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Mercury(II) halide complex of *cis*-[(*t*BuNH)(Se)P(μ -N^{*t*}Bu)₂P(Se)(NH^{*t*}Bu)]

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The mercury(II) halide complex [1,3-di-*tert*-butyl-2,4-bis(*tert*-butylamino)-1,3,2λ⁵,4λ⁵-diazadiphosphetidine-2,4-diselone-κ²Se,Se'] diiodidomercury(II) *N,N*-dimethylformamide monosolvate, [HgI₂(C₁₆H₃₈N₄P₂Se₂)]·C₃H₇NO or (**1**)-HgI₂, **2**, containing *cis*-[(*t*BuNH)(Se)P(μ -N^{*t*}Bu)₂P(Se)(NH^{*t*}Bu)] (**1**) was synthesized and structurally characterized. The crystal structure of **2** confirms the chelation of chalcogen donors to HgI₂ with a natural bite angle of 112.95 (2)°. The coordination geometry around mercury is distorted tetrahedral as indicated by the τ_4 geometry index parameter ($\tau_4 = 0.90$). In the mercury complex, the exocyclic *tert*-butylamido substituents are arranged in an (*endo*, *endo*) fashion, whereas in the free ligand (**1**), the exocyclic substituents are arranged in an (*exo*, *endo*) pattern. Compound **2** displays non-classical N—H···O hydrogen-bonding interactions with the solvent *N,N*-dimethylformamide. These interactions may introduce geometrical distortion and deviation from an ideal geometry. An isostructural HgBr₂ analogue containing *cis*-[(*t*BuNH)(S)P(μ -N^{*t*}Bu)₂P(S)(NH^{*t*}Bu)] was also synthesized and structurally characterized, CIF data for the compound being presented as supporting information.

1. Chemical context

Stable four-membered rings containing phosphorus and nitrogen with the general formula, [(*R*)P(μ -N^{*t*}Bu)₂P(*R*)] (*R* = alkyl or aryl), are commonly referred to as cyclodiphosphazanes. They have been used as building blocks to construct interesting macrocycles and polymers (Balakrishna, 2016). These macrocycles are formed by taking advantage of the *cis* orientation of the substituents and the lone pair available on the phosphorus atom (Balakrishna, 2016; Bashall *et al.*, 2002). The bis(amido)cyclodiphosphazane and its P^V analogue have been used as a versatile framework to stabilize main-group elements and transition metals (Stahl, 2000; Briand *et al.*, 2002). The bis(amido)cyclodiphosph(V)azane, *cis*-{[(*R*)NH](*E*)P(μ -N^{*t*}Bu)₂P(*E*)[NH(*R*)]} [*E* = O, S, Se, N(*R*); *R* = alkyl or aryl] and its di-anionic derivatives exhibit three unique coordination modes as shown in Fig. 1. These ligands

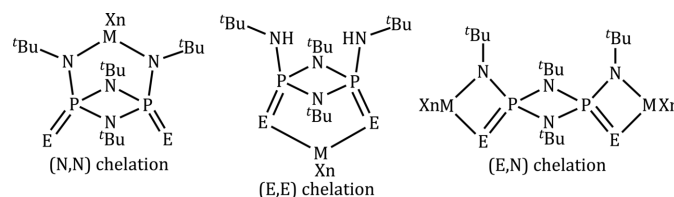
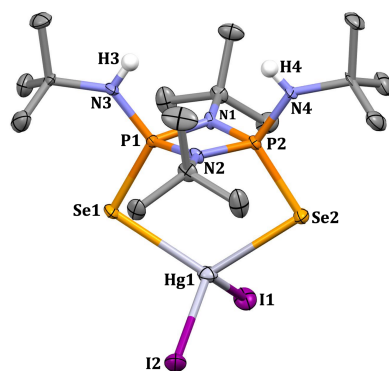
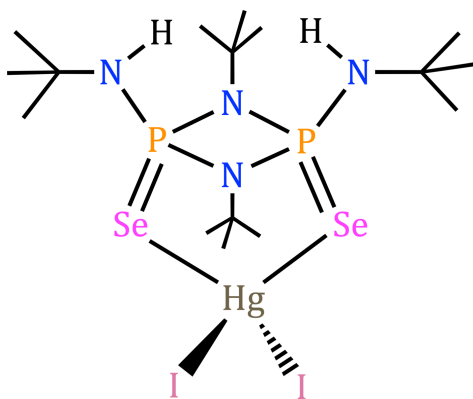


Figure 1
Possible coordination modes of bis(amido)cyclodiphosph(V)azanes.

are capable of bonding to metals and non-metals *via* (*N,N*), (*E,E*) or (*N,E*) chelation modes. The (*N,E*) chelation mode is the most frequently observed because of the rigidity and planarity of the four-membered P₂N₂ ring. More importantly, the (*N,N*) and (*E,E*) chelation modes demand large bite angles, and large-size metal ions are well suited for these coordination modes. In 2001, Chivers *et al.* (2001) reported (*S,S*) chelation of *cis*-[(^tBuN)(S)P(μ-N^tBu)₂P(S)(N^tBu)] to the Pt^{II} center with a bite angle of 99.57 (13)°. Recently, we have reported (*Se,Se*) chelation of *cis*-[(^tBuNH)(Se)P(μ-N^tBu)₂P(Se)(NH^tBu)] by a Pd^{II} complex with a bite angle of 110.54 (1)° (Bonnette *et al.*, 2018). It is evident from these examples that the (*E,E*) coordination mode prefers large metal cations. Mercury ions have been well documented to have an affinity towards sulfur and selenium atoms, and accounting for its larger size, we set out to explore the coordination chemistry of bis(amido)cyclodiphosph(V)azane ligands with mercury(II) halide. Herein, we report the synthesis and solid-state structure of an HgI₂ coordination complex with *cis*-[(^tBuNH)(Se)P(μ-N^tBu)₂P(Se)(NH^tBu)] (**1**), and the results are presented below. An isostructural HgBr₂ analogue was also synthesized and structurally characterized. The CIF data for the compound are presented as supporting information.



