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4-(2,2′:6′,2″-Terpyridyl)benzyltriphenylphosphonium bromide

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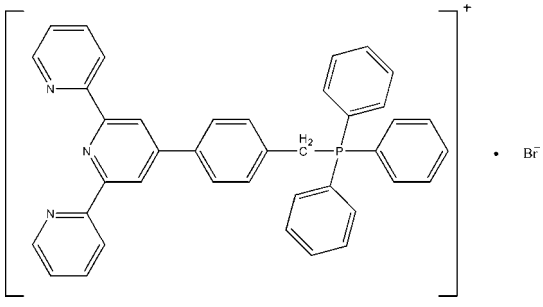
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.007 Å; R factor = 0.051; wR factor = 0.109; data-to-parameter ratio = 15.7.

In the title compound, $C_{40}H_{31}N_3P^+\cdot Br^-$, the 4-(2,2′:6′,2″-terpyridine)benzyl group is in a slightly twisted conformation with a dihedral angle between the benzyl ring and the central pyridyl ring of 28.87 (5)°. The dihedral angles between the two outer and the inner pyridyl rings are only 4.04 (4) and 7.17 (5)°, respectively. The three phenyl groups are in a propeller configuration typical for this type of triphenylphosphonium compound. The title compound exhibits large voids filled with diffuse solvent molecules of 252.5 Å^3 (13.9% of the unit-cell volume), which stretch as channels parallel to the c axis. The packing of the structural components is governed by C—H···Br hydrogen bonds, and C—H···π as well as π–π stacking interactions [the closest centroid-to-centroid distance is 3.843 (2) Å], to form a three-dimensional supramolecular structure.

Related literature

For related literature, see: Czerwinski (1986, 2004); Ponnuswamy & Czerwinski (1986); Schubert *et al.* (2006); Tessore *et al.* (2005).



Experimental

Crystal data

$C_{40}H_{31}N_3P^+\cdot Br^-$
M_r = 664.56
Monoclinic, $P2_1/n$
a = 12.7721 (3) Å
b = 12.5364 (3) Å
c = 23.3622 (6) Å
β = 103.874 (2)°
V = 3631.53 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.21 mm^{-1}
T = 293 (2) K
0.30 × 0.29 × 0.23 mm

Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.714, T_{max} = 0.769
20487 measured reflections
6392 independent reflections
2543 reflections with $I > 2\sigma(I)$
R_{int} = 0.061

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.110
S = 0.83
6392 reflections
406 parameters
7 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Hydrogen-bond and π–π interaction geometry (Å, °).

*Cg*1 is the centroid of ring C16–C21, *Cg*2 is the centroid of ring C23–C28.

D—H···A	D—H	H···A	D···A	D—H···A
C22—H22*B*···Br1	0.97	2.98	3.938 (4)	171
C24—H24···Br1^{i}	0.93	2.83	3.707 (5)	158
C40—H40···*Cg*1	0.93	2.85	3.631 (1)	162
C39—H39···*Cg*2^{ii}	0.93	2.73	3.478 (1)	126

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT* and *PLATON* (Spek, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2004); 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: ZL2064).

References

Bruker (2004). *APEX2* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Czerwinski, E. W. (1986). *Acta Cryst.* C**42**, 236–239.
Czerwinski, E. W. (2004). *Acta Cryst.* E**60**, o1442–o1443.
Ponnuswamy, M. N. & Czerwinski, E. W. (1986). *Acta Cryst.* C**42**, 1019–1022.
Schubert, U. S., Hofmeier, H. & Newkome, G. R. (2006). *Modern Terpyridine Chemistry*. Weinheim: Wiley-VCH.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Tessore, F., Roberto, D., Ugo, R. & Pizzotti, M. (2005). *Inorg. Chem.* **44**, 8967–8978.

supplementary materials

Acta Cryst. (2007). E**63**, o4047 [**doi:10.1107/S1600536807043668**]

4-(2,2':6',2''-Terpyridyl)benzyltriphenylphosphonium bromide

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Comment

Terpyridines are a family of widely used functional ligands (Schubert *et al.*, 2006). In this paper, we descibe the crystal structure of the title compound, a cationic and potentially trichelating terpyridine ligand.

