

Hydrogen bis(tetraphenylimido-diphosphinic acid) triiodide

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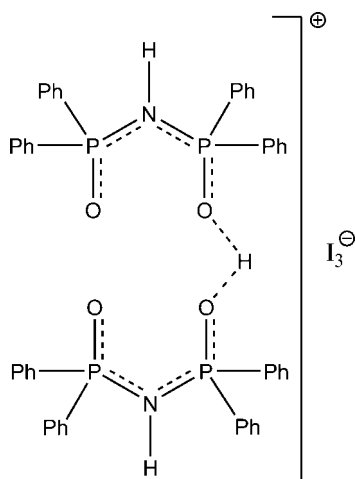
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Key indicators: single-crystal X-ray study; $T = 297$ K; mean $\sigma(\text{C}-\text{C}) = 0.009$ Å; R factor = 0.053; wR factor = 0.120; data-to-parameter ratio = 15.5.

In the crystal structure of the title compound, $[(\text{C}_{24}\text{H}_{21}\text{NO}_2\text{P}_2)_2\text{H}]^+\text{I}_3^-$, the triiodide anion is linear, sitting across a centre of symmetry, with I—I distances of 2.9130 (6) Å. The $[\text{H}\{(\text{OPPh}_2)_2\text{NH}\}_2]^+$ cation is formed by two symmetry-related molecules of tetraphenylimidodiphosphinic acid, $(\text{OPPh}_2)_2\text{NH}$, exhibiting for the O—P—N—P—O skeleton an *anti* conformation [code (−,+)/(+,−)] with an angular P—N—P fragment. The charge is determined by a single proton shared by these two molecules, disordered across a centre of symmetry and forming an O—H···O hydrogen bond.

Related literature

For related literature, see: Bassett *et al.* (2005); Bernstein *et al.* (1995); Garcia-Montalvo *et al.* (2001); Ghesner *et al.* (2005); Godfrey *et al.* (1996); Haiduc (2004); Noth (1982); Rietzel *et al.* (1990); Silvestru *et al.* (2001); Silvestru & Drake (2001); Yi *et al.* (2006).



Experimental

Crystal data

$\text{C}_{48}\text{H}_{43}\text{N}_2\text{O}_4\text{P}_4 \cdot \text{I}_3^-$
 $M_r = 1216.42$
 Triclinic, $P\bar{1}$
 $a = 10.2291$ (14) Å
 $b = 11.3075$ (15) Å
 $c = 11.3840$ (15) Å
 $\alpha = 91.451$ (2)°
 $\beta = 96.411$ (2)°

$\gamma = 108.696$ (2)°
 $V = 1236.8$ (3) Å³
 $Z = 1$
 Mo $K\alpha$ radiation
 $\mu = 2.07$ mm^{−1}
 $T = 297$ (2) K
 $0.23 \times 0.17 \times 0.14$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (*SHELXTL*; Bruker, 2001)
 $T_{\min} = 0.635$, $T_{\max} = 0.745$
 12050 measured reflections
 4347 independent reflections
 3662 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.031$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.053$
 $wR(F^2) = 0.121$
 $S = 1.12$
 4347 reflections
 281 parameters
 1 restraint

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 1.45$ e Å^{−3}
 $\Delta\rho_{\text{min}} = -0.63$ e Å^{−3}

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>
O1—H1A···O1 ⁱ	0.82	1.71	2.411 (5)	143
N1—H1···O2 ⁱⁱ	0.86 (4)	1.90 (4)	2.726 (5)	161

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT-Plus* (Bruker, 2000); program(s) used to solve structure: *SHELXTL* (Bruker, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *PUBLICIF* (Westrip, 2007).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LW2032).