2. Structural commentary

Compound **2** crystallizes in the monoclinic crystal system in space group *P2₁/n*. The molecular structure of **2** is illustrated in Fig. 2. The crystal structure confirms the chelation of **1** through selenium donors to stabilize the HgI₂ moiety, with an Se1–Hg1–Se2 natural bite angle of 112.95 (2)°. The coordination geometry around the mercury atom is distorted tetrahedral, as indicated by the parameter $\tau_4 = 0.90$. The geometry index τ_4 was developed by Okuniewski and co-workers to distinguish various four-coordinate geometries (Okuniewski *et al.*, 2015; Yang *et al.*, 2007) with $\tau_4 = 0$ for a square-planar geometry, 0.24 for seesaw, and 1 for a tetrahedral geometry. The Hg–Se [Hg1–Se1 = 2.7508 (5) Å; Hg1–Se2 = 2.7835 (6) Å], and Hg–I [Hg1–I1 = 2.7290 (4) Å; Hg1–I2 = 2.7409 (4) Å] bond distances are within the typical ranges reported for the HgI₂ complexes with selenium ligands (Palmer & Parkin, 2015). In complex **2**, the P1–Se1 and

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

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
N3–H3···O1	0.82 (6)	2.18 (6)	2.986 (6)	165 (4)
N4–H4···O1	0.79 (5)	2.27 (5)	3.051 (5)	169 (6)

P2–Se2 bonds [2.1260 (13) and 2.1302 (12) Å, respectively] are slightly elongated compared to the P–Se bond [2.078 (1) Å] in the uncoordinated ligand **1**. The four-membered P₂N₂ ring in complex **2** is slightly puckered, as indicated by the angle subtended by the planes N1/P1/N2 and N1/P2/N2 [8.7 (3)°]. The corresponding dihedral angle for the uncoordinated ligand is 3.73 (2)° (Hill *et al.*, 1994).

3. Supramolecular features

In the crystal of **2**, the N–H functional groups present in bis(*tert*-butylamido)cyclodiphosph(V)azane and oxygen from the DMF solvent molecule are involved in N–H···O hydrogen-bonding interactions (Fig. 3, Table 1). Three different conformational isomers are feasible for the *cis*-bis-(amido)cyclodiphosph(V)azane with respect to the relative orientations of the exocyclic nitrogen substituents (Fig. 4). In **2**, the exocyclic substituents are arranged in a (*endo, endo*) fashion, whereas in ligand **1** they are arranged in an (*exo, endo*) orientation (Hill *et al.*, 1994; Chivers *et al.*, 2002). The conformational change in the coordination sphere of **2** may result from the formation of intermolecular interactions. A similar conformational change influenced by hydrogen-bonding interactions has been previously reported (Chandrasekaran *et al.*, 2011).

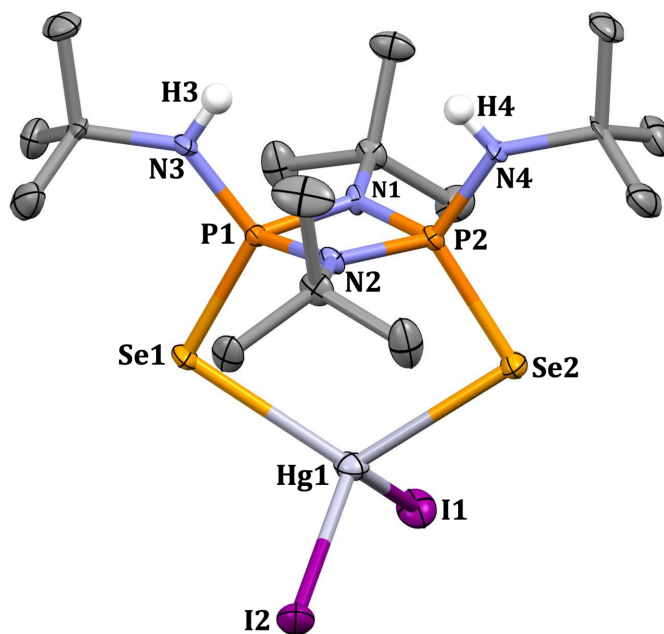


Figure 2
Displacement ellipsoid plot for **2** (50% probability level). The DMF solvent molecule and all the hydrogen atoms are omitted for clarity, except for those at N3 and N4.