The atom labeling and displacement ellipsoids of the title compound are shown in Fig. 1. The 4'-(4-benzyl)-2,2':6',2''-terpyridine moiety, which is attached *via* the terminal C atom of the benzyl group to the P atom, is in a slightly twisted conformation. The dihedral angle between the benzyl ring and the plane of the central pyridyl ring of the terpyridine moiety is 28.87 (5) °. The three pyridyl rings are located almost within the same plane with dihedral angles of 4.04 (4) and 7.17 (5)° between the two outer and the inner pyridyl rings, respectively. The phenyl substituents of the triphenylphosphonium moiety are in a propeller configuration typical for this type of compounds (Czerwinski, 1986, 2004; Ponnuswamy & Czerwinski, 1986).

The title compound exhibits large voids filled with diffuse solvent molecules of 252.5 Å^3 (13.9% of the unit cell volume) which stretch as channels parallel to the *c* axis, and the dataset was corrected for the contributions of the diffuse solvent using *PLATON* (Spek, 2003; see the experimental section for details). The packing of the structural components is governed by C—H···Br hydrogen bonds, and C—H···π as well as π-π stacking interactions to form a three-dimensional supramolecular structure (Fig. 2). The C—H···Br hydrogen bonds are given in the hydrogen-bond geometry table. Short C—H···π contacts are found for C40—H···*Cg*1 (2.85 (2) Å) and C39—H···*Cg*2^{ii} (2.73 (4) Å). The molecules also exhibit π-π stacking interactions, the closest centroid to centroid distance is found for *Cg*1—*Cg*3^{iii} (3.843 (2) Å). [*Cg*1 = Ring (C16—C21); *Cg*2 = Ring (C23—C28); *Cg*3 = Ring (N1, C1—C5); (ii): −*x*, 1 − *y*, −*z*; (iii): 1 − *x*, 1 − *y*, −*z*].

Experimental

The title compound was prepared as described previously (Tessore *et al.*, 2005). A solution of triphenylphosphine (0.26 g, 1 mmol) and 4'-(phenyl-*p*-bromomethyl)-2,2':6',2''-terpyridine (0.401 g, 1 mmol) in toluene (10 ml) was refluxed for 4 h under magnetic stirring. During this time a white solid precipitate formed. The reaction mixture was cooled with an ice bath and the solid was collected by filtration. The residue was recrystallized from toluene/methanol/water (1:4:1, 6 ml), yielding colorless blocky single crystals.

Refinement

Large voids filled with diffuse solvent molecules of two times 252.5 Å^3 were found in the unit cell of the compound (13.9% of the unit cell volume). Due to the amorphous nature of the solvent molecules, possibly water or methanol, no individual atom positions could be identified and thus the dataset was corrected for the contributions of the diffuse solvent using the Squeeze procedure implemented in the molecular geometry program *PLATON* (Spek, 2003). The *R* values improved from 0.0693 to 0.0513 and 0.2299 to 0.1095 for R1 and wR2, respectively, after application of the correction.

Carbon atoms C29 and C34 showed unusually asymmetric anistropic displacement parameters and were subjected to a rigid bond restraint, *i.e.* the components of the anisotropic displacement parameters in the direction of the bonds were restrained to be equal within standard deviations of 0.02 for bonds and 0.01 for 1,3 distances (DELU restraint in *SHELXTL*). The ADPs were also restrained to have the same U^{ij} components as the neighboring atoms within standard deviations of 0.02 and 0.01 for s and st (SIMU restraint in *SHELXTL*).

Carbon-bound H atoms were placed at calculated positions and were treated as riding on the parent C atoms with C—H = 0.93 or 0.97 Å and with U_{iso}(H) = 1.2 U_{eq}(C), respectively.

Figures

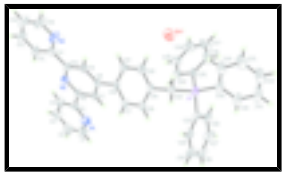

Fig. 1. The structure of (I), showing the atom-numbering scheme and displacement ellipsoids drawn at the 30% probability level.

