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μ_3-Iodo-tri-μ_3-sulfido-sulfidotris[tris(4-methoxyphenyl)phosphine-κP]-tricopper(I)tungsten(VI) *N*,*N*-dimethylformide solvate

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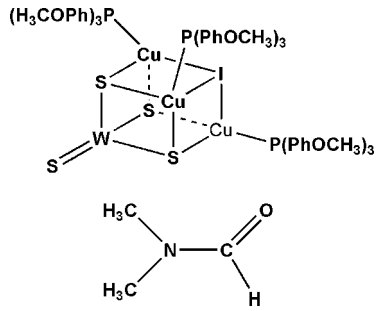
Key indicators: single-crystal X-ray study; T = 193 K; mean σ(C–C) = 0.008 Å; R factor = 0.042; wR factor = 0.088; data-to-parameter ratio = 15.6.

A new W/S/Cu cluster, $[Cu_3WIS_4(C_{21}H_{21}O_3P)_3]\cdot C_3H_7NO$, was formed by the reaction of ammonium tetrathiotungstate(VI), cuprous iodide and tris(4-methoxyphenyl)phosphine in *N*,*N*-dimethylformamide. The title compound exhibits a heavily distorted cubane-like skeleton in which the average Cu—I, Cu—S and W—μ_3-S distances are 2.934, 2.302 and 2.249 Å, respectively. The W atom exhibits tetrahedral geometry, formed by three μ_3-S and one terminal S atom; the W—S(terminal) bond length is 2.1426 (13) Å. Each Cu atom is coordinated by one P atom from a tris(4-methoxyphenyl)phosphine (mop), two μ_3-S and one μ_3-I atom, forming a distorted tetrahedral coordination geometry. Some of the mop ligand methyl groups have large librations. Together with the three neutral mop ligands, the title compound is neutral; this contrasts with the all-halogen-coordinated Mo/S/Ag clusters with the same structure, which carry negative charge.

Related literature

Two relevant analogs of the title compound are $[n\text{-}Bu_4N]_3[MoS_4Ag_3BrX_3]$ (X = Cl and I; Shi *et al.*, 1994). Mo(W)/S/Cu(Ag) clusters have been reviewed by How *et al.* (1996) and Niu *et al.* (2004). The nonlinear optical properties of Mo(W)/S/Cu(Ag) clusters have been reviewed by Zhang *et al.* (2007).



Experimental

Crystal data

$[Cu_3WIS_4(C_{21}H_{21}O_3P)_3]\cdot C_3H_7NO$
M_r = 1759.75
Triclinic, $P\overline{1}$
a = 10.5383 (11) Å
b = 18.5135 (16) Å
c = 19.3412 (19) Å
α = 68.869 (5)°
β = 84.706 (6)°
γ = 80.605 (6)°
V = 3470.2 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 3.25 mm^{-1}
T = 193 (2) K
0.60 × 0.28 × 0.17 mm

Data collection

Rigaku Mercury diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.248, T_{max} = 0.575
34152 measured reflections
12730 independent reflections
11088 reflections with $I > 2\sigma(I)$
R_{int} = 0.042

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.088
S = 1.14
12730 reflections
814 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.16 e Å^{-3}
$\Delta\rho_{min}$ = −1.44 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C64—H64···O10^{i}	0.95	2.62	3.126 (13)	114
C55—H55···I1	0.95	2.96	3.888 (5)	164
C49—H49B···I1^{ii}	0.98	3.30	3.864 (5)	119
C42—H42B···S2^{iii}	0.98	2.85	3.650 (5)	139
C38—H38···S4^{iv}	0.95	2.88	3.646 (5)	138
C34—H34···S2	0.95	2.96	3.860 (5)	158
C23—H23···S1	0.95	2.88	3.794 (5)	162
C17—H17···O2^{iv}	0.95	2.49	3.437 (7)	177
C9—H9···O7^{v}	0.95	2.51	3.273 (6)	137

Symmetry codes: (i) $-x+2, -y+1, -z$; (ii) $-x+2, -y+2, -z$; (iii) $-x+2, -y+1, -z+1$; (iv) $x+1, y, z$; (v) $-x+1, -y+2, -z$.

Data collection: *CrystalClear* (Rigaku, 2000); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BR2058).

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supporting information

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μ_3-Iodo-tri-μ_3-sulfido-sulfidotris[tris(4-methoxyphenyl)phosphine-κP]tricopper(I)tungsten(VI) *N*,*N*-dimethylformide solvate

Guodong Tang, Jinfang Zhang and Chi Zhang

S1. Comment

Thiometallates can aggregate with closed d-shell metals to form clusters having unusal geometry (How, Xin *et al.*, 1996; Niu *et al.*, 2004) and featuring nonlinear optical properties (Zhang *et al.*, 2007). But the crystal structures of these clusters containing neutral tri(4-methoxylphenyl)phosphine ligands (mop) have not been reported until now. In order to explore the chemistry of Mo(W)/S/Cu(Ag) clusters extensively,we have synthesized the title cluster by the reaction in solution at normal temperatures.

As illustrated in Fig. 1, the title compound exhibits a heavily distorted cubane-like skeleton,where the average Cu—I, Cu—S and W—S distances are 2.934, 2.302 and 2.249 Å,respectively. The W(VI) retains the tetrhedral geometry, formed by three μ_3-S and one terminal S atoms; each Cu(I) is coordinated by one P atom from mop, two μ_3-S and one μ_3-I atoms, forming a distorted tetrahedral coordination geometry.

Together with three neutral mop ligands, the title compound is neutral, This contrasts with the all-halogen-coordinated Mo(W)/S/Ag cluster with the same structure,which carries a negative charge (Shi *et al.*, 1994).

S2. Experimental

3 mmol CuI, 1 mmol $[NH_4]_2WS_4$ and 3 mmol mop were added in 5 mL dmf with thorough stir for 5 minutes. After filtration, the orange-red filtrate was carefully laid on the surface with 10 ml *i*-PrOH. Yellow block crystals were obtained after about ten days. Yield: 1.058 g in pure form, 60.1% (based on W). Analysis calculated for $C_{66}H_{70}Cu_3INO_{10}P_3S_4W$: C 45.05, H 4.01, N 0.80%; found: C 45.02, H 4.03, N 0.81%. IR: ν, cm^{-1}, 504.35 s, 444.74 s(W-μ_3-S).

S3. Refinement

H atoms were positioned geometrically and refined with riding model, with $U_{iso} = 1.5U_{eq}$ for methyl H atoms and $1.2U_{eq}$ for phenyl H atoms. C—H bonds are 0.95 Å in phenyl and 0.98 Å in methyl.

