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bonding; halogen bonding; molecular sheets.**CCDC references:** 1893497; 971522**Supporting information:** this article has  
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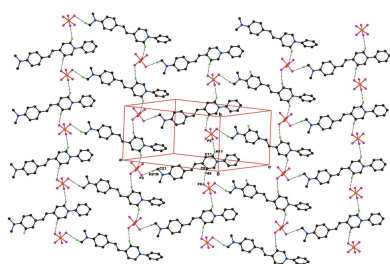
# Crystal structure determination of two pyridine derivatives: 4-[(*E*)-2-(4-methoxyphenyl)ethenyl]-1-methylpyridin-1-ium hexafluoro- $\lambda^6$ -phosphane and 4-[(*E*)-2-[4-(dimethylamino)phenyl]ethenyl]-1-phenyl-1 $\lambda^5$ -pyridin-1-ylum hexafluoro- $\lambda^6$ -phosphane

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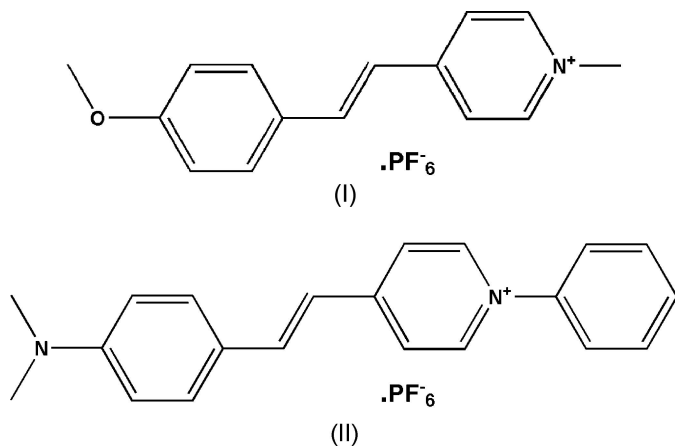
The title molecular salts, C<sub>16</sub>H<sub>16</sub>NO<sup>+</sup>·PF<sub>6</sub><sup>-</sup>, (I), and C<sub>21</sub>H<sub>21</sub>N<sub>2</sub><sup>+</sup>·PF<sub>6</sub><sup>-</sup>, (II), are pyridine derivatives. In compound (I), the cation comprises a methyl N-substituted pyridine ring and a methoxy-substituted benzene ring connected by a C=C double bond. The F atoms of the PF<sub>6</sub><sup>-</sup> anion are disordered over two sets of sites with refined occupancy factors of 0.614 (7):0.386 (7). In compound (II), the cation comprises a pyridine ring attached to unsubstituted phenyl ring and a dimethylaniline ring, which are connected by a C=C double bond. The anion is PF<sub>6</sub><sup>-</sup>. In both salts, the cation adopts an *E* configuration with respect to the C=C bond. The pyridine ring makes a dihedral angle of 9.86 (12)° with the methoxy-substituted benzene ring in compound (I) and 11.2 (3)° with the dimethylamine-substituted benzene ring in compound (II). In compound (I), the crystal packing is stabilized by weak C—H···F intermolecular interactions which result in R<sub>3</sub><sup>4</sup>(14) ring motifs, forming molecular sheets running parallel to (103). These are further stabilized by weak P—F··· $\pi$  interactions. In compound (II), the crystal packing is stabilized by C—H···F interactions, which result in R<sub>6</sub><sup>6</sup>(40) ring motifs, forming molecular sheets running parallel to (101) and these are further connected by  $\pi$ – $\pi$  interactions.

## 1. Chemical context

Stilbene-based compounds are the basic element for a number of biologically active natural and synthetic compounds. These compounds have a wide range of biological activities including anti-inflammatory, anticancer, antiviral, antioxidant and more recently neuroprotective effect (Giacomini *et al.*, 2016). Pyridine and its derivatives play an important role in developing anticancer drugs (Ghattas *et al.*, 2017) and show antibacterial activities (Chanawanno *et al.*, 2010). Pyridine is the parent ring system of a large number of naturally occurring products and important industrial, pharmaceutical and agricultural chemicals. Pyridine derivatives have also shown antichagasic activity against Chagas disease, a parasitic infection caused by *Trypanosoma cruzi*, a parasite that is widely spread in central and South America (Dorigo *et al.*, 1993). The title compounds have been tested for *in vitro* cytotoxicity and anticancer activity, using VERO and MCF-7 (breast cancer) cell lines, respectively. The cells were maintained in minimal essential



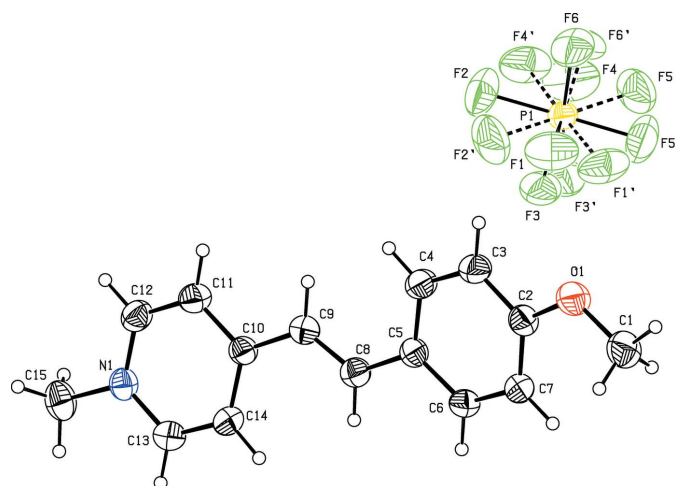
medium supplemented with 10% FBS, penicillin (100 U ml<sup>-1</sup>), and streptomycin (100 microgram ml<sup>-1</sup>) in a humidified atmosphere of 50 microgram ml<sup>-1</sup> CO<sub>2</sub> at 310 K.



## 2. Structural commentary

The molecular structures of the title pyridine derivatives [C<sub>16</sub>H<sub>16</sub>NO<sup>+</sup>·PF<sub>6</sub><sup>-</sup>], (I) and [C<sub>21</sub>H<sub>21</sub>N<sub>2</sub><sup>+</sup>·PF<sub>6</sub><sup>-</sup>], (II), are shown in Figs. 1 and 2, respectively. In compound (I), the cation comprises a methyl N-substituted pyridine ring (N1/C10–C14) and a methoxy-substituted phenyl ring (C2–C7) connected by the C8=C9 bond. The F atoms of the PF<sub>6</sub><sup>-</sup> anion are disordered over two sets of sites with refined occupancy factors of 0.614 (7):0.386 (7). In compound (II), the cation comprises a pyridine ring (N2/C7–C11) attached to an unsubstituted phenyl ring (C1–C6) and a dimethylamino-substituted phenyl ring (C14–C19), connected by the C12=C13 bond. A PF<sub>6</sub><sup>-</sup> anion is also present.

In both compounds, the cations adopt an *E* configuration with respect to the C=C bond [C8=C9 = 1.312 (4) Å in compound (I) and C12=C13 = 1.348 (8) Å in compound (II)].



**Figure 1**  
The molecular structure of the title compound (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. In the anion, dashed bonds indicate the minor disorder component.