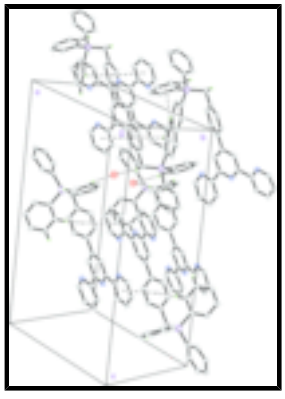

Fig. 2. The packing view of (I). Hydrogen bonds, C—H···π and π···π interactions are shown as dashed lines. H atoms not involved in close contacts were omitted for clarity.

4-(2,2':6',2"-Terpyridyl)benzyltriphenylphosphonium bromide

Crystal data

$C_{40}H_{31}N_3P^+\cdot Br^-$	F_{000} = 1368
M_r = 664.56	D_x = 1.215 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 6500 reflections
a = 12.7721 (3) Å	θ = 1.4–28.0º
b = 12.5364 (3) Å	μ = 1.21 mm^{-1}
c = 23.3622 (6) Å	T = 293 (2) K
β = 103.874 (2)º	Block, colorless
V = 3631.53 (15) Å^3	0.30 × 0.29 × 0.23 mm
Z = 4	

Data collection

Bruker APEXII area-detector diffractometer	6392 independent reflections
Radiation source: fine-focus sealed tube	2543 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.061$
$T = 293(2)$ K	$\theta_{max} = 25.0°$
φ and ω scan	$\theta_{min} = 1.7°$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -15\rightarrow8$
$T_{min} = 0.714$, $T_{max} = 0.769$	$k = -14\rightarrow11$
20487 measured reflections	$l = -25\rightarrow27$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.051$	H-atom parameters constrained
$wR(F^2) = 0.110$	$w = 1/[\sigma^2(F_o^2) + (0.0449P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.83$	$(\Delta/\sigma)_{max} < 0.001$
6392 reflections	$\Delta\rho_{max} = 0.26$ e Å^{-3}
406 parameters	$\Delta\rho_{min} = -0.22$ e Å^{-3}
7 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 $F^2 > 2\text{sigma}(F^2)$ 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}
Br1	0.14824 (3)	0.03312 (4)	0.21079 (2)	0.0791 (2)
C1	0.4092 (4)	0.7413 (4)	−0.1284 (3)	0.0893 (16)
H1	0.3845	0.8047	−0.1155	0.107*
C2	0.4580 (4)	0.7468 (5)	−0.1753 (2)	0.0850 (16)