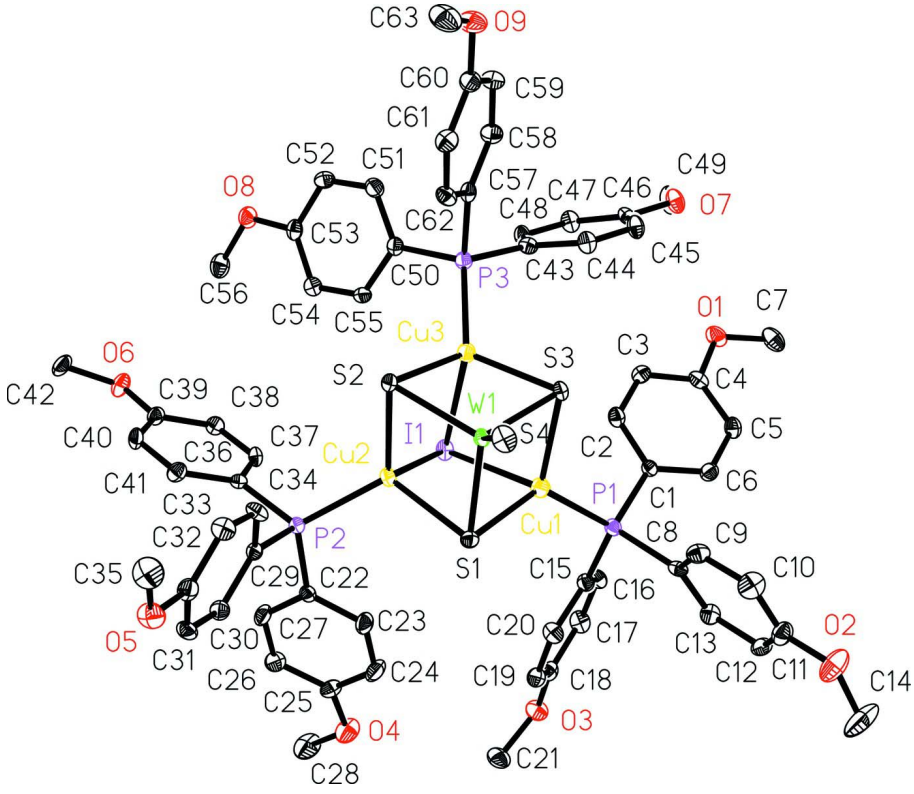

Figure 1

The molecular structure of the title compound, with atom labels and 30% probability displacement ellipsoids. All H atoms have been omitted.

μ_3-Iodo-tri-μ_3-sulfido-sulfidotris[tris(4-methoxyphenyl)phosphine- κP]tricopper(I)tungsten(VI) *N*,*N*-dimethylformide solvate

Crystal data

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Hall symbol: -P 1
$a = 10.5383\ (11)$ Å
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$c = 19.3412\ (19)$ Å
$\alpha = 68.869\ (5)°$
$\beta = 84.706\ (6)°$
$\gamma = 80.605\ (6)°$
$V = 3470.2\ (6)$ Å^3
$Z = 2$
$F(000) = 1752$
$D_x = 1.684$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71070$ Å
Cell parameters from 13164 reflections
$\theta = 3.0–25.3°$
$\mu = 3.25$ mm^{-1}
$T = 193$ K
Block, orange
0.60 × 0.28 × 0.17 mm

Data collection

Rigaku Mercury diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 7.31 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.248$, $T_{max} = 0.575$
34152 measured reflections
12730 independent reflections
11088 reflections with $I > 2\sigma(I)$
$R_{int} = 0.042$

θ_{max} = 25.4°, θ_{min} = 3.0°
h = −12→12
k = −22→22
l = −23→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.088
S = 1.14
12730 reflections
814 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
$w = 1/[\sigma^2(F_o^2) + (0.0341P)^2 + 1.022P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.16 e Å^{-3}
$\Delta\rho_{min}$ = −1.44 e Å^{-3}

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
W1	0.399659 (17)	0.826474 (11)	0.320297 (10)	0.02020 (7)
I1	0.78025 (3)	0.884325 (19)	0.301151 (17)	0.02774 (9)
Cu1	0.50642 (6)	0.95940 (4)	0.27402 (3)	0.03064 (16)
Cu2	0.61078 (6)	0.79755 (4)	0.40397 (3)	0.02737 (15)
Cu3	0.61930 (6)	0.79911 (4)	0.24231 (3)	0.03038 (16)
S1	0.43252 (11)	0.89443 (7)	0.39090 (6)	0.0242 (3)
S2	0.54227 (12)	0.71584 (7)	0.35247 (6)	0.0251 (3)
S3	0.44925 (11)	0.89628 (7)	0.20204 (6)	0.0232 (3)
S4	0.20427 (13)	0.80367 (9)	0.33394 (8)	0.0368 (3)
P1	0.52675 (12)	1.08505 (7)	0.21974 (7)	0.0231 (3)
P2	0.70089 (12)	0.74719 (7)	0.51424 (7)	0.0216 (3)
P3	0.74062 (12)	0.75451 (8)	0.16219 (7)	0.0237 (3)
O1	0.7833 (3)	1.1405 (2)	−0.07965 (18)	0.0391 (9)
O2	0.0315 (4)	1.3019 (3)	0.1789 (3)	0.0623 (13)
O3	0.8203 (3)	1.2235 (2)	0.37847 (19)	0.0374 (9)
O4	0.8635 (4)	1.0038 (2)	0.5884 (2)	0.0504 (11)
O5	0.3289 (4)	0.5759 (2)	0.7424 (2)	0.0429 (10)
O6	1.1917 (3)	0.5281 (2)	0.5370 (2)	0.0386 (9)
O7	0.8379 (3)	0.9912 (2)	−0.13538 (18)	0.0367 (9)
O8	1.2404 (3)	0.5585 (2)	0.27700 (19)	0.0361 (9)
O9	0.4908 (4)	0.5467 (2)	0.0580 (2)	0.0452 (10)
O10	0.9702 (12)	0.4409 (6)	−0.0350 (5)	0.182 (5)
N1	0.8345 (7)	0.3748 (5)	0.0374 (4)	0.087 (2)