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supplementary materials

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Hydrogen bis(tetraphenylimidodiphosphinic acid) triiodide

L. Copolovici, R. A. Varga, V. Lippolis and C. Silvestru

Comment

The tetraphenylimidodiphosphinato ligand $[(OPPh_2)_2N]^-$ is extensively used in coordination chemistry due to its ability to form stable complexes with a variety of main group and transition metals, Garcia-Montalvo *et al.*, 2001; Ghesner *et al.*, 2005; Haiduc, 2004; Silvestru *et al.*, 2001; Silvestru & Drake, 2001; Yi *et al.*, 2006. In metal complexes the *O,O'*-monometallic biconnective pattern of this ligand is the most common, with an angular P—N—P fragment (Silvestru & Drake, 2001). Due to its ideal framework structure for minimization of radiationless decay pathways of NIR *f-f* luminescence, $[(OPPh_2)_2N]^-$ is also an efficient sensitizer for NIR light emitting complexes of Nd^{III}, Er^{III} and Yb^{III} (Bassett *et al.*, 2005).

Metal complexes with the neutral ligand $(OPPh_2)_2NH$ are much less common (Rietzel *et al.*, 1990). The crystal structure of tetraphenylimidodiphosphinic acid, $(OPPh_2)_2NH$ is known (Noth, 1982) and it features a polymeric structure with P—N—P fragments connected by symmetric O—H—O bridges.

The crystal of the title compound contains a perfect linear triiodide anion and the cation $[H\{(OPPh_2)_2NH\}_2]^+$ (Figure 1). In this cation a hydrogen atom (H1A) bridges two symmetry related $(OPPh_2)_2NH$ molecules at O1. H1A appears to be disordered over two positions. A similar disordered hydrogen has been already reported in a similar compound $[(Me_3PO)_2H][AuI_2]$ (Godfrey *et al.*, 1996). This proton bridges two $(OPPh_2)_2NH$ molecules at O1 and its symmetry equivalent O1ⁱ atom [$O1 \cdots O1^i = 2.411$ (5) Å; symmetry code: (i) = $-x, -y + 2, -z + 1$].

The P—O and N—P distances are of the same magnitude [$O1—P1 = 1.516$ (3) Å, $O2—P2 = 1.493$ (4) Å; $N1—P1 = 1.641$ (4) Å, $N1—P2 = 1.669$ (4) Å] suggesting a delocalization of the positive charge over the OPNPO skeleton of both $(OPPh_2)_2NH$ molecules (Silvestru & Drake, 2001*b*).

Strong intermolecular interactions between the NH groups and the unprotonated oxygen atom, O2, from symmetry related cation units [$H1 \cdots O2^{ii} = 1.90$ Å; $N1 \cdots O2^{ii} = 2.726$ (5) Å; $N1—H1—O2^{ii} = 161^\circ$; symmetry code: (ii) = $-x + 1, -y + 2, -z + 1$] result in a chain polymer association (Figure 2). The hydrogen-bonding pattern can be designated as $R_2^2(8)$ (Bernstein *et al.*, 1995).

Experimental

The compound was obtained from the reaction of elemental Se, I₂ and $(SePPh_2)_2NH$ (1:1:1 molar ratio) in dichloromethane. The crystals (33% yield) were obtained by slow diffusion from CH₂Cl₂/hexane. Spectroscopic analysis: ¹H NMR (CDCl₃, 300 MHz): δ 7.34 (ddd, 816H, C₆H₅-*meta*, ³J_{HH} = 7.7, ⁴J_{HH} = 3.4 Hz), 7.51 (7, 8H, C₆H₅-*para*, ³J_{HH} = 6.7 Hz), 7.65 (dd, 16H, C₆H₅-*ortho*, ³J_{PH} = 13.3, ³J_{HH} = 7.5 Hz); ¹³C NMR (CDCl₃, 75.47 MHz): 128.59 (m, C-*meta*), 130.50 (dd, C-*ipso*,

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$^3J_{\text{PP}} = 130.5$, $^4J_{\text{PP}} = 1.7$ Hz), 131.70 (m, *C-ortho*), 132.57 (s, *C-para*); ^{31}P NMR (CDCl_3 , 121.48 MHz): δ 25.4 (s). The same compound was obtained by using elemental Se, I_2 and $(\text{SPhPh}_2)_2\text{NH}$ (1:1:1 molar ratio).