H2	0.4655	0.8116	−0.1932	0.102*
C3	0.4944 (3)	0.6547 (5)	−0.1942 (2)	0.0773 (14)
H3	0.5277	0.6548	−0.2255	0.093*
C4	0.4810 (3)	0.5615 (4)	−0.16598 (19)	0.0607 (12)
H4	0.5048	0.4974	−0.1783	0.073*
C5	0.4320 (3)	0.5628 (4)	−0.11921 (19)	0.0513 (11)
C6	0.4219 (3)	0.4655 (4)	−0.08609 (16)	0.0411 (10)
C7	0.3655 (3)	0.4635 (3)	−0.04207 (16)	0.0476 (10)
H7	0.3325	0.5253	−0.0331	0.057*
C8	0.3581 (3)	0.3695 (3)	−0.01107 (17)	0.0451 (10)
C9	0.4096 (3)	0.2803 (3)	−0.02670 (16)	0.0490 (11)
H9	0.4069	0.2159	−0.0074	0.059*
C10	0.4654 (3)	0.2867 (4)	−0.07129 (18)	0.0476 (11)
C11	0.5220 (3)	0.1930 (4)	−0.08802 (18)	0.0507 (11)
C12	0.5833 (3)	0.1995 (4)	−0.12916 (18)	0.0612 (12)
H12	0.5903	0.2640	−0.1475	0.073*
C13	0.6338 (3)	0.1095 (5)	−0.1427 (2)	0.0744 (14)
H13	0.6755	0.1126	−0.1703	0.089*
C14	0.6226 (4)	0.0160 (5)	−0.1154 (2)	0.0884 (16)
H14	0.6556	−0.0460	−0.1240	0.106*
C15	0.5613 (4)	0.0161 (4)	−0.0749 (2)	0.0991 (18)
H15	0.5548	−0.0477	−0.0558	0.119*
C16	0.2999 (3)	0.3639 (3)	0.03712 (16)	0.0437 (10)
C17	0.2908 (3)	0.4522 (3)	0.07130 (18)	0.0518 (11)
H17	0.3181	0.5175	0.0627	0.062*
C18	0.2419 (3)	0.4450 (3)	0.11786 (17)	0.0521 (11)
H18	0.2393	0.5045	0.1413	0.062*
C19	0.1968 (3)	0.3502 (4)	0.12970 (16)	0.0457 (10)
C20	0.2052 (3)	0.2611 (3)	0.09625 (19)	0.0572 (12)
H20	0.1767	0.1963	0.1046	0.069*
C21	0.2560 (3)	0.2685 (3)	0.05018 (17)	0.0543 (11)
H21	0.2607	0.2083	0.0276	0.065*
C22	0.1367 (3)	0.3417 (3)	0.17850 (16)	0.0505 (11)
H22A	0.1766	0.3805	0.2128	0.061*
H22B	0.1341	0.2674	0.1897	0.061*
C23	0.0014 (4)	0.5363 (3)	0.15640 (18)	0.0500 (11)
C24	0.0839 (4)	0.5943 (5)	0.19176 (19)	0.0668 (13)
H24	0.1414	0.5597	0.2168	0.080*
C25	0.0802 (4)	0.7036 (5)	0.1895 (2)	0.0796 (15)
H25	0.1371	0.7425	0.2123	0.096*
C26	−0.0058 (6)	0.7574 (4)	0.1543 (3)	0.0902 (16)
H26	−0.0067	0.8316	0.1532	0.108*
C27	−0.0901 (4)	0.6999 (5)	0.1208 (2)	0.0849 (15)
H27	−0.1497	0.7351	0.0978	0.102*
C28	−0.0863 (3)	0.5905 (4)	0.12108 (18)	0.0658 (13)
H28	−0.1428	0.5520	0.0975	0.079*
C29	−0.0731 (3)	0.3506 (4)	0.20963 (17)	0.0582 (12)
C30	−0.1347 (3)	0.4209 (4)	0.23347 (19)	0.0801 (14)
H30	−0.1342	0.4931	0.2246	0.096*