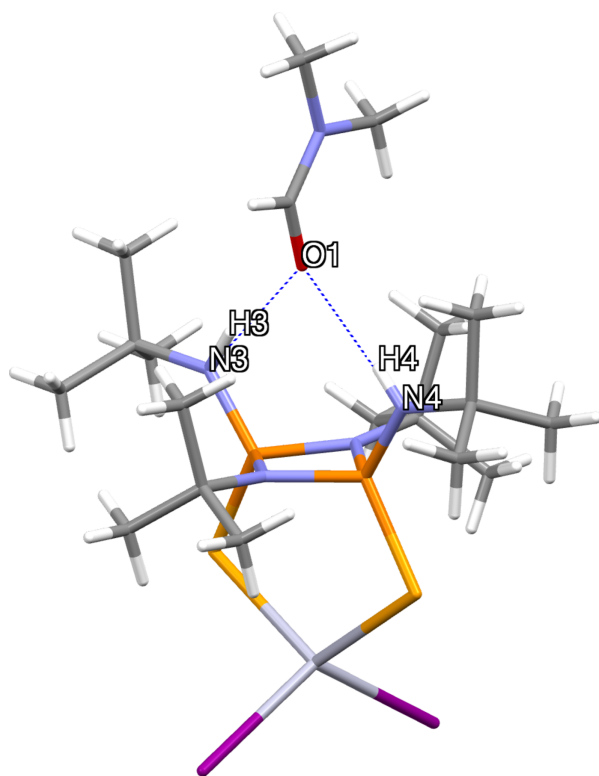


Figure 3
Hydrogen-bonding interactions (Table 1) between the complex and the DMF solvent molecule in the crystal.

4. Database survey

A search of the Cambridge Structural Database (CSD Data, March 2024; Groom *et al.*, 2016) gave the following hits for *cis*-{[(*R*)_{*n*}CN](Se)P[(*R*)_{*n*}N]₂P(Se)[NC(*R*)_{*n*}]}: dichlorido[1,3-di-*tert*-butyl-2,4-bis(*tert*-butylamino)-1,3,2,4-diazadiphosphetidine-2,4-diselone-Se,Se']palladium(II) (Bonnette *et al.*, 2018; CCDC No. 1549758), bis[μ-(2,4-bis(*tert*-butylamido)-1,3-bis(*tert*-butyl)-2,4-diselono-2,4-diphosphetidine)]hexakis(tetrahydrofuran)tetrapotassium, (Chivers *et al.*, 2001; CCDC No. 142628), bis-*N,N'*,1,3-tetra-*tert*-butyl-1,3,2,4-diazadiphosphetidine-2,4-diamine 2,4-bis(selenide)]silver(I) trifluoromethanesulfonate (Knight & Woollins, 2016; CCDC No. 1042705) and [1,3-di-*tert*-butyl-2,4-bis(*tert*-butylamino)-1,3,2,4-diazadiphosphetidine-2,4-diselone]bis(triphenylphosphine)palladium bis(tetrafluoroborate) dichloromethane solvate (Plajer *et al.*, 2020; CCDC No. 1890520). The P=Se and P–N bond distances for **2** are in agreement with those in the above compounds.

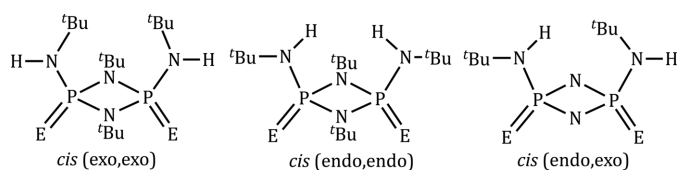


Figure 4
Possible conformational isomers for *cis*-bis(amido)cyclodiphosph(V) azanes.

5. Synthesis and crystallization

Synthesis of *cis*-[HgI₂(**1**)] (**2**):

A dichloromethane (10 mL) solution of *cis*-[(*t*-BuHN)(Se)P(μ-*t*-BuN)₂P(Se)(NH*t*-Bu)] (**1**) (100 mg, 0.197 mmol) was added dropwise over an acetonitrile (5 mL) solution of HgI₂ (88 mg; 0.197 mmol) under an N₂ atmosphere at ambient temperature. The resulting reaction mixture was stirred for 4 h at 295 K. The solution was then concentrated to nearly 5 mL and stored at 248 K for a day to afford an analytically pure white microcrystalline product. Yield: 91% (172 mg). X-ray quality crystals are obtained by slow evaporation from a DMF solution at room temperature, m.p. 465–467 K. ¹H NMR (400 MHz, DMSO-*d*₆): 1.44 (*s*, 18H, *t*-Bu), 1.58 (*s*, 18H, *t*-Bu), 2.58 (*br s*, 2H, NH). IR (cm⁻¹): 3200 (*br w*), 2975 (*w*), 1462 (*w*), 1388 (*w*), 1366 (*m*), 1222 (*w*), 1183 (*m*), 1030 (*vs*), 899 (*s*), 851 (*w*), 731 (*m*), 678 (*m*). Absorption spectrum [DMSO; λ_{max}, nm (ε_M, M⁻¹ cm⁻¹): 270 (13947). Analysis calculated for C₁₆H₃₈N₄P₂Se₂HgI₂: C, 20.00; H, 3.99; N, 5.83. Found: C, 20.26; H, 4.47; N, 5.98.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Methyl (CH₃) hydrogen atoms were treated as a rotating group and added using the riding-model approximation to the carbon atom to which they are attached [C–H = 0.98 Å with *U*_{iso}(H) = 1.5*U*_{eq}(CH₃).