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1C···F2 <sup>i</sup>	0.96	2.51	3.220 (8)	131
C12—H12···F3 <sup>ii</sup>	0.93	2.60	3.509 (8)	165
C12—H12···F3 <sup>iii</sup>	0.93	2.53	3.454 (16)	176
C13—H13···F5 <sup>iii</sup>	0.93	2.40	3.270 (9)	156
C15—H15A···F4 <sup>iii</sup>	0.96	2.53	3.443 (7)	160
C15—H15B···F2 <sup>iv</sup>	0.96	2.46	3.235 (5)	138
C15—H15B···F5 <sup>v</sup>	0.96	2.49	3.162 (7)	127

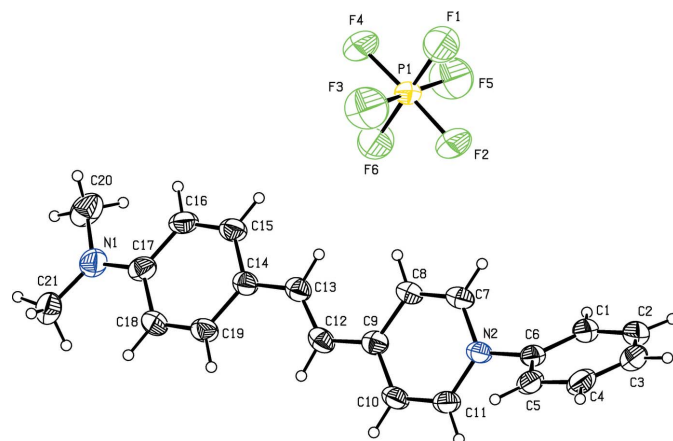
Symmetry codes: (i)  $-x + 1, y - \frac{1}{2}, -z + 1$ ; (ii)  $x - 2, y, z - 1$ ; (iii)  $x - 2, y - 1, z - 1$ ; (iv)  $-x - 1, y - \frac{1}{2}, -z$ ; (v)  $-x, y - \frac{1}{2}, -z$ .

The pyridine ring (N1/C10–C14) makes a dihedral angle of 9.86 (12)<sup>o</sup> with methoxy-substituted benzene ring (C2–C7) in compound (I) whereas in compound (II) the pyridine ring (N2/C7–C11) makes a dihedral angle of 11.2 (3)<sup>o</sup> with dimethylamine-substituted benzene ring (C14–C19). The pyridine ring in compound (II) is inclined to the unsubstituted phenyl ring (C1–C6) by 54.9 (3)<sup>o</sup>. The methoxy group oxygen atom O1 of compound (I) deviates from the benzene ring to which it is attached by 0.0317 (1) Å while the methyl group carbon atom C15 deviates from the benzene ring to which it is attached by 0.022 (3) Å. In compound (II), the methylamine group nitrogen atom (N1) deviates from the benzene ring to which it is attached by 0.017 (5) Å.

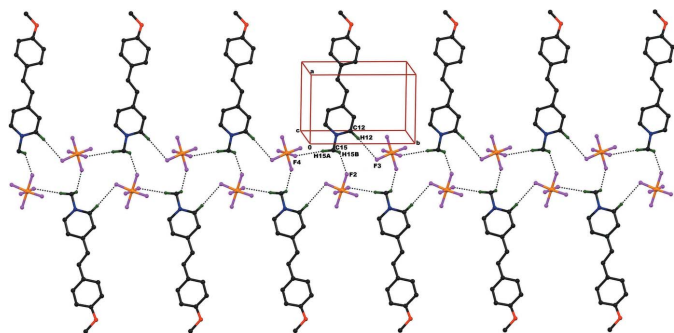
In compound (I), the methoxy group is (+) anti-periplanar to the phenyl ring (C2–C7), as is evident from the torsion angle C3–C2–O1–C1 of 178.2 (3)<sup>o</sup>. In compound II, the methylamine group is (–) anti-periplanar to the phenyl ring (C14–C19), which is evident from the torsion angle C16–C17–N1–C21 of –173.9 (5)<sup>o</sup>.

## 3. Supramolecular features

In the crystal packing of compound (I), the molecules are linked *via* intermolecular C12–H12···F3(–2 + x, y, –1 + z), C15–H15A···F4(–2 + x, –1 + y, –1 + z) and C15–



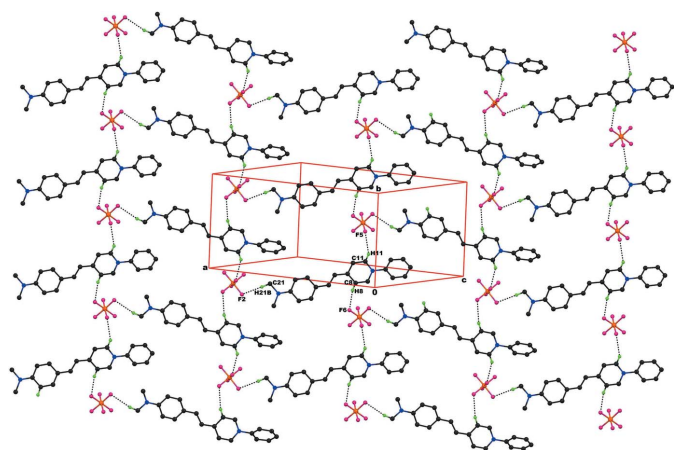
**Figure 2**  
The molecular structure of the title compound (II) with the atom-numbering scheme. Displacement ellipsoids are drawn at 30% probability level.


**Figure 3**

The crystal packing of the title compound (I), viewed along the  $a$  axis, showing C—H...F intermolecular interactions, resulting in  $R_3^2(14)$  ring motifs, which form two-dimensional molecular sheets running parallel to (103). Hydrogen atoms not involved in hydrogen bonding have been omitted for clarity.

H15B...F2( $-1-x, -\frac{1}{2}+y, z$ ) interactions (Table 1), resulting in  $R_3^2(14)$  ring motifs, which form molecular sheets lying parallel to (103) (Fig. 3). The crystal packing is further stabilized by P1—F4...Cg1( $-x, \frac{1}{2}+y, -z$ ) halogen-bond (XB) interactions, where Cg1 is the centroid of the pyridine ring (N1/C10—C14).

In the crystal packing of compound (II), intramolecular C8—H8...F6 and intermolecular C11—H11...F5( $x, 1+y, z$ ) and C21—H21B...F2( $\frac{1}{2}+x, -\frac{1}{2}-y, -\frac{1}{2}+z$ ) interactions (Table 2) result in  $R_6^6(40)$  ring motifs and form molecular sheets lying parallel to (101) (Fig. 4). These molecular sheets are cross-linked by C16—H16...F4( $x, -1-y, -\frac{1}{2}+z$ ) interactions, resulting in a three-dimensional network. The crystal packing is further stabilized by Cg1...Cg3( $x, -y, -\frac{1}{2}+z$ ) interactions [centroid-centroid distance = 3.646 (4) Å and interplanar distance = 3.397 (2) Å], where Cg1 is the centroid of the pyridine ring (N2/C7—C11) and Cg3 is the centroid of the phenyl ring (C14—C19).


**Figure 4**

The crystal packing of the title compound (II), viewed along the  $b$  axis, showing intermolecular C—H...F interactions, resulting in  $R_6^6(40)$  ring motifs, which form molecular sheets lying parallel to (101). Hydrogen atoms not involved in hydrogen bonding have been omitted for clarity.

**Table 2**

Hydrogen-bond geometry (Å, °) for (II).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C8—H8...F6	0.93	2.59	3.486 (8)	162
C11—H11...F5 <sup>i</sup>	0.93	2.55	3.363 (8)	146
C16—H16...F4 <sup>ii</sup>	0.93	2.59	3.289 (7)	132
C21—H21B...F2 <sup>iii</sup>	0.96	2.64	3.516 (8)	152

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

#### 4. Database survey

A search of the Cambridge Structural Database (CSD, V5.39, latest update August 2018; Groom *et al.*, 2016) found no entry for a hexafluoro- $\lambda^6$ -phosphane with pyridine derivatives. However, the cationic structures of substituted pyridine derivatives were found, for example, *r*-1,*t*-3-bis[4-(dimethylamino)phenyl]-*c*-2,*t*-4-bis(pyridin-4-yl)cyclobutane (Zhang & Zhuang, 2014) and 4'-hydroxy-3'-methoxy-*N*-methyl-4-stilbazolium tosylate hydrate (Zhang *et al.*, 1997).