C31	−0.1971 (4)	0.3847 (6)	0.2704 (2)	0.102 (2)
H31	−0.2381	0.4324	0.2865	0.122*
C32	−0.1981 (4)	0.2801 (6)	0.2830 (2)	0.098 (2)
H32	−0.2403	0.2565	0.3078	0.118*
C33	−0.1386 (4)	0.2068 (4)	0.2601 (2)	0.0864 (16)
H33	−0.1407	0.1345	0.2688	0.104*
C34	−0.0751 (3)	0.2450 (4)	0.22351 (18)	0.0723 (13)
H34	−0.0332	0.1972	0.2082	0.087*
C35	−0.0673 (3)	0.3479 (3)	0.08584 (16)	0.0471 (10)
C36	−0.1589 (4)	0.2867 (4)	0.0772 (2)	0.0750 (14)
H36	−0.1867	0.2683	0.1092	0.090*
C37	−0.2107 (4)	0.2520 (4)	0.0210 (3)	0.0937 (16)
H37	−0.2724	0.2101	0.0157	0.112*
C38	−0.1717 (4)	0.2788 (4)	−0.0257 (2)	0.0771 (14)
H38	−0.2058	0.2544	−0.0632	0.093*
C39	−0.0824 (4)	0.3418 (3)	−0.01822 (19)	0.0704 (13)
H39	−0.0563	0.3609	−0.0507	0.085*
C40	−0.0307 (3)	0.3771 (3)	0.03710 (19)	0.0606 (12)
H40	0.0294	0.4210	0.0417	0.073*
N1	0.3952 (3)	0.6523 (4)	−0.10053 (15)	0.0709 (11)
N2	0.4714 (2)	0.3785 (3)	−0.10008 (13)	0.0473 (9)
N3	0.5100 (3)	0.1016 (4)	−0.06086 (17)	0.0832 (12)
P1	−0.00022 (8)	0.39351 (9)	0.15776 (4)	0.0518 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0646 (3)	0.0886 (4)	0.0834 (4)	−0.0003 (3)	0.0167 (3)	0.0069 (3)
C1	0.096 (4)	0.061 (4)	0.117 (5)	0.010 (3)	0.037 (4)	0.010 (4)
C2	0.082 (4)	0.081 (5)	0.094 (5)	0.001 (3)	0.025 (3)	0.036 (3)
C3	0.073 (3)	0.085 (5)	0.075 (4)	0.005 (3)	0.019 (3)	0.025 (4)
C4	0.060 (3)	0.067 (4)	0.057 (3)	0.000 (2)	0.020 (2)	0.007 (3)
C5	0.042 (3)	0.058 (4)	0.051 (3)	−0.002 (2)	0.004 (2)	0.007 (3)
C6	0.035 (2)	0.051 (3)	0.036 (3)	0.004 (2)	0.0080 (19)	0.000 (2)
C7	0.042 (3)	0.052 (3)	0.047 (3)	0.005 (2)	0.007 (2)	−0.001 (2)
C8	0.043 (3)	0.049 (3)	0.044 (3)	0.006 (2)	0.010 (2)	−0.002 (2)
C9	0.047 (3)	0.051 (3)	0.052 (3)	0.003 (2)	0.017 (2)	0.008 (2)
C10	0.045 (3)	0.055 (3)	0.043 (3)	0.005 (2)	0.011 (2)	−0.002 (2)
C11	0.055 (3)	0.055 (4)	0.042 (3)	0.009 (3)	0.011 (2)	0.000 (2)
C12	0.057 (3)	0.075 (4)	0.053 (3)	0.012 (3)	0.015 (2)	−0.006 (2)
C13	0.070 (3)	0.091 (5)	0.064 (4)	0.019 (3)	0.018 (3)	−0.013 (3)
C14	0.098 (4)	0.085 (5)	0.085 (4)	0.030 (3)	0.026 (3)	−0.015 (3)
C15	0.133 (5)	0.067 (4)	0.113 (5)	0.039 (3)	0.059 (4)	0.017 (3)
C16	0.040 (3)	0.050 (3)	0.045 (3)	0.002 (2)	0.018 (2)	0.004 (2)
C17	0.049 (3)	0.053 (3)	0.057 (3)	0.001 (2)	0.019 (2)	−0.008 (2)
C18	0.042 (3)	0.062 (3)	0.058 (3)	0.001 (2)	0.022 (2)	−0.007 (2)
C19	0.035 (2)	0.061 (3)	0.045 (3)	0.007 (2)	0.019 (2)	0.003 (2)
C20	0.063 (3)	0.049 (3)	0.067 (3)	0.002 (2)	0.031 (3)	0.001 (3)