C1	0.5987 (4)	1.1061 (3)	0.1267 (2)	0.0226 (11)
C2	0.6850 (5)	1.0483 (3)	0.1110 (3)	0.0283 (12)
H2	0.7038	0.9985	0.1488	0.034*
C3	0.7433 (5)	1.0617 (3)	0.0422 (3)	0.0309 (12)
H3	0.8025	1.0214	0.0328	0.037*
C4	0.7169 (5)	1.1333 (3)	−0.0137 (3)	0.0287 (12)
C5	0.6296 (5)	1.1911 (3)	−0.0006 (3)	0.0309 (12)
H5	0.6099	1.2402	−0.0392	0.037*
C6	0.5702 (5)	1.1775 (3)	0.0692 (3)	0.0288 (12)
H6	0.5093	1.2174	0.0779	0.035*
C7	0.7590 (6)	1.2122 (4)	−0.1391 (3)	0.0462 (16)
H7A	0.6683	1.2216	−0.1517	0.069*
H7B	0.8137	1.2099	−0.1823	0.069*
H7C	0.7781	1.2547	−0.1246	0.069*
C8	0.3783 (5)	1.1532 (3)	0.2070 (2)	0.0236 (11)
C9	0.2633 (5)	1.1293 (3)	0.2001 (3)	0.0318 (12)
H9	0.2623	1.0770	0.2030	0.038*
C10	0.1505 (5)	1.1811 (4)	0.1889 (3)	0.0453 (15)
H10	0.0728	1.1647	0.1827	0.054*
C11	0.1499 (5)	1.2563 (3)	0.1867 (3)	0.0378 (13)
C12	0.2630 (5)	1.2816 (3)	0.1925 (3)	0.0330 (13)
H12	0.2635	1.3340	0.1895	0.040*
C13	0.3760 (5)	1.2295 (3)	0.2028 (3)	0.0309 (12)
H13	0.4541	1.2467	0.2071	0.037*
C14	0.0244 (7)	1.3766 (4)	0.1859 (5)	0.091 (3)
H14A	0.0593	1.3702	0.2333	0.137*
H14B	−0.0656	1.4011	0.1840	0.137*
H14C	0.0745	1.4099	0.1451	0.137*
C15	0.6246 (5)	1.1232 (3)	0.2678 (3)	0.0262 (11)
C16	0.7235 (5)	1.1658 (3)	0.2345 (3)	0.0286 (12)
H16	0.7474	1.1735	0.1840	0.034*
C17	0.7884 (5)	1.1976 (3)	0.2727 (3)	0.0351 (13)
H17	0.8569	1.2262	0.2488	0.042*
C18	0.7534 (5)	1.1876 (3)	0.3465 (3)	0.0289 (12)
C19	0.6547 (5)	1.1449 (3)	0.3806 (3)	0.0305 (12)
H19	0.6297	1.1380	0.4307	0.037*
C20	0.5925 (5)	1.1123 (3)	0.3421 (3)	0.0305 (12)
H20	0.5264	1.0818	0.3666	0.037*
C21	0.8006 (6)	1.2060 (4)	0.4570 (3)	0.0436 (15)
H21A	0.7112	1.2246	0.4678	0.065*
H21B	0.8586	1.2322	0.4738	0.065*
H21C	0.8187	1.1494	0.4828	0.065*
C22	0.7476 (4)	0.8209 (3)	0.5442 (2)	0.0226 (11)
C23	0.6890 (5)	0.8972 (3)	0.5150 (3)	0.0356 (13)
H23	0.6202	0.9093	0.4824	0.043*
C24	0.7279 (5)	0.9567 (3)	0.5320 (3)	0.0426 (15)
H24	0.6850	1.0086	0.5114	0.051*
C25	0.8288 (5)	0.9411 (3)	0.5786 (3)	0.0311 (12)

C26	0.8862 (5)	0.8650 (3)	0.6096 (3)	0.0351 (13)
H26	0.9542	0.8530	0.6427	0.042*
C27	0.8460 (5)	0.8055 (3)	0.5930 (3)	0.0336 (13)
H27	0.8865	0.7532	0.6153	0.040*
C28	0.9723 (6)	0.9930 (4)	0.6306 (4)	0.0573 (18)
H28A	0.9570	0.9580	0.6815	0.086*
H28B	0.9875	1.0436	0.6311	0.086*
H28C	1.0479	0.9698	0.6084	0.086*
C29	0.5894 (4)	0.6943 (3)	0.5847 (2)	0.0229 (11)
C30	0.5779 (5)	0.6949 (3)	0.6571 (3)	0.0281 (12)
H30	0.6314	0.7233	0.6715	0.034*
C31	0.4906 (5)	0.6551 (3)	0.7077 (3)	0.0312 (12)
H31	0.4837	0.6565	0.7566	0.037*
C32	0.4121 (5)	0.6129 (3)	0.6876 (3)	0.0311 (12)
C33	0.4212 (5)	0.6114 (3)	0.6168 (3)	0.0325 (12)
H33	0.3683	0.5824	0.6028	0.039*
C34	0.5092 (5)	0.6532 (3)	0.5657 (3)	0.0302 (12)
H34	0.5140	0.6532	0.5164	0.036*
C35	0.2483 (6)	0.5303 (4)	0.7255 (3)	0.0519 (17)
H35A	0.1905	0.5643	0.6862	0.078*
H35B	0.1973	0.5043	0.7700	0.078*
H35C	0.3016	0.4908	0.7087	0.078*
C36	0.8465 (4)	0.6772 (3)	0.5241 (2)	0.0224 (11)
C37	0.9537 (4)	0.6993 (3)	0.4773 (2)	0.0241 (11)
H37	0.9483	0.7502	0.4406	0.029*
C38	1.0663 (5)	0.6488 (3)	0.4833 (3)	0.0271 (11)
H38	1.1383	0.6653	0.4514	0.033*
C39	1.0757 (5)	0.5738 (3)	0.5356 (3)	0.0270 (11)
C40	0.9708 (5)	0.5504 (3)	0.5824 (3)	0.0296 (12)
H40	0.9762	0.4989	0.6180	0.036*
C41	0.8581 (5)	0.6020 (3)	0.5771 (3)	0.0286 (12)
H41	0.7874	0.5859	0.6102	0.034*
C42	1.1992 (5)	0.4470 (3)	0.5816 (3)	0.0403 (14)
H42A	1.1891	0.4414	0.6341	0.060*
H42B	1.2831	0.4194	0.5726	0.060*
H42C	1.1306	0.4247	0.5687	0.060*
C43	0.7802 (4)	0.8289 (3)	0.0747 (3)	0.0247 (11)
C44	0.6792 (5)	0.8740 (3)	0.0303 (3)	0.0294 (12)
H44	0.5931	0.8680	0.0483	0.035*
C45	0.7016 (5)	0.9271 (3)	−0.0390 (3)	0.0324 (12)
H45	0.6310	0.9564	−0.0689	0.039*
C46	0.8261 (5)	0.9383 (3)	−0.0658 (3)	0.0274 (12)
C47	0.9280 (5)	0.8961 (3)	−0.0211 (3)	0.0305 (12)
H47	1.0138	0.9045	−0.0378	0.037*
C48	0.9038 (5)	0.8414 (3)	0.0484 (3)	0.0282 (12)
H48	0.9741	0.8119	0.0783	0.034*
C49	0.9607 (5)	1.0150 (4)	−0.1609 (3)	0.0470 (16)
H49A	0.9899	1.0376	−0.1276	0.071*