#### 5. Synthesis and crystallization

##### Compound (I)

A solution of *N*-phenyl-4-picolinium chloride (250 mg, 1.10 mmol), 4-(dimethylamino) benzaldehyde (363 mg, 2.4 mmol), and piperidine (4 drops) in methanol (20 ml) was heated under reflux for 4 h. The addition of diethyl ether to the deep-red solution yielded a dark precipitate, which was filtered, washed with diethyl ether and dried. This crude chloride salt was metathesized to dimethylamino *N*-phenyl stilbazolium hexafluoro phosphate (DAPSH) by precipitation from water/aqueous  $\text{NH}_4\text{PF}_6$ . A supersaturated solution of DAPSH was prepared using acetonitrile as solvent and the solution was filtered into the growth vessel for slow evaporation by covering the vessel with a perforated sheet. Good quality greenish crystals of compound (I) was grown in a period of 15–25 days.

##### Compound (II)

Compound (II) was synthesized by the condensation of 1,4-dimethylpyridinium iodide (2.35 g, 10 mmol), methanol (30 ml) and 4-methoxybenzaldehyde (1.36 g, 10 mmol) in the presence of piperidine (0.2 ml). The total mixture was taken in the round-bottom flask (1000 ml capacity) of a Dean–Stark apparatus and refluxed for 1 d and cooled to room temperature. The product 4-methoxy-*N*-methyl-4-stilbazolium iodide was filtered and recrystallized from methanol. This product (0.706 g, 2 mmol) was dissolved in 70 ml of millipore water and simultaneously sodium hexafluorophosphate (0.338 g, 2 mmol) was dissolved in 30 ml of millipore water by heating at 343 K. Both the solutions were stirred for 3 h and mixed. 4-Methoxy-*N*-methylstilbazolium hexafluorophosphate (MMSHP) was formed as a yellowish precipitate. A solution of MMSHP and aqueous acetone was prepared with 14.4 g of MMSHP in 200 ml of acetone–water mixed solvent (5:1) and stirred. The clear solution was collected in the growth vessel after filtering it by using 0.2 micrometer porosity millipore

**Table 3**  
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	C <sub>15</sub> H <sub>16</sub> NO <sup>+</sup> ·PF <sub>6</sub> <sup>-</sup>	C <sub>21</sub> H <sub>21</sub> N <sub>2</sub> <sup>+</sup> ·PF <sub>6</sub> <sup>-</sup>
<i>M<sub>r</sub></i>	371.26	446.37
Crystal system, space group	Monoclinic, <i>P</i> 2 <sub>1</sub>	Monoclinic, <i>Cc</i>
Temperature (K)	296	296
<i>a</i> , <i>b</i> , <i>c</i> (Å)	6.4320 (2), 9.3645 (3), 13.6070 (5)	19.4596 (14), 10.7416 (8), 11.9654 (9)
$\beta$ (°)	101.868 (2)	125.864 (2)
<i>V</i> (Å <sup>3</sup> )	802.06 (5)	2026.9 (3)
<i>Z</i>	2	4
Radiation type	Mo <i>K</i> $\alpha$	Mo <i>K</i> $\alpha$
$\mu$ (mm <sup>-1</sup> )	0.24	0.20
Crystal size (mm)	0.35 × 0.30 × 0.30	0.35 × 0.30 × 0.30
Data collection		
Diffractometer	Bruker Kappa APEXII CCD	Bruker Kappa APEXII CCD
Absorption correction	Multi-scan ( <i>SADABS</i> ; Bruker, 2008)	Multi-scan ( <i>SADABS</i> ; Bruker, 2008)
<i>T<sub>min</sub></i> , <i>T<sub>max</sub></i>	0.921, 0.932	0.933, 0.943
No. of measured, independent and observed [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )] reflections	8106, 2867, 2606	13796, 3926, 2895
<i>R<sub>int</sub></i>	0.022	0.024
( <i>sin</i> $\theta$ / $\lambda$ ) <sub>max</sub> (Å <sup>-1</sup> )	0.617	0.617
Refinement		
<i>R</i> [ <i>F</i> <sup>2</sup> > 2 $\sigma$ ( <i>F</i> <sup>2</sup> )], <i>wR</i> ( <i>F</i> <sup>2</sup> ), <i>S</i>	0.032, 0.084, 1.02	0.064, 0.203, 1.07
No. of reflections	2867	3926
No. of parameters	273	255
No. of restraints	140	65
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$ , $\Delta\rho_{\min}$ (e Å <sup>-3</sup> )	0.15, -0.14	0.49, -0.41
Absolute structure	Flack (1983), 1198 Friedel pairs	Flack (1983), 1927 Friedel pairs
Absolute structure parameter	0.08 (11)	0.5 (2)

Computer programs: *APEX2* and *SAINT* (Bruker, 2008), *SHELXS97* and *SHELXL97* (Sheldrick, 2008), *ORTEP-3 for Windows* (Farrugia, 2012), *Mercury* (Macrae *et al.*, 2008), and *PLATON* (Spek, 2009).

filters and the solvent was allowed to evaporate slowly at room temperature. After three weeks, yellowish crystals of compound (II) were harvested.

## 6. Refinement

Crystal data, data collection and structure refinement details for compounds (I) and (II) are summarized in Table 3. The positions of the hydrogen atoms were localized from the difference-electron-density maps and their distances were geometrically constrained. The hydrogen atoms bound to the C atoms were treated as riding atoms, with *d*(C–H) = 0.93 and 0.96 Å for aryl and methyl H atoms, respectively, with *U*<sub>iso</sub>(H) = 1.5*U*<sub>eq</sub>(methyl C) and 1.2*U*<sub>eq</sub>(non-methyl C). The rotation angles for methyl groups were optimized by least squares.

## Acknowledgements

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## References

- Bruker (2008). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Chanawanno, K., Chantrapromma, S., Anantapong, T., Kanjana-Opas, A. & Fun, H.-K. (2010). *Eur. J. Med. Chem.* **45**, 4199–4208.
- Dorigo, P., Gaion, R. M., Belluco, P., Fraccarollo, D., Maragno, I., Bombieri, G., Benetollo, F., Mosti, L. & Orsini, F. (1993). *J. Med. Chem.* **36**, 2475–2484.
- Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Ghaffar, A.-E.-B. A. G., Khodairy, A., Moustafa, H. M., Hussein, B. R. M., Farghaly, M. M. & Aboelez, M. O. (2017). *Pharma. Chem. J.* **30**, 652–660.
- Giacomini, E., Rupiani, S., Guidotti, L., Recanatini, M. & Roberti, M. (2016). *Curr. Med. Chem.* **23**, 2439–2489.
- Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* **B72**, 171–179.
- Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
- Zhang, D.-C., Zhang, T.-Z., Zhang, Y.-Q., Fei, Z.-H. & Yu, K.-B. (1997). *Acta Cryst.* **C53**, 364–365.
- Zhang, S. & Zhuang, J. (2014). *Acta Cryst.* **E70**, o311.