C21	0.060 (3)	0.048 (3)	0.061 (3)	−0.002 (2)	0.029 (2)	−0.007 (2)
C22	0.045 (3)	0.064 (3)	0.043 (3)	0.007 (2)	0.011 (2)	0.006 (2)
C23	0.051 (3)	0.054 (3)	0.048 (3)	0.010 (3)	0.017 (2)	−0.006 (3)
C24	0.062 (4)	0.079 (4)	0.060 (3)	0.008 (3)	0.015 (3)	−0.020 (3)
C25	0.070 (4)	0.100 (5)	0.070 (4)	−0.008 (4)	0.020 (3)	−0.035 (4)
C26	0.132 (5)	0.066 (4)	0.078 (4)	0.000 (4)	0.036 (4)	−0.023 (3)
C27	0.099 (4)	0.075 (5)	0.076 (4)	0.025 (4)	0.011 (3)	−0.015 (3)
C28	0.062 (3)	0.066 (4)	0.062 (3)	0.009 (3)	0.001 (3)	−0.011 (3)
C29	0.049 (3)	0.081 (4)	0.047 (3)	0.013 (3)	0.017 (2)	0.009 (3)
C30	0.069 (3)	0.111 (4)	0.070 (4)	0.015 (3)	0.035 (3)	0.004 (3)
C31	0.080 (4)	0.160 (6)	0.081 (4)	0.025 (4)	0.050 (3)	0.010 (4)
C32	0.067 (4)	0.169 (7)	0.063 (4)	0.001 (4)	0.025 (3)	0.035 (4)
C33	0.074 (4)	0.118 (5)	0.067 (4)	−0.006 (3)	0.016 (3)	0.025 (3)
C34	0.051 (3)	0.113 (4)	0.055 (3)	0.004 (3)	0.018 (2)	0.014 (3)
C35	0.041 (3)	0.067 (3)	0.036 (3)	0.003 (2)	0.014 (2)	−0.002 (2)
C36	0.066 (3)	0.109 (4)	0.054 (4)	−0.015 (3)	0.022 (3)	0.004 (3)
C37	0.088 (4)	0.120 (4)	0.068 (4)	−0.051 (3)	0.010 (3)	−0.008 (3)
C38	0.085 (4)	0.085 (4)	0.052 (4)	−0.013 (3)	−0.001 (3)	−0.009 (3)
C39	0.082 (4)	0.086 (4)	0.045 (3)	−0.013 (3)	0.018 (3)	−0.006 (3)
C40	0.065 (3)	0.077 (3)	0.040 (3)	−0.014 (2)	0.012 (2)	−0.011 (2)
N1	0.082 (3)	0.064 (3)	0.071 (3)	−0.004 (2)	0.026 (2)	0.014 (3)
N2	0.043 (2)	0.054 (3)	0.045 (2)	0.0026 (19)	0.0108 (17)	0.001 (2)
N3	0.102 (3)	0.069 (3)	0.093 (3)	0.028 (3)	0.049 (3)	0.013 (3)
P1	0.0418 (7)	0.0747 (10)	0.0410 (7)	0.0074 (6)	0.0141 (5)	0.0003 (6)

Geometric parameters (Å, °)

C1—N1	1.325 (5)	C21—H21	0.9300
C1—C2	1.386 (6)	C22—P1	1.818 (3)
C1—H1	0.9300	C22—H22A	0.9700
C2—C3	1.358 (6)	C22—H22B	0.9700
C2—H2	0.9300	C23—C24	1.380 (5)
C3—C4	1.373 (5)	C23—C28	1.399 (5)
C3—H3	0.9300	C23—P1	1.790 (4)
C4—C5	1.383 (5)	C24—C25	1.372 (5)
C4—H4	0.9300	C24—H24	0.9300
C5—N1	1.330 (4)	C25—C26	1.381 (6)
C5—C6	1.467 (5)	C25—H25	0.9300
C6—N2	1.341 (4)	C26—C27	1.374 (6)
C6—C7	1.389 (4)	C26—H26	0.9300
C7—C8	1.398 (4)	C27—C28	1.372 (5)
C7—H7	0.9300	C27—H27	0.9300
C8—C9	1.390 (4)	C28—H28	0.9300
C8—C16	1.492 (5)	C29—C34	1.365 (5)
C9—C10	1.399 (4)	C29—C30	1.384 (5)
C9—H9	0.9300	C29—P1	1.779 (4)
C10—N2	1.344 (4)	C30—C31	1.385 (6)
C10—C11	1.480 (5)	C30—H30	0.9300
C11—N3	1.336 (4)	C31—C32	1.344 (6)

