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Triethylammonium 1,1′-binaphthyl-2,2′-diyl phosphate

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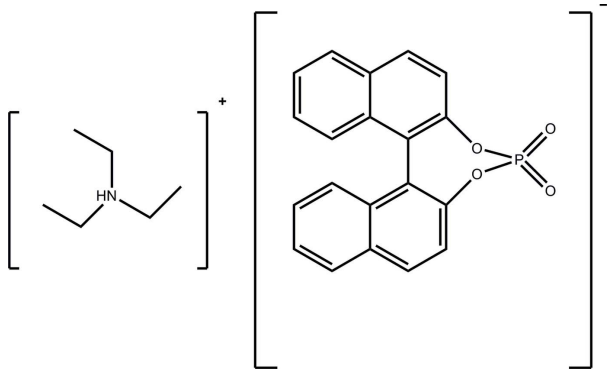
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.043; wR factor = 0.121; data-to-parameter ratio = 18.7.

In the crystal structure of the title compound, $C_6H_{16}N^+\cdot C_{20}H_{12}O_4P^-$, an N—H···O interaction links the cation to the anion. The N atom in the triethylammonium cation exhibits a trigonal-bipyramidal coordination geometry and forms an N—H···O interaction with one phosphate O atom of the 1,1′-binaphthyl-2,2′-diyl phosphate ligand. A bifurcated C—H···O interaction with the other phosphate O atom links molecules along the *a* axis. The dihedral angle between the two naphthyl ring systems is 58.92 (3)°. The refined Flack parameter value of 0.50 (10) indicates inversion twinning.

Related literature

For the use of binolphosphoric acid in synthesis, see: Jacques *et al.* (1971); Moreau *et al.*, (2009). For the binaphthyl unit in host compounds, see: Kyba *et al.* (1977).



Experimental

Crystal data

$C_6H_{16}N^+\cdot C_{20}H_{12}O_4P^-$ M_r = 449.46
Orthorhombic, $P2_12_12_1$
a = 8.4605 (2) Å
b = 13.3603 (4) Å
c = 20.5688 (7) Å
V = 2324.99 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.15 mm^{-1}
T = 298 K
0.32 × 0.27 × 0.22 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.953, T_{max} = 0.968
30327 measured reflections
5561 independent reflections
4823 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.121
S = 1.04
5561 reflections
297 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}
Absolute structure: Flack (1983)
Flack parameter: 0.50 (10)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···O4	0.87 (3)	1.83 (3)	2.689 (2)	172 (2)
C24—H24*B*···O3^{i}	0.97	2.47	3.342 (4)	149
C26—H26*A*···O3^{i}	0.97	2.47	3.373 (3)	155

Symmetry code: (i) $x-1, y, z$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2* and *SAINT-Plus* (Bruker, 2004); data reduction: *SAINT-Plus* and *XPREP* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

The authors acknowledge the Department of Chemistry, IIT Madras, for the X-ray data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2208).

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supplementary materials

Acta Cryst. (2010). E**66**, o1625 [**doi:10.1107/S160053681002026X**]

Triethylammonium 1,1'-binaphthyl-2,2'-diyl phosphate

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Comment

The title compound is a salt of binol phosphoric acid. It represents a useful tool for the resolution of amines. Amines which are unable to resolve using other chiral acids, are resolved using binolphosphoric acid very easily and in high yield (Jacques *et al.*, 1971). Optically active amines are useful as intermediates of medicines, agricultural chemicals, or the like can be produced without special post-treatment in high yield at high optical purity using optically active phosphoric acid derivatives. A recent report depicts phosphoric acid acts as Bronsted acid to catalyze the addition of enolizable β-diketones, β-ketoesters, and vinylogous amides to α,β-unsaturated aldehydes to lead to substituted chromenones, pyranones, and tetra hydroquinolinones in good yields under mild reaction conditions via a formal [3+3] cycloaddition (Moreau *et al.*, 2009).

In the title compound $C_{26}H_{28}N\,O_4P$, (I), the 1,1'-binaphthyl-2-2'diyl phosphate ligand coordinates with the triethyl ammonium to form an intra molecular N-H..O interaction with one phosphate O atom and with another phosphate O atom with which a bifurcated C-H..O interaction (Table 1) along the *a* axis extending into a network (Figure 2). The molecular structure viewed down along the C10-C11 pivot, clearly shows the non co-planar geometry of the two naptha rings system with a dihedral angle of 58.92 (3)°.