Refinement

All hydrogen atoms were placed in calculated positions using a riding model, with C—H = 0.93–0.97 Å and $U_{\text{iso}} = 1.2U_{\text{eq}}$ (C) for aryl H. The H atom bonded to N1 was found in a difference map and refined with a restrained N—H distance of 0.86 (4) Å. The half hydrogen attached to O1 was located on a difference map and refined as a riding atom at 0.82 Å from O1.

There is a Q peak close to the O2 atom and the refinement of the structure was made taking into account this feature by partial protonation at O2 with different occupancy factors. In all our attempts the refinement did not yielded anything meaningful. The main problem is that the proton partially localized on O2 gets very close to the H1 atom bonded to nitrogen N1.

Figures

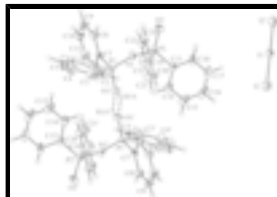


Fig. 1. : A view of title compound showing the atom-numbering scheme at 30% probability thermal ellipsoids (hydrogen bonds shown as dotted lines). H atoms are shown as small spheres of arbitrary radii. Both components of the disorder H1A are showed.

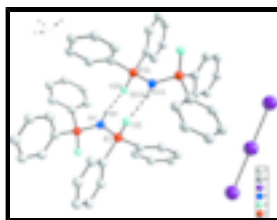


Fig. 2. : View of the secondary interactions H...O (indicated as dotted lines). All another hydrogen atoms were omitted for clarity.

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Crystal data

$\text{C}_{48}\text{H}_{43}\text{N}_2\text{O}_4\text{P}_4^+ \cdot \text{I}_3^-$

$M_r = 1216.42$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 10.2291$ (14) Å

$b = 11.3075$ (15) Å

$c = 11.3840$ (15) Å

$\alpha = 91.451$ (2)°

$\beta = 96.411$ (2)°

$\gamma = 108.696$ (2)°

$V = 1236.8$ (3) Å³

$Z = 1$

$F_{000} = 596$

$D_x = 1.633$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 3014 reflections

$\theta = 2.3$ – 21.7 °

$\mu = 2.07$ mm⁻¹

$T = 297$ (2) K

Block, brown

$0.23 \times 0.17 \times 0.14$ mm

Data collection

Bruker SMART CCD area-detector diffractometer	4347 independent reflections
Radiation source: fine-focus sealed tube	3662 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.031$
$T = 297(2)$ K	$\theta_{\text{max}} = 25.0^\circ$
φ and ω scans	$\theta_{\text{min}} = 1.8^\circ$
Absorption correction: multi-scan (SHELXTL; Bruker, 2001)	$h = -12 \rightarrow 12$
$T_{\text{min}} = 0.635$, $T_{\text{max}} = 0.745$	$k = -13 \rightarrow 13$
12050 measured reflections	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.053$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.121$	$w = 1/[\sigma^2(F_o^2) + (0.0442P)^2 + 2.0708P]$
$S = 1.12$	where $P = (F_o^2 + 2F_c^2)/3$
4347 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
281 parameters	$\Delta\rho_{\text{max}} = 1.45 \text{ e } \text{\AA}^{-3}$
1 restraint	$\Delta\rho_{\text{min}} = -0.62 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
C1	0.2542 (5)	1.2068 (5)	0.5979 (4)	0.0371 (11)	
C2	0.2634 (6)	1.2393 (5)	0.4821 (5)	0.0469 (13)	
H2	0.2488	1.1778	0.4218	0.056*	