H49B	0.9545	1.0541	−0.2111	0.071*
H49C	1.0225	0.9695	−0.1617	0.071*
C50	0.8925 (4)	0.6942 (3)	0.1970 (3)	0.0243 (11)
C51	0.9556 (5)	0.6379 (3)	0.1684 (3)	0.0321 (12)
H51	0.9189	0.6303	0.1288	0.039*
C52	1.0695 (5)	0.5933 (3)	0.1961 (3)	0.0333 (13)
H52	1.1106	0.5549	0.1763	0.040*
C53	1.1244 (4)	0.6047 (3)	0.2537 (3)	0.0268 (11)
C54	1.0637 (5)	0.6590 (3)	0.2832 (3)	0.0311 (12)
H54	1.1009	0.6665	0.3226	0.037*
C55	0.9478 (5)	0.7028 (3)	0.2553 (3)	0.0285 (12)
H55	0.9055	0.7396	0.2766	0.034*
C56	1.3030 (5)	0.5718 (3)	0.3328 (3)	0.0434 (15)
H56A	1.3114	0.6274	0.3172	0.065*
H56B	1.3886	0.5408	0.3400	0.065*
H56C	1.2519	0.5564	0.3794	0.065*
C57	0.6601 (4)	0.6930 (3)	0.1297 (3)	0.0247 (11)
C58	0.6936 (5)	0.6841 (3)	0.0609 (3)	0.0317 (12)
H58	0.7573	0.7120	0.0293	0.038*
C59	0.6351 (5)	0.6354 (3)	0.0392 (3)	0.0335 (13)
H59	0.6586	0.6300	−0.0075	0.040*
C60	0.5423 (5)	0.5940 (3)	0.0844 (3)	0.0334 (13)
C61	0.5060 (5)	0.6027 (3)	0.1520 (3)	0.0339 (13)
H61	0.4411	0.5753	0.1830	0.041*
C62	0.5657 (5)	0.6522 (3)	0.1741 (3)	0.0296 (12)
H62	0.5412	0.6580	0.2205	0.036*
C63	0.3998 (7)	0.4996 (4)	0.1038 (3)	0.0581 (18)
H63A	0.4378	0.4670	0.1514	0.087*
H63B	0.3760	0.4661	0.0792	0.087*
H63C	0.3229	0.5334	0.1124	0.087*
C64	0.8871 (10)	0.4386 (7)	0.0048 (7)	0.102 (3)
H64	0.8531	0.4843	0.0156	0.122*
C65	0.7263 (8)	0.3801 (6)	0.0877 (5)	0.108 (3)
H65A	0.7437	0.3402	0.1367	0.162*
H65B	0.6490	0.3716	0.0688	0.162*
H65C	0.7126	0.4321	0.0917	0.162*
C66	0.8757 (9)	0.3037 (5)	0.0244 (5)	0.092 (3)
H66A	0.9469	0.3108	−0.0127	0.137*
H66B	0.8040	0.2887	0.0062	0.137*
H66C	0.9048	0.2625	0.0708	0.137*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
W1	0.01969 (11)	0.02098 (12)	0.01796 (11)	−0.00449 (8)	−0.00053 (8)	−0.00382 (8)
I1	0.02311 (17)	0.0277 (2)	0.02850 (19)	−0.00488 (14)	−0.00152 (14)	−0.00455 (14)
Cu1	0.0427 (4)	0.0229 (4)	0.0254 (3)	−0.0109 (3)	0.0020 (3)	−0.0054 (3)
Cu2	0.0315 (3)	0.0265 (4)	0.0229 (3)	0.0010 (3)	−0.0087 (3)	−0.0075 (3)