## supporting information

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## Crystal structure determination of two pyridine derivatives: 4-[(*E*)-2-(4-methoxyphenyl)ethenyl]-1-methylpyridin-1-ium hexafluoro- $\lambda^6$ -phosphane and 4-[(*E*)-2-[4-(dimethylamino)phenyl]ethenyl]-1-phenyl-1 $\lambda^5$ -pyridin-1-ylum hexafluoro- $\lambda^6$ -phosphane

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### Computing details

For both structures, data collection: *APEX2* (Bruker, 2008); cell refinement: *APEX2* (Bruker, 2008); data reduction: *SAINTE* (Bruker, 2008); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

### 4-[(*E*)-2-(4-Methoxyphenyl)ethenyl]-1-methylpyridin-1-ium hexafluoro- $\lambda^6$ -phosphane (I)

#### Crystal data

$C_{15}H_{16}NO^+ \cdot PF_6^-$   
 $M_r = 371.26$   
 Monoclinic,  $P2_1$   
 Hall symbol: P 2yb  
 $a = 6.4320$  (2) Å  
 $b = 9.3645$  (3) Å  
 $c = 13.6070$  (5) Å  
 $\beta = 101.868$  (2)°  
 $V = 802.06$  (5) Å<sup>3</sup>  
 $Z = 2$

$F(000) = 380$   
 $D_x = 1.537$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å  
 Cell parameters from 2867 reflections  
 $\theta = 2.7$ – $26.0$ °  
 $\mu = 0.24$  mm<sup>-1</sup>  
 $T = 296$  K  
 Block, green  
 $0.35 \times 0.30 \times 0.30$  mm

#### Data collection

Bruker Kappa APEXII CCD  
 diffractometer  
 Radiation source: fine-focus sealed tube  
 Graphite monochromator  
 $\omega$  &  $\varphi$  scans  
 Absorption correction: multi-scan  
 (SADABS; Bruker, 2008)  
 $T_{\min} = 0.921$ ,  $T_{\max} = 0.932$

8106 measured reflections  
 2867 independent reflections  
 2606 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.022$   
 $\theta_{\max} = 26.0$ °,  $\theta_{\min} = 2.7$ °  
 $h = -7 \rightarrow 7$   
 $k = -9 \rightarrow 11$   
 $l = -16 \rightarrow 16$

*Refinement*Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.032$  $wR(F^2) = 0.084$  $S = 1.02$ 

2867 reflections

273 parameters

140 restraints

Primary atom site location: structure-invariant  
direct methodsSecondary atom site location: difference Fourier  
mapHydrogen site location: inferred from  
neighbouring sitesH atoms treated by a mixture of independent  
and constrained refinement $w = 1/[\sigma^2(F_o^2) + (0.0459P)^2 + 0.106P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\max} < 0.001$  $\Delta\rho_{\max} = 0.15 \text{ e } \text{\AA}^{-3}$  $\Delta\rho_{\min} = -0.14 \text{ e } \text{\AA}^{-3}$ 

Extinction correction: SHELXL,

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

Extinction coefficient: 0.016 (3)

Absolute structure: Flack (1983), 1198 Friedel  
pairs

Absolute structure parameter: 0.08 (11)

*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  $F^2 > 2\sigma(F^2)$  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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
C1	0.6868 (5)	0.3750 (4)	0.1233 (2)	0.0795 (9)	
H1A	0.7984	0.4199	0.1710	0.119*	
H1B	0.7430	0.3396	0.0679	0.119*	
H1C	0.6290	0.2971	0.1550	0.119*	
C2	0.3506 (4)	0.4329 (3)	0.02002 (19)	0.0562 (6)	
C3	0.1982 (4)	0.5355 (3)	-0.0083 (2)	0.0638 (7)	
H3	0.2204	0.6270	0.0182	0.077*	
C4	0.0127 (4)	0.5051 (3)	-0.07556 (19)	0.0605 (6)	
H4	-0.0885	0.5764	-0.0937	0.073*	
C5	-0.0260 (4)	0.3690 (3)	-0.11688 (16)	0.0494 (6)	
C6	0.1323 (4)	0.2680 (3)	-0.08913 (18)	0.0559 (6)	
H6	0.1122	0.1769	-0.1167	0.067*	
C7	0.3206 (4)	0.2978 (3)	-0.0214 (2)	0.0579 (7)	
H7	0.4246	0.2281	-0.0042	0.069*	
C8	-0.2243 (4)	0.3294 (4)	-0.18350 (15)	0.0536 (5)	
H8	-0.2377	0.2349	-0.2049	0.064*	
C9	-0.3863 (4)	0.4140 (3)	-0.21639 (19)	0.0573 (6)	
H9	-0.3690	0.5098	-0.1986	0.069*	
C10	-0.5907 (4)	0.3729 (3)	-0.27799 (17)	0.0500 (6)	
C11	-0.7440 (4)	0.4771 (3)	-0.3090 (2)	0.0601 (6)	
H11	-0.7139	0.5720	-0.2915	0.072*	
C12	-0.9364 (5)	0.4417 (3)	-0.3646 (2)	0.0613 (7)	

H12	-1.0367	0.5131	-0.3844	0.074*	
C13	-0.8419 (5)	0.2036 (3)	-0.3640 (2)	0.0593 (7)	
H13	-0.8759	0.1099	-0.3836	0.071*	
C14	-0.6466 (5)	0.2330 (3)	-0.3077 (2)	0.0588 (7)	
H14	-0.5494	0.1594	-0.2888	0.071*	
C15	-1.1961 (4)	0.2735 (4)	-0.4540 (2)	0.0741 (8)	
H15A	-1.2061	0.1728	-0.4675	0.111*	
H15B	-1.2142	0.3250	-0.5162	0.111*	
H15C	-1.3048	0.3012	-0.4189	0.111*	
N1	-0.9864 (3)	0.3063 (2)	-0.39178 (14)	0.0543 (6)	
O1	0.5245 (3)	0.4760 (2)	0.08788 (15)	0.0736 (6)	
P1	0.74137 (10)	0.83995 (8)	0.65677 (4)	0.05309 (18)	
F1	0.7553 (11)	0.7695 (5)	0.7626 (3)	0.1143 (17)	0.614 (7)
F2	0.5074 (6)	0.8863 (7)	0.6568 (4)	0.1075 (19)	0.614 (7)
F3	0.6535 (12)	0.6952 (7)	0.6034 (6)	0.0961 (19)	0.614 (7)
F4	0.7210 (13)	0.9108 (6)	0.5533 (3)	0.128 (2)	0.614 (7)
F5	0.9725 (6)	0.7933 (8)	0.6592 (6)	0.125 (2)	0.614 (7)
F6	0.800 (3)	0.9748 (11)	0.7254 (10)	0.109 (4)	0.614 (7)
F1'	0.8909 (17)	0.7393 (7)	0.7289 (7)	0.112 (3)	0.386 (7)
F2'	0.5512 (16)	0.7924 (12)	0.6982 (8)	0.140 (4)	0.386 (7)
F3'	0.705 (2)	0.7182 (14)	0.5738 (8)	0.101 (3)	0.386 (7)
F4'	0.5990 (17)	0.9410 (8)	0.5792 (9)	0.127 (4)	0.386 (7)
F5'	0.9347 (16)	0.8884 (11)	0.6106 (7)	0.120 (3)	0.386 (7)
F6'	0.830 (4)	0.9820 (15)	0.7120 (14)	0.084 (4)	0.386 (7)