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C3	0.2939 (7)	1.3619 (6)	0.4563 (6)	0.0638 (17)
H3	0.3014	1.3838	0.3786	0.077*
C4	0.3132 (9)	1.4524 (7)	0.5448 (8)	0.086 (2)
H4	0.3333	1.5356	0.5268	0.103*
C5	0.3034 (10)	1.4215 (7)	0.6585 (7)	0.093 (3)
H5	0.3170	1.4836	0.7180	0.111*
C6	0.2734 (7)	1.2985 (6)	0.6865 (5)	0.0606 (16)
H6	0.2661	1.2776	0.7645	0.073*
C7	0.2370 (5)	1.0310 (5)	0.7854 (4)	0.0360 (11)
C8	0.3676 (6)	1.0853 (5)	0.8477 (5)	0.0507 (14)
H8	0.4422	1.1274	0.8082	0.061*
C9	0.3871 (7)	1.0771 (6)	0.9679 (5)	0.0627 (17)
H9	0.4746	1.1156	1.0100	0.075*
C10	0.2795 (8)	1.0131 (7)	1.0264 (5)	0.0703 (19)
H10	0.2940	1.0072	1.1078	0.084*
C11	0.1503 (7)	0.9575 (6)	0.9654 (5)	0.0633 (17)
H11	0.0773	0.9130	1.0055	0.076*
C12	0.1271 (6)	0.9668 (5)	0.8445 (5)	0.0482 (13)
H12	0.0386	0.9304	0.8034	0.058*
C13	0.2745 (6)	0.7944 (5)	0.4045 (4)	0.0419 (12)
C14	0.1562 (7)	0.8115 (6)	0.3462 (5)	0.0560 (15)
H14	0.1115	0.8588	0.3836	0.067*
C15	0.1043 (8)	0.7589 (7)	0.2329 (5)	0.074 (2)
H15	0.0234	0.7685	0.1949	0.089*
C16	0.1724 (9)	0.6932 (7)	0.1774 (6)	0.085 (3)
H16	0.1382	0.6592	0.1005	0.102*
C17	0.2890 (9)	0.6760 (6)	0.2317 (7)	0.082 (2)
H17	0.3341	0.6308	0.1919	0.098*
C18	0.3418 (7)	0.7259 (5)	0.3472 (5)	0.0564 (16)
H18	0.4211	0.7133	0.3851	0.068*
C19	0.2451 (5)	0.7607 (4)	0.6548 (4)	0.0360 (11)
C20	0.1062 (6)	0.6920 (6)	0.6285 (5)	0.0531 (15)
H20	0.0582	0.6957	0.5551	0.064*
C21	0.0388 (6)	0.6177 (6)	0.7120 (6)	0.0693 (19)
H21	-0.0552	0.5718	0.6949	0.083*
C22	0.1088 (7)	0.6109 (6)	0.8195 (5)	0.0589 (16)
H22	0.0623	0.5603	0.8751	0.071*
C23	0.2466 (6)	0.6780 (5)	0.8460 (5)	0.0513 (14)
H23	0.2934	0.6735	0.9197	0.062*
C24	0.3163 (6)	0.7523 (5)	0.7642 (4)	0.0437 (12)
H24	0.4107	0.7968	0.7818	0.052*
I1	0.5000	0.5000	0.0000	0.0595 (2)
I2	0.22309 (5)	0.35171 (5)	0.04825 (5)	0.0815 (2)
N1	0.3180 (4)	0.9957 (4)	0.5632 (3)	0.0340 (9)
O2	0.4953 (4)	0.8718 (3)	0.5776 (3)	0.0482 (9)
O1	0.0584 (3)	0.9733 (3)	0.5882 (3)	0.0444 (9)
H1A	0.0427	0.9783	0.5166	0.067* 0.50
P1	0.20980 (12)	1.04556 (12)	0.63033 (11)	0.0323 (3)
P2	0.34452 (13)	0.85791 (12)	0.55250 (11)	0.0343 (3)