Experimental

To a stirred ice cold solution of 0.2 g (0.69 mole) binol (Evan *et. al*, 1977) in 20 mL of dichloromethane under nitrogen atmosphere was added 0.07 mL (0.69 mmol) $POCl_3$ drop wise followed by addition of 0.5 mL (3.5 mmol) triethylamine. White fumes of HCl were observed upon addition, reaction mixture was stirred at 0 °C for 30 minutes. Then 0.13 mL (6.9 mmol) H_2O was added slowly at 0 °C. Reaction mixture was stirred at 0 °C for 1 h and warmed up to room temperature and stirred for 40 h. The reaction was monitored using thin layer chromatography. The product was extracted using dichloromethane and purified by crystallization in dichloromethane. Yield is found to be 0.26 g (83.9 %).

Refinement

All hydrogen atoms were fixed geometrically and allowed to ride on the parent carbon atoms with aromatic C-H = 0.93 Å, aliphatic C-H = 0.98 Å and methyl C-H = 0.96 Å. The displacement parameters were set for phenyl and aliphatic H atoms at $U_{iso}(H) = 1.2U_{eq}(C)$ and for methyl H atoms at $U_{iso}(H) = 1.5U_{eq}(C)$. The Flack parameter was refined as a full least-squares variable, and the refined value of 0.50 (10) suggests inversion twinning.

Figures

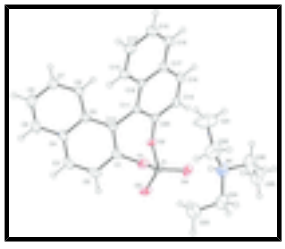

Fig. 1. The *ORTEP* drawing of the molecule with atoms represented as 30% probability ellipsoids.

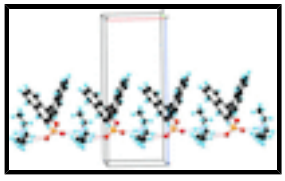

Fig. 2. The Packing diagram showing the N-H···O (blue dashed line) and the bifurcated C-H···O interactions (red dashed line)

Triethylammonium 1,1'-binaphthyl-2,2'-diyl phosphate

Crystal data

$C_6H_{16}N^+·C_{20}H_{12}O_4P^-$	$F(000) = 952$
$M_r = 449.46$	$D_x = 1.284$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: P 2ac 2ab	Cell parameters from 7503 reflections
$a = 8.4605$ (2) Å	$\theta = 2.5$–$27.9°$
$b = 13.3603$ (4) Å	$\mu = 0.15$ mm^{-1}
$c = 20.5688$ (7) Å	$T = 298$ K
$V = 2324.99$ (12) Å^3	Block, white
$Z = 4$	0.32 × 0.27 × 0.22 mm

Data collection

Bruker APEXII CCD area-detector diffractometer	5561 independent reflections
Radiation source: fine-focus sealed tube	4823 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.028$
phi and ω scans	$\theta_{max} = 28.3°$, $\theta_{min} = 2.5°$
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)	$h = -10 \rightarrow 10$
$T_{min} = 0.953$, $T_{max} = 0.968$	$k = -17 \rightarrow 15$
30327 measured reflections	$l = -27 \rightarrow 27$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$	$w = 1/[\sigma^2(F_o^2) + (0.0768P)^2 + 0.3083P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.121$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.04$	$\Delta\rho_{max} = 0.54$ e Å^{-3}
5561 reflections	$\Delta\rho_{min} = -0.31$ e Å^{-3}
297 parameters	Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
0 restraints	Extinction coefficient: 0.000
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983)
Secondary atom site location: difference Fourier map	Flack parameter: 0.50 (10)

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. 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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.9569 (2)	−0.09228 (15)	0.66118 (9)	0.0337 (4)
C2	1.0264 (3)	−0.18489 (16)	0.67555 (11)	0.0431 (5)
H2	0.9953	−0.2203	0.7123	0.052*
C3	1.1397 (3)	−0.22298 (15)	0.63547 (12)	0.0475 (5)
H3	1.1837	−0.2853	0.6444	0.057*
C4	1.1909 (3)	−0.16849 (15)	0.58042 (10)	0.0423 (5)
C5	1.3154 (3)	−0.2041 (2)	0.54033 (13)	0.0600 (7)
H5	1.3603	−0.2662	0.5489	0.072*