7. Data for isostructural HgBr₂ complex

Synthesis and spectroscopic data for an isostructural HgBr₂ with *cis*-[(*t*-BuHN)(S)P(μ-*t*-BuN)₂P(S)(NH*t*-Bu)] are presented below. Spectroscopic analysis and single-crystal structure determination strongly support these are isostructural complexes. For more information regarding solid-state structure determination, please refer to CCDC: 2380829. The CIF data for this compound is available in the supporting information

A dichloromethane (10 mL) solution of *cis*-[(*t*-BuHN)(S)P(μ-*t*-BuN)₂P(S)(NH*t*-Bu)] (**1**) (100 mg, 0.24 mmol) was added dropwise over an acetonitrile (5 mL) solution of HgBr₂ (87.4 mg; 0.24 mmol) under an N₂ atmosphere at ambient temperature. The resulting reaction mixture was stirred for 4 h at 295 K. The solution was then concentrated to nearly 5 mL and stored at 248 K for a day to afford an analytically pure white microcrystalline product. Yield: 83% (156 mg). X-ray quality crystals were obtained by slow evaporation from DMF solution at room temperature, m.p. 483–485 K. ¹H NMR (400 MHz, DMSO-*d*₆): 1.45 (*s*, 18H, *t*-Bu), 1.59 (*s*, 18H, *t*-Bu), 2.54 (*br s*, 2H, NH). IR (cm⁻¹): 3194 (*br m*; N-H), 2977 (*w*), 1471 (*w*), 1391 (*w*), 1369 (*m*), 1225 (*w*), 1185 (*s*), 1046 (*vs*), 907 (*m*), 852 (*m*), 745 (*s*), 705 (*m*). Absorption spectrum [DMSO; λ_{max}, nm (ε_M, M⁻¹ cm⁻¹): 282 (17054). Analysis calculated for C₁₆H₃₈N₄P₂S₂HgBr₂: C, 24.86; H, 4.95; N, 7.25; S, 8.30. Found: C, 25.03; H, 5.08; N, 7.75; S, 8.22.

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Table 2

Experimental details.

Crystal data	
Chemical formula	[HgI ₂ (C ₁₆ H ₃₈ N ₄ P ₂ Se ₂)]·C ₃ H ₇ NO
<i>M_r</i>	1033.85
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁ / <i>n</i>
Temperature (K)	300
<i>a</i> , <i>b</i> , <i>c</i> (Å)	9.1747 (8), 17.3698 (13), 20.841 (2)
β (°)	101.049 (3)
<i>V</i> (Å ³)	3259.7 (5)
<i>Z</i>	4
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	8.97
Crystal size (mm)	0.53 × 0.28 × 0.26
Data collection	
Diffractometer	Bruker <i>SMART</i> X2S
Absorption correction	Multi-scan (<i>SADABS</i> ; Krause <i>et al.</i> , 2015)
<i>T</i> _{min} , <i>T</i> _{max}	0.309, 0.746
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	45600, 9576, 6956
<i>R</i> _{int}	0.077
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.725
Refinement	
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.040, 0.089, 1.00
No. of reflections	9576
No. of parameters	310
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	1.91, -1.92

Computer programs: *APEX2* and *SAINT* (Bruker, 2016), *SHELXT2018/2* (Sheldrick, 2015a), *SHELXL2019/1* (Sheldrick, 2015b), *Mercury* (Macrae *et al.*, 2020) and *OLEX2* (Dolomanov *et al.*, 2009).

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Mercury(II) halide complex of *cis*-[(*t*BuNH)(Se)P(μ -N^{*t*}Bu)₂P(Se)(NH^{*t*}Bu)]

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Computing details

[1,3-Di-*tert*-butyl-2,4-bis(*tert*-butylamino)-1,3,2 λ^5 ,4 λ^5 -diazadiphosphetidine-2,4-diselone- κ^2 Se,Se']diiodidomercury(II) *N,N*-dimethylformamide monosolvate

Crystal data

[HgI₂(C₁₆H₃₈N₄P₂Se₂)]·C₃H₇NO

$M_r = 1033.85$

Monoclinic, $P2_1/n$

$a = 9.1747$ (8) Å

$b = 17.3698$ (13) Å

$c = 20.841$ (2) Å

$\beta = 101.049$ (3)°

$V = 3259.7$ (5) Å³

$Z = 4$

$F(000) = 1944$

$D_x = 2.107$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 8829 reflections

$\theta = 2.3$ – 26.8 °

$\mu = 8.97$ mm⁻¹

$T = 300$ K

Prism, colorless

$0.53 \times 0.28 \times 0.26$ mm

Data collection

Bruker SMART X2S
diffractometer

ω scans

Absorption correction: multi-scan
(SADABS; Krause *et al.*, 2015)

$T_{\min} = 0.309$, $T_{\max} = 0.746$

45600 measured reflections

9576 independent reflections

6956 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.077$

$\theta_{\max} = 31.0$ °, $\theta_{\min} = 2.6$ °

$h = -12 \rightarrow 13$

$k = -24 \rightarrow 19$

$l = -28 \rightarrow 29$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.040$

$wR(F^2) = 0.089$

$S = 1.00$

9576 reflections

310 parameters

0 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0325P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.004$

$\Delta\rho_{\max} = 1.91$ e Å⁻³

$\Delta\rho_{\min} = -1.91$ e Å⁻³

Extinction correction: *SHELXL2019/1*

(Sheldrick, 2015b),

$F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.00052 (5)