Cu3	0.0321 (4)	0.0300 (4)	0.0228 (3)	0.0036 (3)	0.0034 (3)	−0.0063 (3)
S1	0.0253 (6)	0.0242 (7)	0.0228 (6)	−0.0016 (5)	0.0014 (5)	−0.0092 (5)
S2	0.0323 (7)	0.0186 (7)	0.0221 (6)	−0.0013 (5)	−0.0042 (5)	−0.0048 (5)
S3	0.0235 (6)	0.0243 (7)	0.0177 (6)	−0.0023 (5)	−0.0033 (5)	−0.0025 (5)
S4	0.0283 (7)	0.0433 (9)	0.0369 (8)	−0.0150 (6)	0.0023 (6)	−0.0086 (7)
P1	0.0262 (7)	0.0214 (7)	0.0210 (7)	−0.0056 (5)	−0.0008 (5)	−0.0057 (5)
P2	0.0232 (7)	0.0216 (7)	0.0194 (6)	−0.0014 (5)	−0.0030 (5)	−0.0067 (5)
P3	0.0219 (7)	0.0260 (7)	0.0219 (7)	−0.0020 (5)	0.0014 (5)	−0.0080 (6)
O1	0.039 (2)	0.052 (3)	0.024 (2)	−0.0059 (18)	0.0060 (17)	−0.0123 (18)
O2	0.028 (2)	0.054 (3)	0.099 (4)	0.006 (2)	0.003 (2)	−0.026 (3)
O3	0.036 (2)	0.044 (2)	0.038 (2)	−0.0138 (18)	−0.0025 (17)	−0.0179 (18)
O4	0.050 (3)	0.040 (3)	0.073 (3)	−0.004 (2)	−0.024 (2)	−0.029 (2)
O5	0.044 (2)	0.045 (3)	0.040 (2)	−0.018 (2)	0.0146 (19)	−0.0136 (19)
O6	0.030 (2)	0.028 (2)	0.049 (2)	0.0077 (16)	−0.0053 (17)	−0.0086 (18)
O7	0.037 (2)	0.040 (2)	0.026 (2)	−0.0152 (18)	−0.0024 (16)	0.0013 (17)
O8	0.030 (2)	0.037 (2)	0.036 (2)	0.0044 (17)	−0.0045 (17)	−0.0103 (18)
O9	0.050 (2)	0.048 (3)	0.048 (2)	−0.018 (2)	−0.004 (2)	−0.025 (2)
O10	0.272 (14)	0.157 (9)	0.142 (8)	−0.102 (9)	0.029 (8)	−0.061 (7)
N1	0.101 (6)	0.077 (5)	0.068 (4)	−0.014 (5)	−0.006 (4)	−0.007 (4)
C1	0.023 (3)	0.024 (3)	0.022 (3)	−0.003 (2)	−0.004 (2)	−0.008 (2)
C2	0.027 (3)	0.030 (3)	0.023 (3)	−0.003 (2)	−0.001 (2)	−0.004 (2)
C3	0.030 (3)	0.030 (3)	0.033 (3)	0.001 (2)	−0.001 (2)	−0.014 (2)
C4	0.027 (3)	0.040 (3)	0.022 (3)	−0.009 (2)	0.000 (2)	−0.013 (2)
C5	0.032 (3)	0.029 (3)	0.025 (3)	−0.005 (2)	−0.001 (2)	−0.001 (2)
C6	0.029 (3)	0.026 (3)	0.030 (3)	−0.001 (2)	0.001 (2)	−0.010 (2)
C7	0.053 (4)	0.057 (4)	0.024 (3)	−0.019 (3)	0.004 (3)	−0.006 (3)
C8	0.029 (3)	0.027 (3)	0.019 (3)	−0.010 (2)	0.004 (2)	−0.011 (2)
C9	0.030 (3)	0.033 (3)	0.040 (3)	−0.010 (2)	0.000 (2)	−0.020 (3)
C10	0.029 (3)	0.051 (4)	0.060 (4)	−0.012 (3)	0.001 (3)	−0.022 (3)
C11	0.029 (3)	0.037 (4)	0.043 (3)	0.003 (3)	0.000 (3)	−0.012 (3)
C12	0.038 (3)	0.028 (3)	0.034 (3)	0.000 (2)	0.003 (2)	−0.015 (2)
C13	0.029 (3)	0.031 (3)	0.034 (3)	−0.007 (2)	−0.002 (2)	−0.011 (2)
C14	0.065 (5)	0.064 (6)	0.149 (8)	0.027 (4)	−0.008 (5)	−0.057 (6)
C15	0.026 (3)	0.027 (3)	0.025 (3)	−0.002 (2)	−0.002 (2)	−0.009 (2)
C16	0.025 (3)	0.037 (3)	0.022 (3)	−0.008 (2)	0.004 (2)	−0.007 (2)
C17	0.025 (3)	0.043 (3)	0.035 (3)	−0.011 (2)	0.002 (2)	−0.008 (3)
C18	0.022 (3)	0.028 (3)	0.037 (3)	0.001 (2)	−0.012 (2)	−0.012 (2)
C19	0.033 (3)	0.027 (3)	0.030 (3)	−0.004 (2)	−0.001 (2)	−0.008 (2)
C20	0.032 (3)	0.032 (3)	0.028 (3)	−0.015 (2)	0.001 (2)	−0.007 (2)
C21	0.050 (4)	0.051 (4)	0.039 (3)	−0.013 (3)	−0.009 (3)	−0.022 (3)
C22	0.023 (3)	0.025 (3)	0.019 (3)	−0.004 (2)	0.000 (2)	−0.006 (2)
C23	0.035 (3)	0.030 (3)	0.041 (3)	0.006 (2)	−0.018 (3)	−0.012 (3)
C24	0.048 (4)	0.025 (3)	0.057 (4)	0.009 (3)	−0.019 (3)	−0.019 (3)
C25	0.029 (3)	0.028 (3)	0.042 (3)	−0.004 (2)	−0.001 (2)	−0.019 (3)
C26	0.028 (3)	0.041 (4)	0.041 (3)	0.002 (3)	−0.014 (2)	−0.021 (3)
C27	0.041 (3)	0.018 (3)	0.041 (3)	0.000 (2)	−0.014 (3)	−0.009 (2)
C28	0.056 (4)	0.046 (4)	0.086 (5)	−0.010 (3)	−0.023 (4)	−0.036 (4)
C29	0.021 (3)	0.023 (3)	0.022 (3)	0.003 (2)	−0.003 (2)	−0.007 (2)

C30	0.030 (3)	0.027 (3)	0.029 (3)	−0.005 (2)	−0.004 (2)	−0.011 (2)
C31	0.045 (3)	0.030 (3)	0.019 (3)	−0.011 (3)	0.007 (2)	−0.008 (2)
C32	0.027 (3)	0.030 (3)	0.032 (3)	−0.001 (2)	0.004 (2)	−0.008 (2)
C33	0.028 (3)	0.037 (3)	0.035 (3)	−0.011 (2)	0.000 (2)	−0.014 (3)
C34	0.033 (3)	0.041 (3)	0.021 (3)	−0.007 (2)	−0.002 (2)	−0.015 (2)
C35	0.044 (4)	0.054 (4)	0.054 (4)	−0.022 (3)	0.015 (3)	−0.014 (3)
C36	0.027 (3)	0.022 (3)	0.021 (3)	−0.003 (2)	−0.002 (2)	−0.011 (2)
C37	0.025 (3)	0.021 (3)	0.021 (3)	−0.004 (2)	−0.003 (2)	0.000 (2)
C38	0.024 (3)	0.029 (3)	0.028 (3)	−0.005 (2)	0.002 (2)	−0.009 (2)
C39	0.023 (3)	0.026 (3)	0.032 (3)	0.003 (2)	−0.007 (2)	−0.011 (2)
C40	0.035 (3)	0.020 (3)	0.026 (3)	−0.001 (2)	−0.003 (2)	0.001 (2)
C41	0.025 (3)	0.026 (3)	0.034 (3)	−0.003 (2)	0.003 (2)	−0.010 (2)
C42	0.042 (3)	0.025 (3)	0.052 (4)	0.013 (3)	−0.012 (3)	−0.015 (3)
C43	0.025 (3)	0.028 (3)	0.025 (3)	−0.008 (2)	0.002 (2)	−0.011 (2)
C44	0.020 (3)	0.034 (3)	0.031 (3)	−0.007 (2)	−0.001 (2)	−0.007 (2)
C45	0.024 (3)	0.037 (3)	0.032 (3)	−0.003 (2)	−0.009 (2)	−0.005 (2)
C46	0.032 (3)	0.031 (3)	0.020 (3)	−0.007 (2)	−0.003 (2)	−0.008 (2)
C47	0.020 (3)	0.038 (3)	0.030 (3)	−0.008 (2)	0.002 (2)	−0.006 (2)
C48	0.023 (3)	0.031 (3)	0.026 (3)	0.001 (2)	−0.005 (2)	−0.006 (2)
C49	0.041 (3)	0.051 (4)	0.032 (3)	−0.018 (3)	0.007 (3)	0.008 (3)
C50	0.021 (3)	0.026 (3)	0.021 (3)	−0.001 (2)	0.003 (2)	−0.005 (2)
C51	0.032 (3)	0.036 (3)	0.027 (3)	−0.001 (2)	−0.003 (2)	−0.011 (2)
C52	0.036 (3)	0.034 (3)	0.032 (3)	0.002 (2)	0.003 (2)	−0.017 (3)
C53	0.022 (3)	0.025 (3)	0.026 (3)	−0.004 (2)	0.003 (2)	−0.001 (2)
C54	0.034 (3)	0.030 (3)	0.028 (3)	−0.003 (2)	−0.004 (2)	−0.009 (2)
C55	0.032 (3)	0.029 (3)	0.027 (3)	0.003 (2)	−0.003 (2)	−0.015 (2)
C56	0.034 (3)	0.042 (4)	0.048 (4)	0.002 (3)	−0.012 (3)	−0.010 (3)
C57	0.021 (3)	0.024 (3)	0.024 (3)	0.001 (2)	−0.002 (2)	−0.004 (2)
C58	0.029 (3)	0.036 (3)	0.031 (3)	−0.007 (2)	0.005 (2)	−0.015 (3)
C59	0.037 (3)	0.038 (3)	0.032 (3)	−0.008 (3)	0.001 (2)	−0.018 (3)
C60	0.033 (3)	0.032 (3)	0.039 (3)	−0.004 (2)	−0.010 (2)	−0.015 (3)
C61	0.031 (3)	0.032 (3)	0.039 (3)	−0.008 (2)	0.001 (2)	−0.012 (3)
C62	0.030 (3)	0.037 (3)	0.022 (3)	−0.005 (2)	0.001 (2)	−0.012 (2)
C63	0.070 (5)	0.064 (5)	0.052 (4)	−0.036 (4)	−0.006 (3)	−0.023 (4)
C64	0.088 (8)	0.101 (9)	0.112 (9)	−0.026 (7)	−0.003 (6)	−0.026 (7)
C65	0.087 (6)	0.148 (9)	0.070 (6)	0.040 (6)	−0.004 (5)	−0.038 (6)
C66	0.123 (8)	0.090 (7)	0.086 (6)	−0.043 (6)	0.030 (5)	−0.055 (5)