C6	1.3702 (4)	−0.1483 (2)	0.48930 (14)	0.0677 (8)
H6	1.4518	−0.1728	0.4635	0.081*
C7	1.3040 (3)	−0.0549 (2)	0.47599 (11)	0.0546 (6)
H7	1.3435	−0.0165	0.4419	0.065*
C8	1.1824 (3)	−0.01962 (17)	0.51244 (9)	0.0410 (5)
H8	1.1381	0.0421	0.5021	0.049*
C9	1.1211 (2)	−0.07409 (14)	0.56575 (9)	0.0323 (4)
C10	0.9960 (2)	−0.03785 (14)	0.60657 (8)	0.0298 (4)
C11	0.9160 (2)	0.05934 (13)	0.59444 (8)	0.0283 (4)
C12	0.8330 (2)	0.07958 (14)	0.53500 (8)	0.0309 (4)
C13	0.8099 (3)	0.00551 (16)	0.48651 (10)	0.0408 (5)
H13	0.8506	−0.0584	0.4928	0.049*
C14	0.7289 (3)	0.0268 (2)	0.43082 (10)	0.0518 (6)
H14	0.7143	−0.0229	0.3997	0.062*
C15	0.6676 (3)	0.1229 (2)	0.42019 (11)	0.0576 (7)
H15	0.6153	0.1371	0.3815	0.069*
C16	0.6841 (3)	0.19519 (19)	0.46587 (11)	0.0506 (6)
H16	0.6415	0.2583	0.4585	0.061*
C17	0.7658 (3)	0.17582 (15)	0.52499 (9)	0.0368 (4)
C18	0.7767 (3)	0.24789 (15)	0.57516 (10)	0.0432 (5)
H18	0.7358	0.3116	0.5684	0.052*
C19	0.8456 (2)	0.22598 (15)	0.63296 (10)	0.0368 (4)
H19	0.8485	0.2734	0.6660	0.044*
C20	0.9126 (2)	0.13082 (14)	0.64237 (8)	0.0291 (4)
C21	0.4505 (5)	0.0741 (4)	0.64124 (17)	0.1067 (15)
H21A	0.5583	0.0913	0.6504	0.160*
H21B	0.3883	0.1340	0.6381	0.160*
H21C	0.4454	0.0383	0.6008	0.160*
C22	0.3853 (4)	0.0076 (3)	0.69635 (16)	0.0853 (10)
H22A	0.2711	0.0039	0.6928	0.102*
H22B	0.4271	−0.0597	0.6917	0.102*
C23	0.4903 (4)	−0.1247 (2)	0.8033 (2)	0.0832 (10)
H23A	0.5959	−0.1078	0.7903	0.125*
H23B	0.4408	−0.1636	0.7698	0.125*
H23C	0.4936	−0.1629	0.8428	0.125*
C24	0.3995 (4)	−0.0330 (2)	0.81405 (17)	0.0715 (8)
H24A	0.4278	−0.0052	0.8560	0.086*
H24B	0.2879	−0.0494	0.8152	0.086*
C25	0.4079 (4)	0.1960 (3)	0.83499 (17)	0.0788 (9)
H25A	0.5187	0.2078	0.8285	0.118*
H25B	0.3927	0.1586	0.8744	0.118*
H25C	0.3536	0.2589	0.8382	0.118*
C26	0.3437 (3)	0.1379 (3)	0.77885 (19)	0.0791 (9)
H26A	0.2352	0.1200	0.7887	0.095*
H26B	0.3415	0.1816	0.7412	0.095*
N1	0.4281 (2)	0.04695 (16)	0.76106 (11)	0.0490 (5)
O1	0.84192 (17)	−0.05577 (10)	0.70278 (6)	0.0372 (3)
O2	0.98292 (15)	0.11072 (10)	0.70184 (6)	0.0308 (3)
O3	1.01033 (17)	0.00302 (13)	0.79917 (7)	0.0491 (4)