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Hg1	0.70533 (2)	0.77295 (2)	0.58150 (2)	0.02178 (6)
I1	0.60217 (4)	0.91950 (2)	0.55729 (2)	0.02891 (9)
I2	0.90720 (4)	0.72186 (2)	0.50978 (2)	0.02938 (9)
Se1	0.49422 (5)	0.65923 (3)	0.55275 (2)	0.01821 (11)
Se2	0.81087 (5)	0.79005 (3)	0.71512 (2)	0.01874 (11)
P1	0.51042 (12)	0.62623 (6)	0.65210 (6)	0.0119 (2)
P2	0.67826 (12)	0.69810 (6)	0.73987 (6)	0.0120 (2)
N2	0.6846 (4)	0.61881 (19)	0.69491 (18)	0.0131 (8)
N1	0.4974 (4)	0.6991 (2)	0.70322 (19)	0.0127 (7)
O1	0.4763 (4)	0.5489 (2)	0.80940 (19)	0.0317 (9)
N3	0.4072 (4)	0.5541 (2)	0.6635 (2)	0.0164 (8)
H3	0.413 (5)	0.547 (3)	0.703 (3)	0.020*
N4	0.7002 (4)	0.6801 (2)	0.81733 (19)	0.0158 (8)
H4	0.649 (5)	0.644 (3)	0.820 (3)	0.019*
N5	0.3773 (5)	0.4941 (2)	0.8902 (2)	0.0274 (10)
C5	0.8046 (5)	0.5593 (3)	0.6959 (3)	0.0193 (10)
C6	0.7986 (5)	0.5287 (3)	0.6268 (3)	0.0243 (11)
H6A	0.704074	0.504766	0.611382	0.036*
H6B	0.876292	0.491634	0.627173	0.036*
H6C	0.811614	0.570583	0.598316	0.036*
C11	0.4061 (6)	0.4524 (3)	0.5785 (3)	0.0333 (14)
H11A	0.450455	0.488033	0.552709	0.050*
H11B	0.343410	0.417284	0.550175	0.050*
H11C	0.482767	0.424099	0.606659	0.050*
C9	0.3141 (5)	0.4963 (2)	0.6196 (2)	0.0181 (10)
C1	0.3696 (5)	0.7508 (3)	0.7113 (2)	0.0163 (9)
C10	0.1830 (6)	0.5362 (3)	0.5759 (3)	0.0323 (13)
H10A	0.124250	0.562406	0.602559	0.048*
H10B	0.122930	0.498552	0.549292	0.048*
H10C	0.219481	0.572836	0.548227	0.048*
C13	0.7805 (5)	0.7175 (3)	0.8783 (2)	0.0187 (10)
C4	0.4225 (5)	0.8335 (3)	0.7189 (3)	0.0261 (12)
H4A	0.493585	0.838807	0.758917	0.039*
H4B	0.339431	0.866754	0.719846	0.039*
H4C	0.468055	0.847230	0.682688	0.039*
C7	0.9542 (5)	0.5971 (3)	0.7207 (3)	0.0355 (14)
H7A	0.969762	0.637485	0.691327	0.053*
H7B	1.031512	0.559332	0.723061	0.053*
H7C	0.955868	0.618207	0.763393	0.053*

C15	0.9469 (5)	0.7146 (3)	0.8823 (3)	0.0311 (13)
H15A	0.978069	0.662043	0.880656	0.047*
H15B	0.995907	0.737546	0.922644	0.047*
H15C	0.972166	0.742541	0.846240	0.047*
C8	0.7780 (7)	0.4949 (3)	0.7420 (3)	0.0422 (16)
H8A	0.783163	0.515386	0.785149	0.063*
H8B	0.852543	0.455882	0.743044	0.063*
H8C	0.681607	0.472865	0.726814	0.063*
C3	0.2467 (5)	0.7432 (3)	0.6512 (3)	0.0314 (13)
H3A	0.282388	0.760832	0.613343	0.047*
H3B	0.163030	0.773861	0.656945	0.047*
H3C	0.217140	0.690297	0.645353	0.047*
C12	0.2550 (6)	0.4405 (3)	0.6658 (3)	0.0279 (12)
H12A	0.336981	0.416839	0.694597	0.042*
H12B	0.195746	0.401487	0.640602	0.042*
H12C	0.195348	0.468331	0.691109	0.042*
C16	0.7383 (6)	0.6705 (3)	0.9345 (2)	0.0261 (11)
H16A	0.632584	0.671853	0.931349	0.039*
H16B	0.785992	0.692134	0.975582	0.039*
H16C	0.770054	0.618135	0.931728	0.039*
C19	0.3824 (7)	0.4343 (3)	0.9396 (3)	0.0422 (16)
H19A	0.291802	0.405187	0.930901	0.063*
H19B	0.393780	0.457611	0.982021	0.063*
H19C	0.464897	0.400734	0.938354	0.063*
C14	0.7257 (6)	0.8005 (3)	0.8827 (3)	0.0302 (12)
H14A	0.748134	0.829921	0.846829	0.045*
H14B	0.774377	0.823051	0.923183	0.045*
H14C	0.620304	0.800291	0.880742	0.045*
C17	0.4744 (6)	0.4989 (3)	0.8515 (3)	0.0310 (13)
H17	0.548158	0.461504	0.856240	0.037*
C2	0.3118 (6)	0.7242 (3)	0.7719 (3)	0.0269 (12)
H2A	0.281575	0.671275	0.766623	0.040*
H2B	0.228388	0.755317	0.777098	0.040*
H2C	0.389085	0.729280	0.809801	0.040*
C18	0.2577 (8)	0.5492 (4)	0.8855 (4)	0.072 (3)
H18A	0.164600	0.523685	0.870597	0.108*
H18B	0.270631	0.588982	0.855095	0.108*
H18C	0.258555	0.571560	0.927697	0.108*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Hg1	0.02744 (11)	0.02015 (11)	0.01887 (11)	-0.00093 (7)	0.00728 (8)	0.00364 (8)
I1	0.0387 (2)	0.02087 (18)	0.0257 (2)	0.00404 (14)	0.00268 (15)	0.00612 (14)
I2	0.02607 (17)	0.0364 (2)	0.0285 (2)	-0.00545 (14)	0.01224 (15)	-0.00543 (16)
Se1	0.0244 (2)	0.0215 (2)	0.0084 (2)	-0.00507 (19)	0.00213 (18)	0.00068 (19)
Se2	0.0234 (2)	0.0186 (2)	0.0138 (2)	-0.00661 (18)	0.00253 (19)	0.00001 (19)
P1	0.0155 (5)	0.0115 (5)	0.0088 (6)	-0.0001 (4)	0.0026 (4)	-0.0007 (4)

