78 (17)
C33—C32—C37—C36	0.9 (3)	C13—C8—P2—C14	−88.67 (17)
P1—C32—C37—C36	−176.27 (16)	C9—C8—P2—C20	−156.06 (16)
C35—C36—C37—C32	−1.4 (3)	C13—C8—P2—C20	22.50 (19)
C43—C38—C39—C40	−0.4 (3)	C9—C8—P2—Ag1	−32.22 (18)
P1—C38—C39—C40	−179.50 (19)	C13—C8—P2—Ag1	146.34 (14)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···Cl1^{i}	0.88	2.41	3.2454 (17)	159
N2—H2*A*···Cl1^{i}	0.88 (2)	2.39 (2)	3.2257 (18)	160 (2)
N2—H2*B*···Cl1	0.87 (2)	2.50 (2)	3.3247 (18)	159 (2)
C12—H12···O2^{ii}	0.95	2.60	3.272 (3)	129

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