C11—C12	1.380 (5)	C31—H31	0.9300
C12—C13	1.373 (5)	C32—C33	1.379 (6)
C12—H12	0.9300	C32—H32	0.9300
C13—C14	1.358 (5)	C33—C34	1.397 (5)
C13—H13	0.9300	C33—H33	0.9300
C14—C15	1.364 (6)	C34—H34	0.9300
C14—H14	0.9300	C35—C36	1.373 (5)
C15—N3	1.338 (5)	C35—C40	1.380 (4)
C15—H15	0.9300	C35—P1	1.787 (4)
C16—C21	1.385 (4)	C36—C37	1.390 (5)
C16—C17	1.386 (4)	C36—H36	0.9300
C17—C18	1.381 (4)	C37—C38	1.346 (5)
C17—H17	0.9300	C37—H37	0.9300
C18—C19	1.377 (4)	C38—C39	1.364 (5)
C18—H18	0.9300	C38—H38	0.9300
C19—C20	1.382 (5)	C39—C40	1.376 (5)
C19—C22	1.524 (4)	C39—H39	0.9300
C20—C21	1.387 (5)	C40—H40	0.9300
C20—H20	0.9300		
N1—C1—C2	124.6 (5)	C19—C22—H22B	108.8
N1—C1—H1	117.7	P1—C22—H22B	108.8
C2—C1—H1	117.7	H22A—C22—H22B	107.7
C3—C2—C1	118.1 (5)	C24—C23—C28	119.1 (4)
C3—C2—H2	121.0	C24—C23—P1	121.8 (4)
C1—C2—H2	121.0	C28—C23—P1	119.0 (4)
C2—C3—C4	118.3 (5)	C25—C24—C23	119.3 (5)
C2—C3—H3	120.8	C25—C24—H24	120.4
C4—C3—H3	120.8	C23—C24—H24	120.4
C3—C4—C5	120.1 (5)	C24—C25—C26	121.7 (5)
C3—C4—H4	120.0	C24—C25—H25	119.1
C5—C4—H4	120.0	C26—C25—H25	119.1
N1—C5—C4	122.1 (4)	C27—C26—C25	119.1 (5)
N1—C5—C6	116.5 (4)	C27—C26—H26	120.5
C4—C5—C6	121.4 (5)	C25—C26—H26	120.5
N2—C6—C7	121.6 (4)	C28—C27—C26	120.0 (5)
N2—C6—C5	116.2 (4)	C28—C27—H27	120.0
C7—C6—C5	122.2 (4)	C26—C27—H27	120.0
C6—C7—C8	120.7 (4)	C27—C28—C23	120.8 (4)
C6—C7—H7	119.7	C27—C28—H28	119.6
C8—C7—H7	119.7	C23—C28—H28	119.6
C9—C8—C7	116.5 (4)	C34—C29—C30	118.4 (4)
C9—C8—C16	120.8 (4)	C34—C29—P1	119.9 (3)
C7—C8—C16	122.6 (4)	C30—C29—P1	121.5 (4)
C8—C9—C10	120.5 (4)	C29—C30—C31	120.7 (5)
C8—C9—H9	119.8	C29—C30—H30	119.6
C10—C9—H9	119.8	C31—C30—H30	119.6
N2—C10—C9	121.4 (4)	C32—C31—C30	119.5 (5)
N2—C10—C11	117.2 (4)	C32—C31—H31	120.3
C9—C10—C11	121.3 (4)	C30—C31—H31	120.3