H1 0.370 (4) 1.051 (4) 0.523 (4) 0.038 (14)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.036 (3)	0.037 (3)	0.041 (3)	0.016 (2)	0.006 (2)	0.005 (2)
C2	0.053 (3)	0.047 (3)	0.044 (3)	0.022 (3)	0.003 (3)	0.008 (3)
C3	0.075 (4)	0.062 (4)	0.064 (4)	0.031 (3)	0.017 (3)	0.032 (3)
C4	0.119 (7)	0.046 (4)	0.104 (6)	0.038 (4)	0.023 (5)	0.020 (4)
C5	0.162 (9)	0.050 (4)	0.076 (5)	0.046 (5)	0.024 (5)	0.001 (4)
C6	0.089 (5)	0.054 (4)	0.049 (4)	0.034 (3)	0.017 (3)	0.007 (3)
C7	0.038 (3)	0.040 (3)	0.033 (3)	0.016 (2)	0.007 (2)	0.005 (2)
C8	0.051 (3)	0.054 (3)	0.043 (3)	0.013 (3)	0.003 (3)	0.008 (3)
C9	0.073 (4)	0.070 (4)	0.040 (3)	0.023 (4)	-0.015 (3)	-0.001 (3)
C10	0.108 (6)	0.080 (5)	0.028 (3)	0.039 (4)	0.005 (4)	0.009 (3)
C11	0.083 (5)	0.073 (4)	0.048 (4)	0.035 (4)	0.033 (3)	0.023 (3)
C12	0.044 (3)	0.055 (3)	0.049 (3)	0.018 (3)	0.011 (3)	0.013 (3)
C13	0.051 (3)	0.037 (3)	0.036 (3)	0.008 (2)	0.016 (2)	0.003 (2)
C14	0.068 (4)	0.061 (4)	0.038 (3)	0.019 (3)	0.012 (3)	0.001 (3)
C15	0.083 (5)	0.081 (5)	0.043 (4)	0.004 (4)	0.005 (3)	0.002 (3)
C16	0.093 (6)	0.080 (5)	0.048 (4)	-0.022 (5)	0.024 (4)	-0.021 (4)
C17	0.093 (6)	0.052 (4)	0.082 (5)	-0.012 (4)	0.052 (5)	-0.027 (4)
C18	0.065 (4)	0.041 (3)	0.061 (4)	0.007 (3)	0.031 (3)	-0.004 (3)
C19	0.042 (3)	0.034 (3)	0.035 (3)	0.014 (2)	0.013 (2)	0.011 (2)
C20	0.045 (3)	0.062 (4)	0.051 (3)	0.014 (3)	0.004 (3)	0.024 (3)
C21	0.046 (4)	0.076 (5)	0.076 (5)	0.001 (3)	0.017 (3)	0.034 (4)
C22	0.065 (4)	0.060 (4)	0.053 (4)	0.013 (3)	0.026 (3)	0.031 (3)
C23	0.065 (4)	0.057 (4)	0.036 (3)	0.024 (3)	0.009 (3)	0.015 (3)
C24	0.051 (3)	0.042 (3)	0.038 (3)	0.014 (3)	0.010 (2)	0.006 (2)
I1	0.0620 (4)	0.0660 (4)	0.0568 (4)	0.0341 (3)	-0.0025 (3)	-0.0116 (3)
I2	0.0716 (3)	0.0742 (3)	0.0942 (4)	0.0167 (3)	0.0177 (3)	-0.0173 (3)
N1	0.035 (2)	0.033 (2)	0.032 (2)	0.0074 (18)	0.0092 (18)	0.0063 (18)
O2	0.040 (2)	0.052 (2)	0.059 (2)	0.0169 (17)	0.0231 (17)	0.0202 (19)
O1	0.0338 (19)	0.051 (2)	0.045 (2)	0.0096 (16)	0.0002 (15)	0.0119 (17)
P1	0.0285 (6)	0.0367 (7)	0.0316 (7)	0.0103 (5)	0.0035 (5)	0.0051 (5)
P2	0.0359 (7)	0.0367 (7)	0.0339 (7)	0.0137 (6)	0.0117 (5)	0.0090 (5)