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0625 (16)	0.083 (3)	0.0841 (18)	-0.0033 (15)	-0.0058 (14)	0.0087 (17)
C2	0.0537 (15)	0.0581 (17)	0.0568 (14)	-0.0038 (12)	0.0116 (11)	0.0032 (12)
C3	0.0628 (16)	0.0509 (17)	0.0749 (16)	-0.0001 (12)	0.0080 (13)	-0.0089 (13)
C4	0.0593 (15)	0.0527 (16)	0.0669 (14)	0.0059 (12)	0.0070 (12)	-0.0023 (13)
C5	0.0503 (12)	0.0519 (17)	0.0473 (10)	-0.0017 (10)	0.0130 (9)	0.0013 (10)
C6	0.0586 (15)	0.0464 (14)	0.0637 (14)	-0.0030 (12)	0.0145 (12)	-0.0061 (12)
C7	0.0516 (14)	0.0518 (18)	0.0697 (15)	0.0023 (10)	0.0114 (11)	0.0056 (12)
C8	0.0580 (12)	0.0529 (15)	0.0515 (11)	0.0006 (13)	0.0147 (10)	0.0023 (13)
C9	0.0562 (14)	0.0503 (15)	0.0651 (13)	-0.0013 (11)	0.0116 (11)	0.0012 (12)
C10	0.0523 (13)	0.0486 (19)	0.0512 (11)	-0.0008 (10)	0.0157 (9)	0.0026 (10)
C11	0.0652 (17)	0.0521 (16)	0.0623 (14)	0.0028 (13)	0.0113 (12)	-0.0033 (13)
C12	0.0594 (16)	0.0594 (19)	0.0648 (15)	0.0099 (13)	0.0123 (13)	0.0080 (13)
C13	0.0645 (16)	0.0513 (17)	0.0620 (14)	-0.0023 (13)	0.0129 (13)	-0.0010 (13)
C14	0.0569 (16)	0.0503 (17)	0.0700 (16)	0.0082 (12)	0.0144 (13)	0.0036 (13)
C15	0.0552 (15)	0.097 (2)	0.0656 (15)	-0.0093 (15)	0.0013 (12)	0.0037 (16)
N1	0.0497 (11)	0.0629 (19)	0.0507 (10)	-0.0013 (9)	0.0112 (8)	0.0030 (9)
O1	0.0622 (11)	0.0680 (14)	0.0816 (12)	-0.0055 (10)	-0.0061 (9)	-0.0044 (11)
P1	0.0518 (3)	0.0467 (3)	0.0587 (3)	0.0013 (3)	0.0066 (2)	-0.0026 (3)
F1	0.172 (5)	0.089 (3)	0.079 (2)	0.006 (3)	0.017 (3)	0.028 (2)
F2	0.0611 (19)	0.140 (4)	0.113 (3)	0.024 (2)	-0.0018 (19)	-0.038 (3)

F3	0.095 (4)	0.068 (3)	0.116 (4)	-0.008 (2)	-0.001 (3)	-0.025 (3)
F4	0.213 (6)	0.109 (4)	0.073 (2)	0.015 (4)	0.057 (3)	0.015 (2)
F5	0.063 (2)	0.139 (5)	0.173 (5)	0.018 (2)	0.019 (3)	-0.057 (4)
F6	0.111 (5)	0.091 (6)	0.121 (6)	0.004 (4)	0.018 (4)	-0.057 (5)
F1'	0.136 (6)	0.074 (4)	0.099 (5)	0.026 (4)	-0.037 (4)	0.005 (4)
F2'	0.115 (6)	0.149 (7)	0.182 (7)	-0.035 (5)	0.092 (5)	-0.004 (6)
F3'	0.116 (7)	0.093 (6)	0.092 (5)	-0.020 (4)	0.018 (4)	-0.039 (4)
F4'	0.127 (6)	0.080 (4)	0.136 (7)	0.007 (4)	-0.064 (5)	0.017 (4)
F5'	0.115 (5)	0.120 (6)	0.146 (6)	-0.027 (4)	0.079 (5)	-0.019 (5)
F6'	0.102 (7)	0.056 (5)	0.091 (5)	-0.021 (5)	0.010 (5)	-0.009 (4)

*Geometric parameters (Å, °)*

C1—O1	1.418 (4)	C11—H11	0.9300
C1—H1A	0.9600	C12—N1	1.341 (4)
C1—H1B	0.9600	C12—H12	0.9300
C1—H1C	0.9600	C13—N1	1.338 (3)
C2—O1	1.357 (3)	C13—C14	1.358 (4)
C2—C3	1.370 (4)	C13—H13	0.9300
C2—C7	1.382 (4)	C14—H14	0.9300
C3—C4	1.376 (4)	C15—N1	1.470 (3)
C3—H3	0.9300	C15—H15A	0.9600
C4—C5	1.395 (4)	C15—H15B	0.9600
C4—H4	0.9300	C15—H15C	0.9600
C5—C6	1.385 (4)	P1—F2'	1.515 (6)
C5—C8	1.452 (3)	P1—F4	1.537 (4)
C6—C7	1.391 (4)	P1—F5	1.543 (4)
C6—H6	0.9300	P1—F1'	1.545 (6)
C7—H7	0.9300	P1—F4'	1.566 (6)
C8—C9	1.312 (4)	P1—F2	1.566 (4)
C8—H8	0.9300	P1—F1	1.570 (4)
C9—C10	1.458 (4)	P1—F6	1.570 (8)
C9—H9	0.9300	P1—F5'	1.571 (6)
C10—C11	1.390 (4)	P1—F6'	1.575 (12)
C10—C14	1.396 (4)	P1—F3	1.586 (6)
C11—C12	1.352 (4)	P1—F3'	1.588 (10)
O1—C1—H1A	109.5	F2'—P1—F1'	91.7 (6)
O1—C1—H1B	109.5	F4—P1—F1'	140.3 (5)
H1A—C1—H1B	109.5	F5—P1—F1'	48.9 (4)
O1—C1—H1C	109.5	F2'—P1—F4'	91.4 (6)
H1A—C1—H1C	109.5	F5—P1—F4'	127.8 (6)
H1B—C1—H1C	109.5	F1'—P1—F4'	176.6 (7)
O1—C2—C3	115.2 (3)	F4—P1—F2	88.8 (3)
O1—C2—C7	125.2 (3)	F5—P1—F2	178.8 (4)
C3—C2—C7	119.7 (2)	F1'—P1—F2	129.9 (5)
C2—C3—C4	121.0 (3)	F4'—P1—F2	53.4 (5)
C2—C3—H3	119.5	F2'—P1—F1	55.5 (5)