O4	0.74028 (17)	0.08038 (13)	0.77268 (7)	0.0451 (4)
P1	0.89188 (6)	0.03523 (4)	0.75172 (2)	0.03329 (13)
H1N	0.529 (4)	0.0593 (18)	0.7609 (12)	0.044 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0328 (10)	0.0342 (10)	0.0342 (9)	−0.0053 (8)	−0.0050 (8)	−0.0002 (7)
C2	0.0506 (13)	0.0361 (10)	0.0424 (10)	−0.0078 (10)	−0.0115 (10)	0.0096 (9)
C3	0.0560 (15)	0.0293 (10)	0.0573 (12)	0.0071 (10)	−0.0186 (11)	0.0003 (9)
C4	0.0451 (13)	0.0371 (11)	0.0446 (11)	0.0090 (9)	−0.0150 (10)	−0.0086 (8)
C5	0.0611 (17)	0.0540 (14)	0.0649 (16)	0.0286 (13)	−0.0112 (13)	−0.0187 (12)
C6	0.0567 (18)	0.090 (2)	0.0565 (15)	0.0292 (15)	0.0067 (13)	−0.0209 (14)
C7	0.0493 (14)	0.0724 (17)	0.0421 (12)	0.0084 (12)	0.0076 (10)	−0.0062 (10)
C8	0.0397 (12)	0.0492 (12)	0.0341 (9)	0.0076 (9)	0.0003 (8)	−0.0012 (8)
C9	0.0336 (10)	0.0330 (9)	0.0303 (8)	0.0042 (8)	−0.0060 (7)	−0.0057 (7)
C10	0.0314 (10)	0.0295 (8)	0.0285 (8)	−0.0011 (8)	−0.0059 (7)	−0.0013 (7)
C11	0.0272 (9)	0.0312 (9)	0.0266 (8)	0.0016 (7)	0.0007 (7)	−0.0001 (6)
C12	0.0300 (10)	0.0345 (9)	0.0281 (8)	0.0037 (8)	−0.0001 (7)	−0.0002 (7)
C13	0.0452 (13)	0.0416 (11)	0.0355 (10)	0.0086 (9)	−0.0082 (9)	−0.0055 (8)
C14	0.0579 (15)	0.0613 (14)	0.0363 (10)	0.0111 (12)	−0.0154 (10)	−0.0122 (10)
C15	0.0617 (16)	0.0760 (17)	0.0351 (11)	0.0244 (14)	−0.0169 (11)	−0.0014 (11)
C16	0.0550 (15)	0.0526 (13)	0.0443 (12)	0.0177 (11)	−0.0136 (11)	0.0031 (10)
C17	0.0364 (11)	0.0393 (10)	0.0347 (9)	0.0072 (9)	−0.0023 (8)	0.0025 (8)
C18	0.0501 (13)	0.0339 (10)	0.0456 (11)	0.0124 (9)	0.0008 (10)	0.0005 (8)
C19	0.0387 (11)	0.0341 (9)	0.0375 (9)	0.0034 (8)	0.0028 (8)	−0.0092 (8)
C20	0.0251 (9)	0.0348 (9)	0.0275 (8)	−0.0043 (7)	0.0019 (7)	−0.0002 (7)
C21	0.106 (3)	0.148 (4)	0.066 (2)	−0.055 (3)	−0.031 (2)	0.016 (2)
C22	0.0621 (19)	0.124 (3)	0.0700 (19)	−0.028 (2)	−0.0083 (16)	−0.0116 (19)
C23	0.066 (2)	0.0684 (19)	0.115 (3)	−0.0038 (17)	0.019 (2)	0.0201 (19)
C24	0.0518 (16)	0.081 (2)	0.0814 (19)	−0.0096 (16)	0.0100 (15)	0.0077 (16)
C25	0.067 (2)	0.080 (2)	0.090 (2)	−0.0061 (17)	0.0116 (18)	−0.0214 (18)
C26	0.0399 (14)	0.0747 (19)	0.123 (3)	−0.0043 (14)	−0.0115 (17)	−0.0150 (19)
N1	0.0287 (10)	0.0563 (11)	0.0620 (12)	−0.0120 (8)	−0.0041 (8)	−0.0065 (9)
O1	0.0352 (7)	0.0421 (8)	0.0344 (7)	−0.0118 (6)	0.0018 (6)	0.0011 (5)
O2	0.0270 (7)	0.0402 (7)	0.0253 (6)	−0.0071 (6)	−0.0016 (5)	−0.0022 (5)
O3	0.0333 (8)	0.0790 (11)	0.0349 (7)	−0.0104 (8)	−0.0052 (6)	0.0127 (7)
O4	0.0269 (7)	0.0676 (10)	0.0408 (7)	−0.0058 (7)	0.0043 (6)	−0.0114 (7)
P1	0.0237 (2)	0.0516 (3)	0.0245 (2)	−0.0080 (2)	0.00039 (18)	−0.0002 (2)