Geometric parameters (Å, °)

C1—C6	1.380 (7)	C14—H14	0.9300
C1—C2	1.383 (7)	C15—C16	1.358 (11)
C1—P1	1.791 (5)	C15—H15	0.9300
C2—C3	1.366 (8)	C16—C17	1.354 (11)
C2—H2	0.9300	C16—H16	0.9300
C3—C4	1.370 (10)	C17—C18	1.395 (9)
C3—H3	0.9300	C17—H17	0.9300
C4—C5	1.355 (10)	C18—H18	0.9300
C4—H4	0.9300	C19—C20	1.378 (7)
C5—C6	1.378 (9)	C19—C24	1.393 (7)

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C5—H5	0.9300	C19—P2	1.792 (5)
C6—H6	0.9300	C20—C21	1.378 (8)
C7—C8	1.381 (7)	C20—H20	0.9300
C7—C12	1.383 (7)	C21—C22	1.365 (8)
C7—P1	1.776 (5)	C21—H21	0.9300
C8—C9	1.371 (8)	C22—C23	1.364 (8)
C8—H8	0.9300	C22—H22	0.9300
C9—C10	1.362 (10)	C23—C24	1.373 (7)
C9—H9	0.9300	C23—H23	0.9300
C10—C11	1.365 (9)	C24—H24	0.9300
C10—H10	0.9300	I1—I2	2.9130 (6)
C11—C12	1.383 (8)	N1—P1	1.641 (4)
C11—H11	0.9300	N1—P2	1.669 (4)
C12—H12	0.9300	N1—H1	0.86 (4)
C13—C18	1.385 (7)	O2—P2	1.493 (4)
C13—C14	1.385 (8)	O1—P1	1.516 (3)
C13—P2	1.791 (5)	O1—H1A	0.82
C14—C15	1.379 (8)		
C6—C1—C2	119.5 (5)	C17—C16—C15	121.3 (7)
C6—C1—P1	120.7 (4)	C17—C16—H16	119.3
C2—C1—P1	119.7 (4)	C15—C16—H16	119.3
C3—C2—C1	120.1 (6)	C16—C17—C18	120.2 (7)
C3—C2—H2	120.0	C16—C17—H17	119.9
C1—C2—H2	120.0	C18—C17—H17	119.9
C2—C3—C4	120.1 (6)	C13—C18—C17	119.1 (7)
C2—C3—H3	120.0	C13—C18—H18	120.4
C4—C3—H3	120.0	C17—C18—H18	120.4
C5—C4—C3	120.4 (6)	C20—C19—C24	119.7 (5)
C5—C4—H4	119.8	C20—C19—P2	123.5 (4)
C3—C4—H4	119.8	C24—C19—P2	116.8 (4)
C4—C5—C6	120.5 (7)	C19—C20—C21	119.5 (5)
C4—C5—H5	119.8	C19—C20—H20	120.3
C6—C5—H5	119.8	C21—C20—H20	120.3
C5—C6—C1	119.5 (6)	C22—C21—C20	120.5 (6)
C5—C6—H6	120.2	C22—C21—H21	119.7
C1—C6—H6	120.2	C20—C21—H21	119.7
C8—C7—C12	119.7 (5)	C23—C22—C21	120.4 (5)
C8—C7—P1	120.1 (4)	C23—C22—H22	119.8
C12—C7—P1	120.2 (4)	C21—C22—H22	119.8
C9—C8—C7	119.8 (6)	C22—C23—C24	120.2 (5)
C9—C8—H8	120.1	C22—C23—H23	119.9
C7—C8—H8	120.1	C24—C23—H23	119.9
C10—C9—C8	120.7 (6)	C23—C24—C19	119.7 (5)
C10—C9—H9	119.7	C23—C24—H24	120.2
C8—C9—H9	119.7	C19—C24—H24	120.2
C9—C10—C11	120.0 (6)	I2—I1—I2	180.0
C9—C10—H10	120.0	P1—N1—P2	133.4 (3)
C11—C10—H10	120.0	P1—N1—H1	113 (3)
C10—C11—C12	120.5 (6)	P2—N1—H1	113 (3)