C4—C3—H3	119.5	F4—P1—F1	178.2 (4)
C3—C4—C5	121.0 (3)	F5—P1—F1	89.4 (4)
C3—C4—H4	119.5	F4'—P1—F1	142.0 (6)
C5—C4—H4	119.5	F2—P1—F1	89.4 (3)
C6—C5—C4	117.0 (2)	F2'—P1—F6	97.4 (7)
C6—C5—C8	119.9 (2)	F4—P1—F6	99.3 (6)
C4—C5—C8	123.1 (2)	F5—P1—F6	95.9 (7)
C5—C6—C7	122.3 (3)	F1'—P1—F6	94.1 (6)
C5—C6—H6	118.8	F4'—P1—F6	87.2 (7)
C7—C6—H6	118.8	F2—P1—F6	83.7 (6)
C2—C7—C6	118.9 (2)	F1—P1—F6	80.3 (6)
C2—C7—H7	120.5	F2'—P1—F5'	178.3 (5)
C6—C7—H7	120.5	F4—P1—F5'	55.6 (4)
C9—C8—C5	126.3 (3)	F1'—P1—F5'	89.4 (5)
C9—C8—H8	116.8	F4'—P1—F5'	87.5 (6)
C5—C8—H8	116.8	F2—P1—F5'	139.4 (5)
C8—C9—C10	126.5 (3)	F1—P1—F5'	126.0 (5)
C8—C9—H9	116.7	F6—P1—F5'	83.8 (6)
C10—C9—H9	116.7	F2'—P1—F6'	108.1 (9)
C11—C10—C14	116.5 (2)	F4—P1—F6'	91.5 (7)
C11—C10—C9	119.2 (2)	F5—P1—F6'	88.7 (10)
C14—C10—C9	124.2 (2)	F1'—P1—F6'	95.5 (8)
C12—C11—C10	120.5 (3)	F4'—P1—F6'	85.1 (8)
C12—C11—H11	119.7	F2—P1—F6'	91.0 (9)
C10—C11—H11	119.7	F1—P1—F6'	88.3 (7)
N1—C12—C11	121.7 (3)	F5'—P1—F6'	73.1 (9)
N1—C12—H12	119.1	F2'—P1—F3	71.4 (4)
C11—C12—H12	119.1	F4—P1—F3	89.7 (3)
N1—C13—C14	121.5 (3)	F5—P1—F3	91.1 (3)
N1—C13—H13	119.3	F1'—P1—F3	83.5 (4)
C14—C13—H13	119.3	F4'—P1—F3	96.0 (4)
C13—C14—C10	120.4 (3)	F2—P1—F3	89.1 (3)
C13—C14—H14	119.8	F1—P1—F3	90.5 (3)
C10—C14—H14	119.8	F6—P1—F3	168.4 (6)
N1—C15—H15A	109.5	F5'—P1—F3	107.5 (4)
N1—C15—H15B	109.5	F6'—P1—F3	178.8 (8)
H15A—C15—H15B	109.5	F2'—P1—F3'	92.4 (5)
N1—C15—H15C	109.5	F4—P1—F3'	71.7 (5)
H15A—C15—H15C	109.5	F5—P1—F3'	79.2 (5)
H15B—C15—H15C	109.5	F1'—P1—F3'	89.7 (6)
C13—N1—C12	119.3 (2)	F4'—P1—F3'	88.5 (7)
C13—N1—C15	121.0 (3)	F2—P1—F3'	101.4 (5)
C12—N1—C15	119.8 (2)	F1—P1—F3'	108.9 (5)
C2—O1—C1	118.6 (3)	F6—P1—F3'	169.4 (7)
F2'—P1—F4	123.0 (5)	F5'—P1—F3'	86.3 (5)
F2'—P1—F5	139.2 (6)	F6'—P1—F3'	158.7 (9)
F4—P1—F5	92.4 (4)		

O1—C2—C3—C4	-179.0 (2)	C8—C9—C10—C14	2.9 (4)
C7—C2—C3—C4	1.8 (4)	C14—C10—C11—C12	0.4 (4)
C2—C3—C4—C5	0.0 (4)	C9—C10—C11—C12	-178.7 (2)
C3—C4—C5—C6	-1.6 (4)	C10—C11—C12—N1	-0.1 (4)
C3—C4—C5—C8	176.3 (2)	N1—C13—C14—C10	-0.6 (4)
C4—C5—C6—C7	1.5 (4)	C11—C10—C14—C13	0.0 (4)
C8—C5—C6—C7	-176.5 (2)	C9—C10—C14—C13	179.0 (2)
O1—C2—C7—C6	179.0 (2)	C14—C13—N1—C12	0.9 (4)
C3—C2—C7—C6	-1.9 (4)	C14—C13—N1—C15	179.1 (3)
C5—C6—C7—C2	0.2 (4)	C11—C12—N1—C13	-0.5 (4)
C6—C5—C8—C9	-179.4 (2)	C11—C12—N1—C15	-178.7 (2)
C4—C5—C8—C9	2.8 (4)	C3—C2—O1—C1	178.2 (3)
C5—C8—C9—C10	-175.8 (2)	C7—C2—O1—C1	-2.7 (4)
C8—C9—C10—C11	-178.1 (2)		

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C1—H1C $\cdots$ F2 <sup>ii</sup>	0.96	2.51	3.220 (8)	131
C12—H12 $\cdots$ F3 <sup>ii</sup>	0.93	2.60	3.509 (8)	165
C12—H12 $\cdots$ F3 <sup>iii</sup>	0.93	2.53	3.454 (16)	176
C13—H13 $\cdots$ F5 <sup>iii</sup>	0.93	2.40	3.270 (9)	156
C15—H15A $\cdots$ F4 <sup>iii</sup>	0.96	2.53	3.443 (7)	160
C15—H15B $\cdots$ F2 <sup>iv</sup>	0.96	2.46	3.235 (5)	138
C15—H15B $\cdots$ F5 <sup>v</sup>	0.96	2.49	3.162 (7)	127

Symmetry codes: (i)  $-x+1, y-1/2, -z+1$ ; (ii)  $x-2, y, z-1$ ; (iii)  $x-2, y-1, z-1$ ; (iv)  $-x-1, y-1/2, -z$ ; (v)  $-x, y-1/2, -z$ .4- $\{(E)-2-[4-(\text{Dimethylamino})\text{phenyl}]\text{ethenyl}\}-1\text{-phenyl-}1\lambda^5\text{-pyridin-}1\text{-ylium hexafluoro-}\lambda^6\text{-phosphane (II)}$ 

## Crystal data

 $\text{C}_{21}\text{H}_{21}\text{N}_2^+\text{PF}_6^-$  $M_r = 446.37$ Monoclinic,  $Cc$ Hall symbol:  $C -2yc$  $a = 19.4596$  (14)  $\text{\AA}$  $b = 10.7416$  (8)  $\text{\AA}$  $c = 11.9654$  (9)  $\text{\AA}$  $\beta = 125.864$  (2) $^\circ$  $V = 2026.9$  (3)  $\text{\AA}^3$  $Z = 4$  $F(000) = 920$  $D_x = 1.463$   $\text{Mg m}^{-3}$ Mo  $K\alpha$  radiation,  $\lambda = 0.71073$   $\text{\AA}$ 

Cell parameters from 3926 reflections

 $\theta = 2.3\text{--}26.0^\circ$  $\mu = 0.20$   $\text{mm}^{-1}$  $T = 296$  K

Block, yellow

 $0.35 \times 0.30 \times 0.30$  mm

## Data collection

Bruker Kappa APEXII CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 $\varphi$  &  $\omega$  scans

Absorption correction: multi-scan

(SADABS; Bruker, 2008)

 $T_{\min} = 0.933, T_{\max} = 0.943$ 

13796 measured reflections

3926 independent reflections

2895 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.024$  $\theta_{\max} = 26.0^\circ, \theta_{\min} = 2.3^\circ$  $h = -23 \rightarrow 23$  $k = -13 \rightarrow 13$  $l = -14 \rightarrow 14$

*Refinement*Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.064$  $wR(F^2) = 0.203$  $S = 1.07$ 

3926 reflections

255 parameters

65 restraints

Primary atom site location: structure-invariant  
direct methodsSecondary atom site location: difference Fourier  
mapHydrogen site location: inferred from  
neighbouring sitesH atoms treated by a mixture of independent  
and constrained refinement $w = 1/[\sigma^2(F_o^2) + (0.1113P)^2 + 2.2166P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\max} < 0.001$  $\Delta\rho_{\max} = 0.49 \text{ e } \text{\AA}^{-3}$  $\Delta\rho_{\min} = -0.41 \text{ e } \text{\AA}^{-3}$ Absolute structure: Flack (1983), 1927 Fridel  
pairs

Absolute structure parameter: 0.5 (2)