Geometric parameters (Å, º)

W1—S4	2.1426 (13)	C21—H21B	0.9800
W1—S1	2.2440 (12)	C21—H21C	0.9800
W1—S3	2.2458 (12)	C22—C23	1.379 (7)
W1—S2	2.2560 (12)	C22—C27	1.392 (6)
W1—Cu1	2.6931 (7)	C23—C24	1.387 (7)
W1—Cu3	2.7227 (7)	C23—H23	0.9500
W1—Cu2	2.7398 (7)	C24—C25	1.385 (7)
I1—Cu2	2.7754 (7)	C24—H24	0.9500

I1—Cu1	2.9963 (8)	C25—C26	1.374 (7)
I1—Cu3	3.0308 (8)	C26—C27	1.390 (7)
Cu1—P1	2.2182 (14)	C26—H26	0.9500
Cu1—S1	2.2769 (13)	C27—H27	0.9500
Cu1—S3	2.2856 (13)	C28—H28A	0.9800
Cu2—P2	2.2295 (13)	C28—H28B	0.9800
Cu2—S2	2.3189 (13)	C28—H28C	0.9800
Cu2—S1	2.3389 (13)	C29—C34	1.378 (7)
Cu3—P3	2.2202 (14)	C29—C30	1.398 (6)
Cu3—S3	2.2911 (13)	C30—C31	1.371 (7)
Cu3—S2	2.3013 (13)	C30—H30	0.9500
P1—C1	1.817 (5)	C31—C32	1.392 (7)
P1—C8	1.818 (5)	C31—H31	0.9500
P1—C15	1.820 (5)	C32—C33	1.374 (7)
P2—C22	1.811 (5)	C33—C34	1.396 (7)
P2—C36	1.815 (5)	C33—H33	0.9500
P2—C29	1.821 (5)	C34—H34	0.9500
P3—C43	1.819 (5)	C35—H35A	0.9800
P3—C50	1.826 (5)	C35—H35B	0.9800
P3—C57	1.827 (5)	C35—H35C	0.9800
O1—C4	1.371 (6)	C36—C41	1.394 (7)
O1—C7	1.411 (6)	C36—C37	1.399 (6)
O2—C11	1.375 (6)	C37—C38	1.371 (6)
O2—C14	1.427 (8)	C37—H37	0.9500
O3—C18	1.361 (6)	C38—C39	1.386 (7)
O3—C21	1.436 (6)	C38—H38	0.9500
O4—C25	1.351 (6)	C39—C40	1.384 (7)
O4—C28	1.414 (7)	C40—C41	1.384 (7)
O5—C32	1.365 (6)	C40—H40	0.9500
O5—C35	1.423 (7)	C41—H41	0.9500
O6—C39	1.366 (6)	C42—H42A	0.9800
O6—C42	1.430 (6)	C42—H42B	0.9800
O7—C46	1.359 (6)	C42—H42C	0.9800
O7—C49	1.423 (6)	C43—C48	1.378 (6)
O8—C53	1.383 (6)	C43—C44	1.389 (7)
O8—C56	1.427 (6)	C44—C45	1.375 (7)
O9—C60	1.359 (6)	C44—H44	0.9500
O9—C63	1.422 (7)	C45—C46	1.386 (7)
O10—C64	1.107 (12)	C45—H45	0.9500
N1—C64	1.308 (12)	C46—C47	1.385 (7)
N1—C66	1.421 (10)	C47—C48	1.392 (7)
N1—C65	1.443 (10)	C47—H47	0.9500
C1—C2	1.390 (7)	C48—H48	0.9500
C1—C6	1.395 (6)	C49—H49A	0.9800
C2—C3	1.368 (7)	C49—H49B	0.9800
C2—H2	0.9500	C49—H49C	0.9800
C3—C4	1.380 (7)	C50—C55	1.386 (6)
C3—H3	0.9500	C50—C51	1.399 (7)

C4—C5	1.373 (7)	C51—C52	1.374 (7)
C5—C6	1.389 (7)	C51—H51	0.9500
C5—H5	0.9500	C52—C53	1.397 (7)
C6—H6	0.9500	C52—H52	0.9500
C7—H7A	0.9800	C53—C54	1.371 (7)
C7—H7B	0.9800	C54—C55	1.387 (7)
C7—H7C	0.9800	C54—H54	0.9500
C8—C13	1.380 (7)	C55—H55	0.9500
C8—C9	1.391 (7)	C56—H56A	0.9800
C9—C10	1.380 (7)	C56—H56B	0.9800
C9—H9	0.9500	C56—H56C	0.9800
C10—C11	1.377 (8)	C57—C62	1.385 (7)
C10—H10	0.9500	C57—C58	1.406 (7)
C11—C12	1.378 (7)	C58—C59	1.367 (7)
C12—C13	1.385 (7)	C58—H58	0.9500
C12—H12	0.9500	C59—C60	1.384 (7)
C13—H13	0.9500	C59—H59	0.9500
C14—H14A	0.9800	C60—C61	1.388 (7)
C14—H14B	0.9800	C61—C62	1.390 (7)
C14—H14C	0.9800	C61—H61	0.9500
C15—C16	1.379 (6)	C62—H62	0.9500
C15—C20	1.394 (7)	C63—H63A	0.9800
C16—C17	1.378 (7)	C63—H63B	0.9800
C16—H16	0.9500	C63—H63C	0.9800
C17—C18	1.393 (7)	C64—H64	0.9500
C17—H17	0.9500	C65—H65A	0.9800
C18—C19	1.381 (7)	C65—H65B	0.9800
C19—C20	1.375 (7)	C65—H65C	0.9800
C19—H19	0.9500	C66—H66A	0.9800
C20—H20	0.9500	C66—H66B	0.9800
C21—H21A	0.9800	C66—H66C	0.9800