C10—C11—H11	119.8	P1—O1—H1A	109.5
C12—C11—H11	119.8	O1—P1—N1	113.0 (2)
C11—C12—C7	119.3 (6)	O1—P1—C7	107.2 (2)
C11—C12—H12	120.3	N1—P1—C7	110.3 (2)
C7—C12—H12	120.3	O1—P1—C1	112.0 (2)
C18—C13—C14	119.4 (5)	N1—P1—C1	103.6 (2)
C18—C13—P2	118.3 (5)	C7—P1—C1	110.7 (2)
C14—C13—P2	122.4 (4)	O2—P2—N1	111.7 (2)
C15—C14—C13	120.4 (6)	O2—P2—C13	111.6 (2)
C15—C14—H14	119.8	N1—P2—C13	105.7 (2)
C13—C14—H14	119.8	O2—P2—C19	111.4 (2)
C16—C15—C14	119.5 (7)	N1—P2—C19	107.0 (2)
C16—C15—H15	120.2	C13—P2—C19	109.2 (2)
C14—C15—H15	120.2		
C6—C1—C2—C3	1.2 (8)	P2—C19—C24—C23	-179.1 (4)
P1—C1—C2—C3	178.4 (4)	P2—N1—P1—O1	-59.5 (4)
C1—C2—C3—C4	-1.0 (10)	P2—N1—P1—C7	60.5 (4)
C2—C3—C4—C5	0.5 (12)	P2—N1—P1—C1	179.1 (3)
C3—C4—C5—C6	-0.2 (14)	C8—C7—P1—O1	178.3 (4)
C4—C5—C6—C1	0.4 (12)	C12—C7—P1—O1	-2.6 (5)
C2—C1—C6—C5	-1.0 (9)	C8—C7—P1—N1	54.9 (5)
P1—C1—C6—C5	-178.1 (6)	C12—C7—P1—N1	-126.0 (4)
C12—C7—C8—C9	-1.1 (8)	C8—C7—P1—C1	-59.2 (5)
P1—C7—C8—C9	178.0 (5)	C12—C7—P1—C1	119.9 (4)
C7—C8—C9—C10	1.7 (10)	C6—C1—P1—O1	105.7 (5)
C8—C9—C10—C11	-0.7 (10)	C2—C1—P1—O1	-71.4 (5)
C9—C10—C11—C12	-0.8 (10)	C6—C1—P1—N1	-132.2 (5)
C10—C11—C12—C7	1.3 (9)	C2—C1—P1—N1	50.7 (5)
C8—C7—C12—C11	-0.4 (8)	C6—C1—P1—C7	-14.0 (5)
P1—C7—C12—C11	-179.5 (4)	C2—C1—P1—C7	168.9 (4)
C18—C13—C14—C15	1.2 (9)	P1—N1—P2—O2	-131.9 (3)
P2—C13—C14—C15	-178.8 (5)	P1—N1—P2—C13	106.6 (4)
C13—C14—C15—C16	-1.9 (10)	P1—N1—P2—C19	-9.7 (4)
C14—C15—C16—C17	1.2 (11)	C18—C13—P2—O2	22.7 (5)
C15—C16—C17—C18	0.2 (11)	C14—C13—P2—O2	-157.3 (4)
C14—C13—C18—C17	0.2 (8)	C18—C13—P2—N1	144.3 (4)
P2—C13—C18—C17	-179.8 (4)	C14—C13—P2—N1	-35.7 (5)
C16—C17—C18—C13	-0.9 (9)	C18—C13—P2—C19	-100.9 (4)
C24—C19—C20—C21	1.3 (9)	C14—C13—P2—C19	79.1 (5)
P2—C19—C20—C21	178.7 (5)	C20—C19—P2—O2	-153.2 (4)
C19—C20—C21—C22	-0.6 (10)	C24—C19—P2—O2	24.3 (5)
C20—C21—C22—C23	0.2 (11)	C20—C19—P2—N1	84.4 (5)
C21—C22—C23—C24	-0.5 (10)	C24—C19—P2—N1	-98.1 (4)
C22—C23—C24—C19	1.2 (8)	C20—C19—P2—C13	-29.5 (5)
C20—C19—C24—C23	-1.6 (8)	C24—C19—P2—C13	148.0 (4)