Geometric parameters (Å, °)

C1—C10	1.378 (3)	C18—C19	1.356 (3)
C1—O1	1.385 (2)	C18—H18	0.9300
C1—C2	1.401 (3)	C19—C20	1.405 (3)
C2—C3	1.363 (4)	C19—H19	0.9300
C2—H2	0.9300	C20—O2	1.386 (2)
C3—C4	1.414 (3)	C21—C22	1.542 (5)
C3—H3	0.9300	C21—H21A	0.9600

C4—C5	1.419 (3)	C21—H21B	0.9600
C4—C9	1.425 (3)	C21—H21C	0.9600
C5—C6	1.368 (4)	C22—N1	1.476 (4)
C5—H5	0.9300	C22—H22A	0.9700
C6—C7	1.396 (4)	C22—H22B	0.9700
C6—H6	0.9300	C23—C24	1.463 (4)
C7—C8	1.358 (3)	C23—H23A	0.9600
C7—H7	0.9300	C23—H23B	0.9600
C8—C9	1.415 (3)	C23—H23C	0.9600
C8—H8	0.9300	C24—N1	1.545 (4)
C9—C10	1.435 (3)	C24—H24A	0.9700
C10—C11	1.485 (3)	C24—H24B	0.9700
C11—C20	1.373 (2)	C25—C26	1.494 (4)
C11—C12	1.436 (2)	C25—H25A	0.9600
C12—C13	1.418 (3)	C25—H25B	0.9600
C12—C17	1.421 (3)	C25—H25C	0.9600
C13—C14	1.365 (3)	C26—N1	1.456 (4)
C13—H13	0.9300	C26—H26A	0.9700
C14—C15	1.402 (3)	C26—H26B	0.9700
C14—H14	0.9300	N1—H1N	0.87 (3)
C15—C16	1.355 (3)	O1—P1	1.6340 (14)
C15—H15	0.9300	O2—P1	1.6319 (13)
C16—C17	1.422 (3)	O3—P1	1.4636 (15)
C16—H16	0.9300	O4—P1	1.4815 (16)
C17—C18	1.414 (3)		
C10—C1—O1	119.10 (18)	C18—C19—H19	120.3
C10—C1—C2	122.5 (2)	C20—C19—H19	120.3
O1—C1—C2	118.39 (18)	C11—C20—O2	119.33 (16)
C3—C2—C1	119.8 (2)	C11—C20—C19	122.59 (17)
C3—C2—H2	120.1	O2—C20—C19	118.03 (16)
C1—C2—H2	120.1	C22—C21—H21A	109.5
C2—C3—C4	120.50 (19)	C22—C21—H21B	109.5
C2—C3—H3	119.7	H21A—C21—H21B	109.5
C4—C3—H3	119.7	C22—C21—H21C	109.5
C3—C4—C5	121.3 (2)	H21A—C21—H21C	109.5
C3—C4—C9	119.9 (2)	H21B—C21—H21C	109.5
C5—C4—C9	118.8 (2)	N1—C22—C21	111.7 (3)
C6—C5—C4	121.0 (2)	N1—C22—H22A	109.3
C6—C5—H5	119.5	C21—C22—H22A	109.3
C4—C5—H5	119.5	N1—C22—H22B	109.3
C5—C6—C7	120.1 (2)	C21—C22—H22B	109.3
C5—C6—H6	120.0	H22A—C22—H22B	107.9
C7—C6—H6	120.0	C24—C23—H23A	109.5
C8—C7—C6	120.4 (3)	C24—C23—H23B	109.5
C8—C7—H7	119.8	H23A—C23—H23B	109.5
C6—C7—H7	119.8	C24—C23—H23C	109.5
C7—C8—C9	121.8 (2)	H23A—C23—H23C	109.5
C7—C8—H8	119.1	H23B—C23—H23C	109.5
C9—C8—H8	119.1	C23—C24—N1	113.0 (2)