P2	0.0137 (5)	0.0134 (6)	0.0084 (6)	0.0007 (4)	0.0011 (4)	-0.0003 (5)
N2	0.0138 (17)	0.0145 (19)	0.0106 (19)	0.0040 (14)	0.0015 (15)	-0.0008 (15)
N1	0.0149 (17)	0.0106 (17)	0.012 (2)	0.0012 (14)	0.0005 (15)	-0.0037 (15)
O1	0.040 (2)	0.033 (2)	0.025 (2)	-0.0009 (17)	0.0146 (18)	0.0076 (17)
N3	0.025 (2)	0.016 (2)	0.009 (2)	-0.0055 (16)	0.0064 (17)	-0.0010 (16)
N4	0.023 (2)	0.019 (2)	0.0047 (19)	-0.0072 (16)	0.0006 (15)	0.0008 (16)
N5	0.039 (3)	0.026 (2)	0.018 (2)	-0.0017 (19)	0.008 (2)	0.0066 (19)
C5	0.020 (2)	0.016 (2)	0.023 (3)	0.0082 (18)	0.006 (2)	-0.001 (2)
C6	0.019 (2)	0.025 (3)	0.031 (3)	0.003 (2)	0.010 (2)	-0.013 (2)
C11	0.036 (3)	0.027 (3)	0.041 (4)	-0.010 (2)	0.018 (3)	-0.014 (3)
C9	0.021 (2)	0.014 (2)	0.021 (3)	-0.0110 (18)	0.010 (2)	-0.006 (2)
C1	0.017 (2)	0.015 (2)	0.018 (3)	0.0061 (18)	0.0039 (19)	-0.0005 (19)
C10	0.029 (3)	0.038 (3)	0.029 (3)	-0.013 (2)	0.000 (2)	-0.006 (3)
C13	0.024 (2)	0.028 (3)	0.004 (2)	-0.001 (2)	0.0003 (18)	-0.0053 (19)
C4	0.026 (3)	0.016 (2)	0.039 (4)	0.005 (2)	0.012 (2)	-0.001 (2)
C7	0.023 (3)	0.044 (3)	0.037 (4)	0.007 (2)	-0.001 (2)	-0.017 (3)
C15	0.028 (3)	0.050 (3)	0.015 (3)	-0.003 (2)	0.002 (2)	-0.001 (2)
C8	0.052 (4)	0.031 (3)	0.049 (4)	0.028 (3)	0.023 (3)	0.023 (3)
C3	0.023 (3)	0.036 (3)	0.032 (3)	0.013 (2)	-0.003 (2)	-0.010 (3)
C12	0.037 (3)	0.019 (3)	0.032 (3)	-0.008 (2)	0.017 (3)	0.002 (2)
C16	0.037 (3)	0.031 (3)	0.010 (3)	-0.001 (2)	0.005 (2)	0.003 (2)
C19	0.068 (4)	0.036 (3)	0.022 (3)	-0.008 (3)	0.007 (3)	0.012 (3)
C14	0.043 (3)	0.029 (3)	0.017 (3)	0.000 (2)	0.001 (2)	-0.004 (2)
C17	0.027 (3)	0.034 (3)	0.032 (3)	0.007 (2)	0.007 (2)	0.006 (3)
C2	0.028 (3)	0.029 (3)	0.027 (3)	0.009 (2)	0.013 (2)	0.005 (2)
C18	0.077 (5)	0.085 (6)	0.069 (6)	0.043 (4)	0.050 (5)	0.041 (5)

Geometric parameters (Å, °)

Hg1—I1	2.7290 (4)	C10—H10B	0.9600
Hg1—I2	2.7409 (4)	C10—H10C	0.9600
Hg1—Se1	2.7508 (5)	C13—C15	1.514 (6)
Hg1—Se2	2.7835 (6)	C13—C16	1.536 (7)
Se1—P1	2.1260 (13)	C13—C14	1.535 (7)
Se2—P2	2.1302 (12)	C4—H4A	0.9600
P1—P2	2.4893 (16)	C4—H4B	0.9600
P1—N2	1.679 (4)	C4—H4C	0.9600
P1—N1	1.674 (4)	C7—H7A	0.9600
P1—N3	1.615 (4)	C7—H7B	0.9600
P2—N2	1.673 (4)	C7—H7C	0.9600
P2—N1	1.689 (4)	C15—H15A	0.9600
P2—N4	1.619 (4)	C15—H15B	0.9600
N2—C5	1.507 (5)	C15—H15C	0.9600
N1—C1	1.511 (5)	C8—H8A	0.9600
O1—C17	1.237 (6)	C8—H8B	0.9600
N3—H3	0.83 (5)	C8—H8C	0.9600
N3—C9	1.508 (6)	C3—H3A	0.9600
N4—H4	0.79 (5)	C3—H3B	0.9600