S4—W1—S1	110.32 (5)	C22—C23—C24	121.7 (5)
S4—W1—S3	111.68 (5)	C22—C23—H23	119.2
S1—W1—S3	107.85 (4)	C24—C23—H23	119.2
S4—W1—S2	112.53 (5)	C25—C24—C23	120.5 (5)
S1—W1—S2	107.60 (4)	C25—C24—H24	119.7
S3—W1—S2	106.64 (4)	C23—C24—H24	119.7
Cu1—W1—Cu3	73.99 (2)	O4—C25—C26	125.7 (5)
Cu1—W1—Cu2	73.26 (2)	O4—C25—C24	115.8 (5)
Cu3—W1—Cu2	69.40 (2)	C26—C25—C24	118.4 (5)
Cu2—I1—Cu1	68.219 (19)	C25—C26—C27	120.7 (5)
Cu2—I1—Cu3	64.589 (19)	C25—C26—H26	119.6
Cu1—I1—Cu3	65.468 (19)	C27—C26—H26	119.6
P1—Cu1—S1	129.39 (5)	C26—C27—C22	121.3 (5)
P1—Cu1—S3	117.61 (5)	C26—C27—H27	119.3
S1—Cu1—S3	105.38 (5)	C22—C27—H27	119.3
P1—Cu1—W1	159.90 (4)	O4—C28—H28A	109.5

S1—Cu1—W1	52.88 (3)	O4—C28—H28B	109.5
S3—Cu1—W1	52.86 (3)	H28A—C28—H28B	109.5
P1—Cu1—I1	102.35 (4)	O4—C28—H28C	109.5
S1—Cu1—I1	94.84 (4)	H28A—C28—H28C	109.5
S3—Cu1—I1	99.94 (4)	H28B—C28—H28C	109.5
W1—Cu1—I1	97.05 (2)	C34—C29—C30	117.8 (4)
P2—Cu2—S2	119.96 (5)	C34—C29—P2	118.8 (4)
P2—Cu2—S1	116.90 (5)	C30—C29—P2	123.4 (4)
S2—Cu2—S1	102.45 (5)	C31—C30—C29	121.1 (5)
P2—Cu2—W1	149.55 (4)	C31—C30—H30	119.5
S2—Cu2—W1	52.16 (3)	C29—C30—H30	119.5
S1—Cu2—W1	51.71 (3)	C30—C31—C32	120.1 (5)
P2—Cu2—I1	108.72 (4)	C30—C31—H31	119.9
S2—Cu2—I1	107.10 (4)	C32—C31—H31	119.9
S1—Cu2—I1	99.48 (4)	O5—C32—C33	125.0 (5)
W1—Cu2—I1	101.39 (2)	O5—C32—C31	114.9 (5)
P3—Cu3—S3	120.23 (5)	C33—C32—C31	120.0 (5)
P3—Cu3—S2	121.70 (5)	C32—C33—C34	119.0 (5)
S3—Cu3—S2	103.66 (5)	C32—C33—H33	120.5
P3—Cu3—W1	155.95 (4)	C34—C33—H33	120.5
S3—Cu3—W1	52.36 (3)	C29—C34—C33	122.0 (5)
S2—Cu3—W1	52.55 (3)	C29—C34—H34	119.0
P3—Cu3—I1	108.41 (4)	C33—C34—H34	119.0
S3—Cu3—I1	98.83 (4)	O5—C35—H35A	109.5
S2—Cu3—I1	99.84 (4)	O5—C35—H35B	109.5
W1—Cu3—I1	95.61 (2)	H35A—C35—H35B	109.5
W1—S1—Cu1	73.12 (4)	O5—C35—H35C	109.5
W1—S1—Cu2	73.40 (4)	H35A—C35—H35C	109.5
Cu1—S1—Cu2	89.22 (5)	H35B—C35—H35C	109.5
W1—S2—Cu3	73.37 (4)	C41—C36—C37	117.7 (4)
W1—S2—Cu2	73.56 (4)	C41—C36—P2	123.2 (4)
Cu3—S2—Cu2	84.60 (5)	C37—C36—P2	119.2 (4)
W1—S3—Cu1	72.92 (4)	C38—C37—C36	121.2 (4)
W1—S3—Cu3	73.75 (4)	C38—C37—H37	119.4
Cu1—S3—Cu3	90.82 (5)	C36—C37—H37	119.4
C1—P1—C8	104.1 (2)	C37—C38—C39	120.4 (4)
C1—P1—C15	105.7 (2)	C37—C38—H38	119.8
C8—P1—C15	102.5 (2)	C39—C38—H38	119.8
C1—P1—Cu1	111.77 (16)	O6—C39—C40	124.7 (4)
C8—P1—Cu1	116.08 (16)	O6—C39—C38	115.8 (4)
C15—P1—Cu1	115.46 (16)	C40—C39—C38	119.5 (4)
C22—P2—C36	103.2 (2)	C41—C40—C39	120.0 (5)
C22—P2—C29	107.7 (2)	C41—C40—H40	120.0
C36—P2—C29	104.1 (2)	C39—C40—H40	120.0
C22—P2—Cu2	112.87 (16)	C40—C41—C36	121.2 (5)
C36—P2—Cu2	117.92 (15)	C40—C41—H41	119.4
C29—P2—Cu2	110.30 (15)	C36—C41—H41	119.4
C43—P3—C50	106.3 (2)	O6—C42—H42A	109.5