*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  $F^2 > 2\sigma(F^2)$  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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.0124 (4)	0.0746 (5)	0.2218 (7)	0.0672 (15)
H1	-0.0060	0.0194	0.1495	0.081*
C2	-0.0338 (4)	0.0925 (6)	0.2738 (7)	0.0755 (18)
H2	-0.0856	0.0520	0.2332	0.091*
C3	-0.0043 (4)	0.1701 (6)	0.3865 (8)	0.0776 (17)
H3	-0.0345	0.1785	0.4242	0.093*
C4	0.0694 (4)	0.2339 (5)	0.4407 (7)	0.0746 (16)
H4	0.0884	0.2868	0.5149	0.090*
C5	0.1166 (3)	0.2228 (5)	0.3898 (6)	0.0621 (13)
H5	0.1663	0.2681	0.4274	0.075*
C6	0.0876 (3)	0.1414 (5)	0.2800 (5)	0.0537 (12)
C7	0.1625 (3)	0.0050 (5)	0.2239 (5)	0.0556 (12)
H7	0.1460	-0.0616	0.2530	0.067*
C8	0.2108 (3)	-0.0160 (5)	0.1788 (5)	0.0548 (12)
H8	0.2272	-0.0971	0.1778	0.066*
C9	0.2369 (3)	0.0807 (5)	0.1335 (5)	0.0535 (12)
C10	0.2116 (3)	0.2004 (5)	0.1416 (5)	0.0600 (13)
H10	0.2280	0.2682	0.1140	0.072*
C11	0.1632 (3)	0.2204 (5)	0.1894 (5)	0.0586 (13)
H11	0.1482	0.3010	0.1957	0.070*
C12	0.2873 (3)	0.0638 (5)	0.0829 (5)	0.0569 (12)
H12	0.3022	0.1343	0.0566	0.068*
C13	0.3147 (3)	-0.0474 (5)	0.0709 (5)	0.0560 (12)

H13	0.2995	-0.1165	0.0990	0.067*
C14	0.3642 (3)	-0.0719 (5)	0.0201 (5)	0.0541 (12)
C15	0.3748 (4)	-0.1927 (5)	-0.0088 (6)	0.0614 (13)
H15	0.3505	-0.2577	0.0080	0.074*
C16	0.4201 (4)	-0.2204 (5)	-0.0616 (6)	0.0656 (14)
H16	0.4237	-0.3024	-0.0826	0.079*
C17	0.4603 (3)	-0.1265 (5)	-0.0834 (6)	0.0600 (13)
C18	0.4494 (3)	-0.0039 (5)	-0.0559 (6)	0.0626 (13)
H18	0.4744	0.0610	-0.0715	0.075*
C19	0.4029 (3)	0.0225 (5)	-0.0064 (5)	0.0591 (13)
H19	0.3967	0.1050	0.0100	0.071*
C20	0.5037 (5)	-0.2756 (7)	-0.1855 (8)	0.091 (2)
H20A	0.5358	-0.3311	-0.1083	0.137*
H20B	0.5284	-0.2734	-0.2353	0.137*
H20C	0.4462	-0.3045	-0.2453	0.137*
C21	0.5524 (4)	-0.0558 (7)	-0.1456 (7)	0.0751 (16)
H21A	0.5151	0.0107	-0.2024	0.113*
H21B	0.5792	-0.0895	-0.1855	0.113*
H21C	0.5950	-0.0244	-0.0550	0.113*
N1	0.5049 (3)	-0.1511 (5)	-0.1362 (5)	0.0724 (13)
N2	0.1375 (2)	0.1224 (4)	0.2273 (4)	0.0515 (10)
P1	0.26522 (10)	-0.40739 (11)	0.33958 (15)	0.0597 (4)
F5	0.1834 (3)	-0.4680 (5)	0.2131 (5)	0.1541 (16)
F4	0.3249 (3)	-0.5071 (4)	0.3457 (7)	0.1356 (14)
F3	0.3451 (3)	-0.3462 (6)	0.4685 (5)	0.1541 (16)
F2	0.2065 (3)	-0.3017 (4)	0.3315 (7)	0.1356 (14)
F6	0.2762 (3)	-0.3260 (6)	0.2444 (6)	0.1486 (17)
F1	0.2515 (3)	-0.4916 (5)	0.4289 (6)	0.1486 (17)

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.054 (3)	0.051 (3)	0.085 (4)	0.003 (2)	0.034 (3)	0.011 (3)
C2	0.063 (4)	0.061 (4)	0.097 (5)	0.006 (3)	0.044 (4)	0.024 (3)
C3	0.076 (4)	0.067 (4)	0.104 (5)	0.022 (3)	0.060 (4)	0.023 (4)
C4	0.069 (4)	0.058 (3)	0.090 (4)	0.019 (3)	0.043 (3)	0.002 (3)
C5	0.054 (3)	0.049 (3)	0.068 (3)	0.010 (2)	0.028 (3)	0.003 (2)
C6	0.050 (3)	0.042 (2)	0.060 (3)	0.004 (2)	0.027 (2)	0.009 (2)
C7	0.065 (3)	0.043 (3)	0.051 (3)	-0.007 (2)	0.029 (2)	0.004 (2)
C8	0.063 (3)	0.043 (3)	0.058 (3)	-0.007 (2)	0.035 (2)	0.002 (2)
C9	0.052 (3)	0.051 (3)	0.042 (2)	-0.002 (2)	0.019 (2)	0.0032 (19)
C10	0.059 (3)	0.049 (3)	0.061 (3)	-0.008 (2)	0.029 (3)	0.012 (2)
C11	0.058 (3)	0.043 (3)	0.061 (3)	0.002 (2)	0.027 (3)	0.007 (2)
C12	0.057 (3)	0.059 (3)	0.048 (2)	-0.011 (2)	0.027 (2)	0.006 (2)
C13	0.053 (3)	0.053 (3)	0.050 (3)	-0.006 (2)	0.024 (2)	0.005 (2)
C14	0.046 (3)	0.050 (3)	0.049 (2)	-0.006 (2)	0.018 (2)	0.003 (2)
C15	0.058 (3)	0.044 (3)	0.065 (3)	-0.006 (2)	0.027 (3)	0.001 (2)
C16	0.063 (3)	0.039 (3)	0.074 (3)	0.003 (2)	0.029 (3)	0.001 (2)

C17	0.054 (3)	0.048 (3)	0.056 (3)	0.002 (2)	0.020 (2)	-0.005 (2)
C18	0.062 (3)	0.047 (3)	0.076 (3)	-0.014 (2)	0.039 (3)	-0.009 (2)
C19	0.069 (3)	0.042 (3)	0.070 (3)	-0.008 (2)	0.042 (3)	-0.009 (2)
C20	0.085 (4)	0.081 (4)	0.096 (5)	0.009 (4)	0.046 (4)	-0.030 (4)
C21	0.063 (3)	0.086 (4)	0.082 (4)	0.003 (3)	0.045 (3)	-0.007 (3)
N1	0.067 (3)	0.058 (3)	0.091 (3)	-0.003 (2)	0.046 (3)	-0.019 (2)
N2	0.050 (2)	0.040 (2)	0.052 (2)	-0.0041 (16)	0.0224 (19)	0.0060 (16)
P1	0.0620 (7)	0.0432 (6)	0.0712 (8)	0.0011 (6)	0.0376 (6)	0.0017 (6)
F5	0.116 (3)	0.133 (3)	0.129 (3)	-0.033 (2)	0.025 (2)	-0.040 (2)
F4	0.150 (3)	0.080 (2)	0.227 (4)	0.0386 (19)	0.139 (3)	0.023 (2)
F3	0.116 (3)	0.133 (3)	0.129 (3)	-0.033 (2)	0.025 (2)	-0.040 (2)
F2	0.150 (3)	0.080 (2)	0.227 (4)	0.0386 (19)	0.139 (3)	0.023 (2)
F6	0.159 (3)	0.155 (3)	0.194 (4)	0.054 (3)	0.138 (3)	0.096 (3)
F1	0.159 (3)	0.155 (3)	0.194 (4)	0.054 (3)	0.138 (3)	0.096 (3)