N4—C13	1.491 (6)	C3—H3C	0.9600
N5—C19	1.457 (6)	C12—H12A	0.9600
N5—C17	1.314 (7)	C12—H12B	0.9600
N5—C18	1.444 (7)	C12—H12C	0.9600
C5—C6	1.526 (7)	C16—H16A	0.9600
C5—C7	1.520 (7)	C16—H16B	0.9600
C5—C8	1.525 (7)	C16—H16C	0.9600
C6—H6A	0.9600	C19—H19A	0.9600
C6—H6B	0.9600	C19—H19B	0.9600
C6—H6C	0.9600	C19—H19C	0.9600
C11—H11A	0.9600	C14—H14A	0.9600
C11—H11B	0.9600	C14—H14B	0.9600
C11—H11C	0.9600	C14—H14C	0.9600
C11—C9	1.519 (6)	C17—H17	0.9300
C9—C10	1.530 (7)	C2—H2A	0.9600
C9—C12	1.536 (6)	C2—H2B	0.9600
C1—C4	1.515 (6)	C2—H2C	0.9600
C1—C3	1.522 (7)	C18—H18A	0.9600
C1—C2	1.531 (7)	C18—H18B	0.9600
C10—H10A	0.9600	C18—H18C	0.9600
I1—Hg1—I2	116.603 (13)	H10A—C10—H10C	109.5
I1—Hg1—Se1	115.134 (15)	H10B—C10—H10C	109.5
I1—Hg1—Se2	97.310 (14)	N4—C13—C15	111.2 (4)
I2—Hg1—Se1	99.784 (14)	N4—C13—C16	105.3 (4)
I2—Hg1—Se2	115.997 (16)	N4—C13—C14	110.2 (4)
Se1—Hg1—Se2	112.950 (15)	C15—C13—C16	109.9 (4)
P1—Se1—Hg1	93.82 (3)	C15—C13—C14	111.4 (4)
P2—Se2—Hg1	93.27 (4)	C14—C13—C16	108.7 (4)
Se1—P1—P2	119.89 (6)	C1—C4—H4A	109.5
N2—P1—Se1	114.83 (14)	C1—C4—H4B	109.5
N2—P1—P2	41.95 (12)	C1—C4—H4C	109.5
N1—P1—Se1	114.57 (14)	H4A—C4—H4B	109.5
N1—P1—P2	42.48 (12)	H4A—C4—H4C	109.5
N1—P1—N2	84.07 (18)	H4B—C4—H4C	109.5
N3—P1—Se1	114.75 (16)	C5—C7—H7A	109.5
N3—P1—P2	125.36 (16)	C5—C7—H7B	109.5
N3—P1—N2	112.8 (2)	C5—C7—H7C	109.5
N3—P1—N1	112.2 (2)	H7A—C7—H7B	109.5
Se2—P2—P1	119.98 (6)	H7A—C7—H7C	109.5
N2—P2—Se2	113.51 (14)	H7B—C7—H7C	109.5
N2—P2—P1	42.15 (12)	C13—C15—H15A	109.5
N2—P2—N1	83.79 (17)	C13—C15—H15B	109.5
N1—P2—Se2	116.10 (14)	C13—C15—H15C	109.5
N1—P2—P1	42.01 (12)	H15A—C15—H15B	109.5
N4—P2—Se2	114.77 (15)	H15A—C15—H15C	109.5
N4—P2—P1	125.25 (15)	H15B—C15—H15C	109.5
N4—P2—N2	112.8 (2)	C5—C8—H8A	109.5