C8—C9—C4	117.86 (19)	C23—C24—H24A	109.0
C8—C9—C10	123.39 (17)	N1—C24—H24A	109.0
C4—C9—C10	118.73 (18)	C23—C24—H24B	109.0
C1—C10—C9	118.40 (17)	N1—C24—H24B	109.0
C1—C10—C11	119.26 (17)	H24A—C24—H24B	107.8
C9—C10—C11	122.20 (16)	C26—C25—H25A	109.5
C20—C11—C12	118.06 (16)	C26—C25—H25B	109.5
C20—C11—C10	119.80 (15)	H25A—C25—H25B	109.5
C12—C11—C10	122.05 (16)	C26—C25—H25C	109.5
C13—C12—C17	118.33 (17)	H25A—C25—H25C	109.5
C13—C12—C11	122.32 (17)	H25B—C25—H25C	109.5
C17—C12—C11	119.31 (17)	N1—C26—C25	116.7 (3)
C14—C13—C12	120.9 (2)	N1—C26—H26A	108.1
C14—C13—H13	119.5	C25—C26—H26A	108.1
C12—C13—H13	119.5	N1—C26—H26B	108.1
C13—C14—C15	120.5 (2)	C25—C26—H26B	108.1
C13—C14—H14	119.8	H26A—C26—H26B	107.3
C15—C14—H14	119.8	C26—N1—C22	113.8 (3)
C16—C15—C14	120.4 (2)	C26—N1—C24	108.8 (2)
C16—C15—H15	119.8	C22—N1—C24	110.6 (2)
C14—C15—H15	119.8	C26—N1—H1N	108.9 (16)
C15—C16—C17	120.9 (2)	C22—N1—H1N	107.8 (17)
C15—C16—H16	119.6	C24—N1—H1N	106.7 (16)
C17—C16—H16	119.6	C1—O1—P1	117.46 (12)
C18—C17—C12	118.96 (17)	C20—O2—P1	118.16 (11)
C18—C17—C16	122.1 (2)	O3—P1—O4	121.23 (9)
C12—C17—C16	118.87 (19)	O3—P1—O2	106.13 (8)
C19—C18—C17	121.39 (18)	O4—P1—O2	109.87 (9)
C19—C18—H18	119.3	O3—P1—O1	111.67 (9)
C17—C18—H18	119.3	O4—P1—O1	104.96 (8)
C18—C19—C20	119.30 (18)	O2—P1—O1	101.22 (7)
C10—C1—C2—C3	1.5 (3)	C12—C13—C14—C15	0.5 (4)
O1—C1—C2—C3	179.86 (19)	C13—C14—C15—C16	−1.8 (4)
C1—C2—C3—C4	1.8 (3)	C14—C15—C16—C17	1.0 (4)
C2—C3—C4—C5	176.2 (2)	C13—C12—C17—C18	174.9 (2)
C2—C3—C4—C9	−1.5 (3)	C11—C12—C17—C18	−2.7 (3)
C3—C4—C5—C6	−176.3 (2)	C13—C12—C17—C16	−2.6 (3)
C9—C4—C5—C6	1.4 (4)	C11—C12—C17—C16	179.8 (2)
C4—C5—C6—C7	0.1 (4)	C15—C16—C17—C18	−176.1 (3)
C5—C6—C7—C8	−1.6 (4)	C15—C16—C17—C12	1.3 (4)
C6—C7—C8—C9	1.7 (4)	C12—C17—C18—C19	−2.0 (3)
C7—C8—C9—C4	−0.2 (3)	C16—C17—C18—C19	175.4 (2)
C7—C8—C9—C10	178.0 (2)	C17—C18—C19—C20	2.3 (3)
C3—C4—C9—C8	176.4 (2)	C12—C11—C20—O2	175.82 (16)
C5—C4—C9—C8	−1.3 (3)	C10—C11—C20—O2	−0.6 (3)
C3—C4—C9—C10	−1.9 (3)	C12—C11—C20—C19	−6.7 (3)
C5—C4—C9—C10	−179.6 (2)	C10—C11—C20—C19	176.85 (18)
O1—C1—C10—C9	176.82 (16)	C18—C19—C20—C11	2.2 (3)
C2—C1—C10—C9	−4.8 (3)	C18—C19—C20—O2	179.68 (19)