*Geometric parameters (Å, °)*

C1—C2	1.375 (9)	C13—H13	0.9300
C1—C6	1.395 (8)	C14—C15	1.389 (7)
C1—H1	0.9300	C14—C19	1.406 (7)
C2—C3	1.391 (10)	C15—C16	1.384 (8)
C2—H2	0.9300	C15—H15	0.9300
C3—C4	1.363 (9)	C16—C17	1.392 (8)
C3—H3	0.9300	C16—H16	0.9300
C4—C5	1.372 (9)	C17—N1	1.366 (7)
C4—H4	0.9300	C17—C18	1.403 (7)
C5—C6	1.393 (7)	C18—C19	1.371 (7)
C5—H5	0.9300	C18—H18	0.9300
C6—N2	1.449 (7)	C19—H19	0.9300
C7—C8	1.350 (7)	C20—N1	1.456 (8)
C7—N2	1.360 (6)	C20—H20A	0.9600
C7—H7	0.9300	C20—H20B	0.9600
C8—C9	1.397 (7)	C20—H20C	0.9600
C8—H8	0.9300	C21—N1	1.428 (8)
C9—C10	1.401 (7)	C21—H21A	0.9600
C9—C12	1.435 (8)	C21—H21B	0.9600
C10—C11	1.375 (8)	C21—H21C	0.9600
C10—H10	0.9300	P1—F1	1.539 (4)
C11—N2	1.353 (6)	P1—F6	1.547 (4)
C11—H11	0.9300	P1—F4	1.550 (4)
C12—C13	1.348 (8)	P1—F3	1.555 (4)
C12—H12	0.9300	P1—F5	1.557 (4)
C13—C14	1.433 (7)	P1—F2	1.574 (4)
C2—C1—C6	118.4 (6)	C15—C16—C17	120.5 (5)
C2—C1—H1	120.8	C15—C16—H16	119.8
C6—C1—H1	120.8	C17—C16—H16	119.8
C1—C2—C3	121.0 (6)	N1—C17—C16	121.7 (5)

C1—C2—H2	119.5	N1—C17—C18	121.0 (5)
C3—C2—H2	119.5	C16—C17—C18	117.2 (5)
C4—C3—C2	118.9 (6)	C19—C18—C17	121.6 (5)
C4—C3—H3	120.5	C19—C18—H18	119.2
C2—C3—H3	120.5	C17—C18—H18	119.2
C3—C4—C5	122.5 (6)	C18—C19—C14	121.6 (5)
C3—C4—H4	118.8	C18—C19—H19	119.2
C5—C4—H4	118.8	C14—C19—H19	119.2
C4—C5—C6	117.8 (6)	N1—C20—H20A	109.5
C4—C5—H5	121.1	N1—C20—H20B	109.5
C6—C5—H5	121.1	H20A—C20—H20B	109.5
C5—C6—C1	121.4 (5)	N1—C20—H20C	109.5
C5—C6—N2	119.6 (4)	H20A—C20—H20C	109.5
C1—C6—N2	119.0 (5)	H20B—C20—H20C	109.5
C8—C7—N2	120.8 (5)	N1—C21—H21A	109.5
C8—C7—H7	119.6	N1—C21—H21B	109.5
N2—C7—H7	119.6	H21A—C21—H21B	109.5
C7—C8—C9	121.9 (5)	N1—C21—H21C	109.5
C7—C8—H8	119.1	H21A—C21—H21C	109.5
C9—C8—H8	119.1	H21B—C21—H21C	109.5
C8—C9—C10	115.6 (5)	C17—N1—C21	120.9 (5)
C8—C9—C12	124.3 (5)	C17—N1—C20	120.1 (6)
C10—C9—C12	120.1 (5)	C21—N1—C20	119.0 (5)
C11—C10—C9	121.8 (5)	C11—N2—C7	120.2 (5)
C11—C10—H10	119.1	C11—N2—C6	120.6 (4)
C9—C10—H10	119.1	C7—N2—C6	119.1 (4)
N2—C11—C10	119.7 (5)	F1—P1—F6	177.6 (4)
N2—C11—H11	120.2	F1—P1—F4	89.5 (3)
C10—C11—H11	120.2	F6—P1—F4	90.1 (3)
C13—C12—C9	124.5 (5)	F1—P1—F3	92.2 (3)
C13—C12—H12	117.7	F6—P1—F3	90.1 (3)
C9—C12—H12	117.7	F4—P1—F3	87.7 (3)
C12—C13—C14	127.8 (5)	F1—P1—F5	86.3 (3)
C12—C13—H13	116.1	F6—P1—F5	91.4 (3)
C14—C13—H13	116.1	F4—P1—F5	93.9 (3)
C15—C14—C19	116.1 (5)	F3—P1—F5	177.8 (4)
C15—C14—C13	120.8 (5)	F1—P1—F2	92.9 (3)
C19—C14—C13	123.1 (5)	F6—P1—F2	87.5 (3)
C16—C15—C14	122.9 (5)	F4—P1—F2	177.5 (3)
C16—C15—H15	118.6	F3—P1—F2	91.4 (3)
C14—C15—H15	118.6	F5—P1—F2	87.1 (3)
C6—C1—C2—C3	-3.3 (8)	C14—C15—C16—C17	2.4 (8)
C1—C2—C3—C4	3.4 (9)	C15—C16—C17—N1	-179.6 (5)
C2—C3—C4—C5	-1.1 (9)	C15—C16—C17—C18	-2.9 (8)
C3—C4—C5—C6	-1.0 (8)	N1—C17—C18—C19	178.2 (5)
C4—C5—C6—C1	1.0 (7)	C16—C17—C18—C19	1.5 (8)
C4—C5—C6—N2	-177.1 (5)	C17—C18—C19—C14	0.5 (8)

C2—C1—C6—C5	1.1 (8)	C15—C14—C19—C18	-1.0 (7)
C2—C1—C6—N2	179.2 (5)	C13—C14—C19—C18	-179.6 (5)
N2—C7—C8—C9	-0.2 (7)	C16—C17—N1—C21	-173.9 (5)
C7—C8—C9—C10	1.7 (7)	C18—C17—N1—C21	9.5 (8)
C7—C8—C9—C12	-179.2 (5)	C16—C17—N1—C20	6.8 (9)
C8—C9—C10—C11	-0.9 (7)	C18—C17—N1—C20	-169.8 (6)
C12—C9—C10—C11	179.9 (5)	C10—C11—N2—C7	2.9 (7)
C9—C10—C11—N2	-1.4 (8)	C10—C11—N2—C6	179.0 (4)
C8—C9—C12—C13	1.0 (8)	C8—C7—N2—C11	-2.1 (7)
C10—C9—C12—C13	-179.9 (5)	C8—C7—N2—C6	-178.3 (4)
C9—C12—C13—C14	179.2 (5)	C5—C6—N2—C11	-53.1 (6)
C12—C13—C14—C15	-168.5 (5)	C1—C6—N2—C11	128.8 (5)
C12—C13—C14—C19	10.0 (8)	C5—C6—N2—C7	123.1 (5)
C19—C14—C15—C16	-0.4 (8)	C1—C6—N2—C7	-55.0 (6)
C13—C14—C15—C16	178.1 (5)		

*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>
C8—H8...F6	0.93	2.59	3.486 (8)	162
C11—H11...F5 <sup>i</sup>	0.93	2.55	3.363 (8)	146
C16—H16...F4 <sup>ii</sup>	0.93	2.59	3.289 (7)	132
C21—H21 <sup>B</sup> ...F2 <sup>iii</sup>	0.96	2.64	3.516 (8)	152

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