Hydrogen-bond geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
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supplementary materials

O1—H1A···O1 ⁱ	0.82	1.71	2.411 (5)	143
N1—H1···O2 ⁱⁱ	0.86 (4)	1.90 (4)	2.726 (5)	161

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

Fig. 1

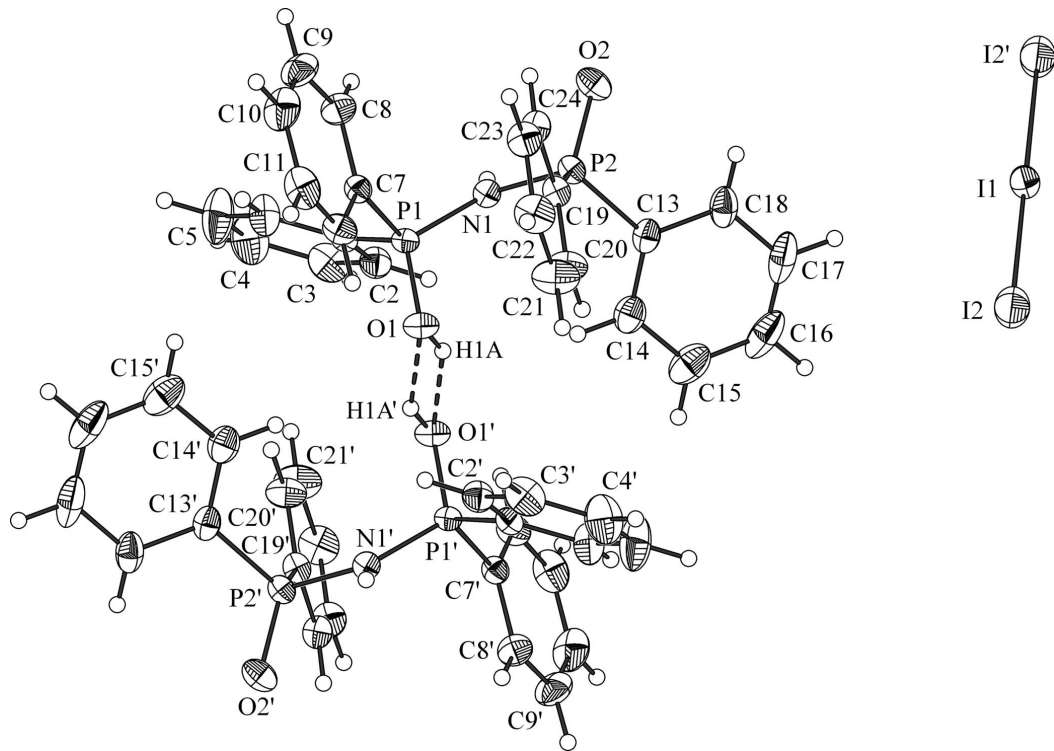


Fig. 2