N3—C11—C12	122.0 (4)	C31—C32—C33	122.1 (5)
N3—C11—C10	115.8 (4)	C31—C32—H32	118.9
C12—C11—C10	122.1 (4)	C33—C32—H32	118.9
C13—C12—C11	119.2 (4)	C32—C33—C34	117.6 (5)
C13—C12—H12	120.4	C32—C33—H33	121.2
C11—C12—H12	120.4	C34—C33—H33	121.2
C14—C13—C12	119.5 (5)	C29—C34—C33	121.7 (4)
C14—C13—H13	120.2	C29—C34—H34	119.2
C12—C13—H13	120.2	C33—C34—H34	119.2
C13—C14—C15	117.9 (5)	C36—C35—C40	118.2 (4)
C13—C14—H14	121.1	C36—C35—P1	121.1 (3)
C15—C14—H14	121.1	C40—C35—P1	120.7 (3)
N3—C15—C14	124.5 (5)	C35—C36—C37	120.6 (4)
N3—C15—H15	117.7	C35—C36—H36	119.7
C14—C15—H15	117.7	C37—C36—H36	119.7
C21—C16—C17	117.9 (4)	C38—C37—C36	120.2 (5)
C21—C16—C8	120.6 (4)	C38—C37—H37	119.9
C17—C16—C8	121.5 (4)	C36—C37—H37	119.9
C18—C17—C16	121.2 (4)	C37—C38—C39	120.0 (4)
C18—C17—H17	119.4	C37—C38—H38	120.0
C16—C17—H17	119.4	C39—C38—H38	120.0
C19—C18—C17	120.3 (4)	C38—C39—C40	120.4 (4)
C19—C18—H18	119.9	C38—C39—H39	119.8
C17—C18—H18	119.9	C40—C39—H39	119.8
C18—C19—C20	119.4 (4)	C39—C40—C35	120.6 (4)
C18—C19—C22	121.3 (4)	C39—C40—H40	119.7
C20—C19—C22	119.2 (4)	C35—C40—H40	119.7
C19—C20—C21	119.9 (4)	C1—N1—C5	116.7 (4)
C19—C20—H20	120.0	C6—N2—C10	119.2 (4)
C21—C20—H20	120.0	C11—N3—C15	116.8 (4)
C16—C21—C20	121.2 (4)	C29—P1—C35	109.4 (2)
C16—C21—H21	119.4	C29—P1—C23	108.95 (19)
C20—C21—H21	119.4	C35—P1—C23	107.97 (19)
C19—C22—P1	113.6 (2)	C29—P1—C22	109.69 (18)
C19—C22—H22A	108.8	C35—P1—C22	110.46 (17)
P1—C22—H22A	108.8	C23—P1—C22	110.30 (19)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C22—H22B···Br1	0.97	2.98	3.938 (4)	171
C24—H24···Br1^{i}	0.93	2.83	3.707 (5)	158

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

Fig. 1

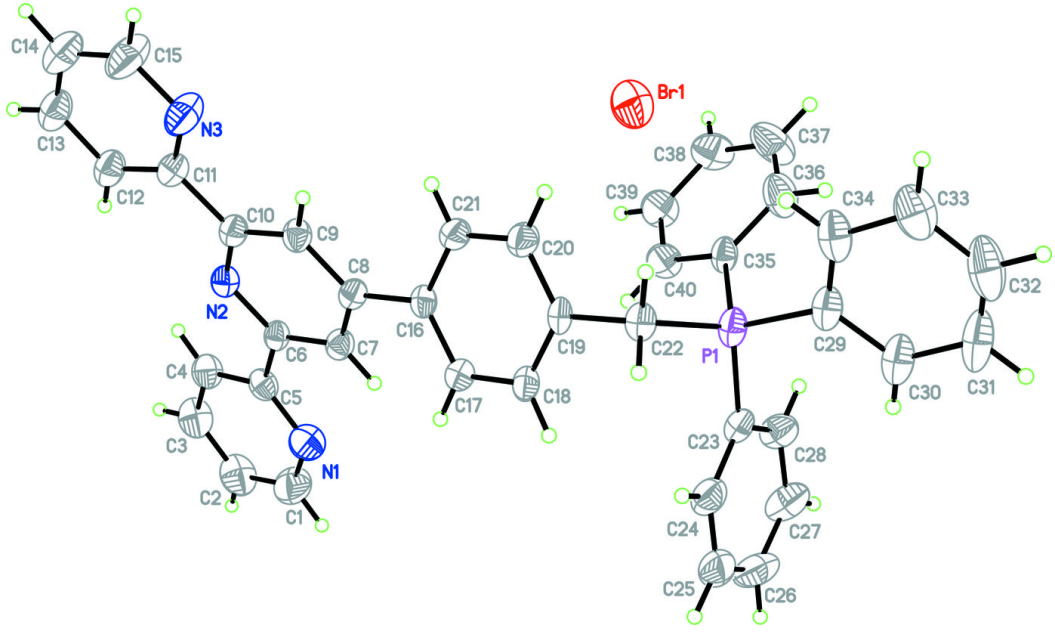
C14
C15
N3
C13
C11
C12
C10
C9
C8
N2
C6
C7
C16
C21
C20
C19
C22
C4
C5
C3
N1
C2
C1
C17
C18
Br1
C37
C38
C36
C39
C34
C33
C35
C40
C32
P1
C29
C30
C31
C23
C28
C24
C27
C25
C26

Fig. 2