O1—C1—C10—C11	1.1 (3)	C25—C26—N1—C22	−165.1 (3)
C2—C1—C10—C11	179.47 (18)	C25—C26—N1—C24	71.1 (3)
C8—C9—C10—C1	−173.24 (18)	C21—C22—N1—C26	71.5 (4)
C4—C9—C10—C1	4.9 (3)	C21—C22—N1—C24	−165.7 (3)
C8—C9—C10—C11	2.3 (3)	C23—C24—N1—C26	−175.3 (3)
C4—C9—C10—C11	−179.53 (17)	C23—C24—N1—C22	59.1 (4)
C1—C10—C11—C20	52.0 (3)	C10—C1—O1—P1	−76.8 (2)
C9—C10—C11—C20	−123.5 (2)	C2—C1—O1—P1	104.78 (18)
C1—C10—C11—C12	−124.3 (2)	C11—C20—O2—P1	−74.93 (19)
C9—C10—C11—C12	60.2 (3)	C19—C20—O2—P1	107.47 (18)
C20—C11—C12—C13	−170.67 (19)	C20—O2—P1—O3	162.56 (13)
C10—C11—C12—C13	5.7 (3)	C20—O2—P1—O4	−64.72 (14)
C20—C11—C12—C17	6.8 (3)	C20—O2—P1—O1	45.86 (14)
C10—C11—C12—C17	−176.80 (18)	C1—O1—P1—O3	−65.43 (15)
C17—C12—C13—C14	1.7 (3)	C1—O1—P1—O4	161.44 (14)
C11—C12—C13—C14	179.3 (2)	C1—O1—P1—O2	47.12 (14)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N···O4	0.87 (3)	1.83 (3)	2.689 (2)	172 (2)
C24—H24B···O3^{i}	0.97	2.47	3.342 (4)	149
C26—H26A···O3^{i}	0.97	2.47	3.373 (3)	155

Symmetry codes: (i) *x*−1, *y*, *z*.

Fig. 1

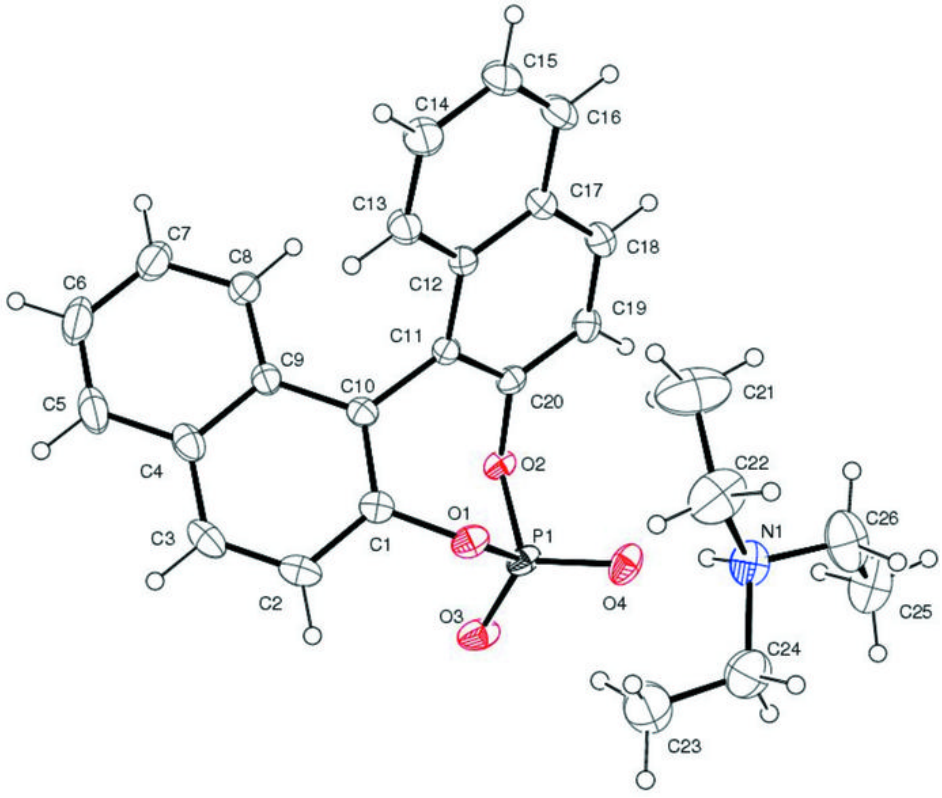
C15
C14
C16
C13
C17
C7
C8
C18
C6
C12
C19
C11
C9
C10
C20
C21
C5
O2
C22
C4
O1
P1
N1
C26
C3
C1
C2
O3
O4
C25
C24
C23

Fig. 2