C43—P3—C57	101.1 (2)	O6—C42—H42B	109.5
C50—P3—C57	104.2 (2)	H42A—C42—H42B	109.5
C43—P3—Cu3	115.25 (16)	O6—C42—H42C	109.5
C50—P3—Cu3	115.59 (16)	H42A—C42—H42C	109.5
C57—P3—Cu3	112.84 (16)	H42B—C42—H42C	109.5
C4—O1—C7	117.8 (4)	C48—C43—C44	117.9 (4)
C11—O2—C14	117.3 (5)	C48—C43—P3	124.4 (4)
C18—O3—C21	117.4 (4)	C44—C43—P3	117.7 (4)
C25—O4—C28	118.8 (4)	C45—C44—C43	121.2 (5)
C32—O5—C35	117.4 (4)	C45—C44—H44	119.4
C39—O6—C42	117.4 (4)	C43—C44—H44	119.4
C46—O7—C49	118.3 (4)	C44—C45—C46	120.5 (5)
C53—O8—C56	116.9 (4)	C44—C45—H45	119.7
C60—O9—C63	118.7 (4)	C46—C45—H45	119.7
C64—N1—C66	123.9 (9)	O7—C46—C47	125.0 (4)
C64—N1—C65	116.2 (9)	O7—C46—C45	115.9 (4)
C66—N1—C65	119.9 (8)	C47—C46—C45	119.1 (4)
C2—C1—C6	117.7 (4)	C46—C47—C48	119.6 (5)
C2—C1—P1	118.9 (4)	C46—C47—H47	120.2
C6—C1—P1	123.4 (4)	C48—C47—H47	120.2
C3—C2—C1	121.2 (5)	C43—C48—C47	121.6 (4)
C3—C2—H2	119.4	C43—C48—H48	119.2
C1—C2—H2	119.4	C47—C48—H48	119.2
C2—C3—C4	120.5 (5)	O7—C49—H49A	109.5
C2—C3—H3	119.8	O7—C49—H49B	109.5
C4—C3—H3	119.8	H49A—C49—H49B	109.5
O1—C4—C5	124.8 (5)	O7—C49—H49C	109.5
O1—C4—C3	115.3 (5)	H49A—C49—H49C	109.5
C5—C4—C3	119.9 (4)	H49B—C49—H49C	109.5
C4—C5—C6	119.6 (5)	C55—C50—C51	117.7 (4)
C4—C5—H5	120.2	C55—C50—P3	119.2 (4)
C6—C5—H5	120.2	C51—C50—P3	123.0 (4)
C5—C6—C1	121.1 (5)	C52—C51—C50	121.5 (5)
C5—C6—H6	119.5	C52—C51—H51	119.3
C1—C6—H6	119.5	C50—C51—H51	119.3
O1—C7—H7A	109.5	C51—C52—C53	119.4 (5)
O1—C7—H7B	109.5	C51—C52—H52	120.3
H7A—C7—H7B	109.5	C53—C52—H52	120.3
O1—C7—H7C	109.5	C54—C53—O8	124.4 (5)
H7A—C7—H7C	109.5	C54—C53—C52	120.2 (5)
H7B—C7—H7C	109.5	O8—C53—C52	115.4 (5)
C13—C8—C9	118.3 (5)	C53—C54—C55	119.7 (5)
C13—C8—P1	121.9 (4)	C53—C54—H54	120.1
C9—C8—P1	119.8 (4)	C55—C54—H54	120.1
C10—C9—C8	120.2 (5)	C50—C55—C54	121.5 (5)
C10—C9—H9	119.9	C50—C55—H55	119.3
C8—C9—H9	119.9	C54—C55—H55	119.3
C11—C10—C9	120.5 (5)	O8—C56—H56A	109.5

C11—C10—H10	119.8	O8—C56—H56B	109.5
C9—C10—H10	119.8	H56A—C56—H56B	109.5
O2—C11—C10	115.7 (5)	O8—C56—H56C	109.5
O2—C11—C12	124.1 (5)	H56A—C56—H56C	109.5
C10—C11—C12	120.2 (5)	H56B—C56—H56C	109.5
C11—C12—C13	118.8 (5)	C62—C57—C58	118.3 (5)
C11—C12—H12	120.6	C62—C57—P3	120.0 (4)
C13—C12—H12	120.6	C58—C57—P3	121.7 (4)
C8—C13—C12	121.9 (5)	C59—C58—C57	120.5 (5)
C8—C13—H13	119.1	C59—C58—H58	119.8
C12—C13—H13	119.1	C57—C58—H58	119.8
O2—C14—H14A	109.5	C58—C59—C60	120.8 (5)
O2—C14—H14B	109.5	C58—C59—H59	119.6
H14A—C14—H14B	109.5	C60—C59—H59	119.6
O2—C14—H14C	109.5	O9—C60—C59	116.3 (5)
H14A—C14—H14C	109.5	O9—C60—C61	123.9 (5)
H14B—C14—H14C	109.5	C59—C60—C61	119.8 (5)
C16—C15—C20	118.0 (4)	C60—C61—C62	119.2 (5)
C16—C15—P1	124.3 (4)	C60—C61—H61	120.4
C20—C15—P1	117.6 (4)	C62—C61—H61	120.4
C17—C16—C15	121.5 (5)	C57—C62—C61	121.4 (5)
C17—C16—H16	119.2	C57—C62—H62	119.3
C15—C16—H16	119.2	C61—C62—H62	119.3
C16—C17—C18	119.8 (5)	O9—C63—H63A	109.5
C16—C17—H17	120.1	O9—C63—H63B	109.5
C18—C17—H17	120.1	H63A—C63—H63B	109.5
O3—C18—C19	125.4 (5)	O9—C63—H63C	109.5
O3—C18—C17	115.3 (4)	H63A—C63—H63C	109.5
C19—C18—C17	119.3 (5)	H63B—C63—H63C	109.5
C20—C19—C18	120.2 (5)	O10—C64—N1	121.9 (14)
C20—C19—H19	119.9	O10—C64—H64	119.0
C18—C19—H19	119.9	N1—C64—H64	119.0
C19—C20—C15	121.2 (5)	N1—C65—H65A	109.5
C19—C20—H20	119.4	N1—C65—H65B	109.5
C15—C20—H20	119.4	H65A—C65—H65B	109.5
O3—C21—H21A	109.5	N1—C65—H65C	109.5
O3—C21—H21B	109.5	H65A—C65—H65C	109.5
H21A—C21—H21B	109.5	H65B—C65—H65C	109.5
O3—C21—H21C	109.5	N1—C66—H66A	109.5
H21A—C21—H21C	109.5	N1—C66—H66B	109.5
H21B—C21—H21C	109.5	H66A—C66—H66B	109.5
C23—C22—C27	117.2 (5)	N1—C66—H66C	109.5
C23—C22—P2	119.5 (4)	H66A—C66—H66C	109.5
C27—C22—P2	123.2 (4)	H66B—C66—H66C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C64—H64···O10^{i}	0.95	2.62	3.126 (13)	114
C55—H55···I1	0.95	2.96	3.888 (5)	164
C49—H49*B*···I1^{ii}	0.98	3.30	3.864 (5)	119
C42—H42*B*···S2^{iii}	0.98	2.85	3.650 (5)	139
C38—H38···S4^{iv}	0.95	2.88	3.646 (5)	138
C34—H34···S2	0.95	2.96	3.860 (5)	158
C23—H23···S1	0.95	2.88	3.794 (5)	162
C17—H17···O2^{iv}	0.95	2.49	3.437 (7)	177
C9—H9···O7^{v}	0.95	2.51	3.273 (6)	137

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