N4—P2—N1	112.1 (2)	C5—C8—H8B	109.5
P2—N2—P1	95.90 (17)	C5—C8—H8C	109.5
C5—N2—P1	132.7 (3)	H8A—C8—H8B	109.5
C5—N2—P2	131.3 (3)	H8A—C8—H8C	109.5
P1—N1—P2	95.51 (18)	H8B—C8—H8C	109.5
C1—N1—P1	132.2 (3)	C1—C3—H3A	109.5
C1—N1—P2	132.2 (3)	C1—C3—H3B	109.5
P1—N3—H3	110 (3)	C1—C3—H3C	109.5
C9—N3—P1	135.0 (3)	H3A—C3—H3B	109.5
C9—N3—H3	115 (3)	H3A—C3—H3C	109.5
P2—N4—H4	105 (4)	H3B—C3—H3C	109.5
C13—N4—P2	135.5 (3)	C9—C12—H12A	109.5
C13—N4—H4	119 (4)	C9—C12—H12B	109.5
C17—N5—C19	123.0 (5)	C9—C12—H12C	109.5
C17—N5—C18	120.8 (5)	H12A—C12—H12B	109.5
C18—N5—C19	116.2 (5)	H12A—C12—H12C	109.5
N2—C5—C6	109.4 (4)	H12B—C12—H12C	109.5
N2—C5—C7	108.6 (4)	C13—C16—H16A	109.5
N2—C5—C8	107.9 (4)	C13—C16—H16B	109.5
C7—C5—C6	109.8 (4)	C13—C16—H16C	109.5
C7—C5—C8	110.2 (5)	H16A—C16—H16B	109.5
C8—C5—C6	111.0 (4)	H16A—C16—H16C	109.5
C5—C6—H6A	109.5	H16B—C16—H16C	109.5
C5—C6—H6B	109.5	N5—C19—H19A	109.5
C5—C6—H6C	109.5	N5—C19—H19B	109.5
H6A—C6—H6B	109.5	N5—C19—H19C	109.5
H6A—C6—H6C	109.5	H19A—C19—H19B	109.5
H6B—C6—H6C	109.5	H19A—C19—H19C	109.5
H11A—C11—H11B	109.5	H19B—C19—H19C	109.5
H11A—C11—H11C	109.5	C13—C14—H14A	109.5
H11B—C11—H11C	109.5	C13—C14—H14B	109.5
C9—C11—H11A	109.5	C13—C14—H14C	109.5
C9—C11—H11B	109.5	H14A—C14—H14B	109.5
C9—C11—H11C	109.5	H14A—C14—H14C	109.5
N3—C9—C11	111.4 (4)	H14B—C14—H14C	109.5
N3—C9—C10	110.4 (4)	O1—C17—N5	125.7 (5)
N3—C9—C12	105.5 (4)	O1—C17—H17	117.2
C11—C9—C10	110.5 (5)	N5—C17—H17	117.2
C11—C9—C12	109.8 (4)	C1—C2—H2A	109.5
C10—C9—C12	109.2 (4)	C1—C2—H2B	109.5
N1—C1—C4	109.5 (4)	C1—C2—H2C	109.5
N1—C1—C3	109.0 (4)	H2A—C2—H2B	109.5
N1—C1—C2	108.1 (4)	H2A—C2—H2C	109.5
C4—C1—C3	109.9 (4)	H2B—C2—H2C	109.5
C4—C1—C2	110.8 (4)	N5—C18—H18A	109.5
C3—C1—C2	109.5 (4)	N5—C18—H18B	109.5
C9—C10—H10A	109.5	N5—C18—H18C	109.5
C9—C10—H10B	109.5	H18A—C18—H18B	109.5

C9—C10—H10C	109.5	H18A—C18—H18C	109.5
H10A—C10—H10B	109.5	H18B—C18—H18C	109.5
Se1—P1—N2—P2	107.86 (15)	P2—N1—C1—C4	40.6 (6)
Se1—P1—N2—C5	-75.0 (4)	P2—N1—C1—C3	160.8 (4)
Se1—P1—N1—P2	-108.19 (15)	P2—N1—C1—C2	-80.2 (5)
Se1—P1—N1—C1	69.0 (4)	P2—N4—C13—C15	-66.1 (6)
Se1—P1—N3—C9	9.6 (5)	P2—N4—C13—C16	175.0 (4)
Se2—P2—N2—P1	-109.36 (14)	P2—N4—C13—C14	58.0 (6)
Se2—P2—N2—C5	73.5 (4)	N2—P1—N1—P2	6.44 (18)
Se2—P2—N1—P1	106.70 (14)	N2—P1—N1—C1	-176.4 (4)
Se2—P2—N1—C1	-70.4 (4)	N2—P1—N3—C9	-124.4 (4)
Se2—P2—N4—C13	7.9 (5)	N2—P2—N1—P1	-6.47 (19)
P1—P2—N2—C5	-177.2 (5)	N2—P2—N1—C1	176.4 (4)
P1—P2—N1—C1	-177.1 (5)	N2—P2—N4—C13	140.0 (4)
P1—P2—N4—C13	-173.4 (4)	N1—P1—N2—P2	-6.51 (19)
P1—N2—C5—C6	36.4 (6)	N1—P1—N2—C5	170.6 (4)
P1—N2—C5—C7	156.1 (4)	N1—P1—N3—C9	142.7 (4)
P1—N2—C5—C8	-84.5 (5)	N1—P2—N2—P1	6.45 (19)
P1—N1—C1—C4	-135.6 (4)	N1—P2—N2—C5	-170.7 (4)
P1—N1—C1—C3	-15.3 (6)	N1—P2—N4—C13	-127.4 (4)
P1—N1—C1—C2	103.6 (5)	N3—P1—N2—P2	-118.1 (2)
P1—N3—C9—C11	57.2 (6)	N3—P1—N2—C5	59.0 (5)
P1—N3—C9—C10	-65.9 (6)	N3—P1—N1—P2	118.7 (2)
P1—N3—C9—C12	176.3 (4)	N3—P1—N1—C1	-64.2 (5)
P2—P1—N2—C5	177.1 (5)	N4—P2—N2—P1	117.9 (2)
P2—P1—N1—C1	177.1 (5)	N4—P2—N2—C5	-59.3 (4)
P2—P1—N3—C9	-170.7 (4)	N4—P2—N1—P1	-118.6 (2)
P2—N2—C5—C6	-147.5 (4)	N4—P2—N1—C1	64.2 (5)
P2—N2—C5—C7	-27.7 (6)	C19—N5—C17—O1	-179.1 (6)
P2—N2—C5—C8	91.7 (5)	C18—N5—C17—O1	0.9 (10)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N3—H3...O1	0.82 (6)	2.18 (6)	2.986 (6)	165 (4)
N4—H4...O1	0.79 (5)	2.27 (5)	3.051 (5)	169 (6)