# organic compounds

# 1-[Bicyclo[4.2.0]octa-1(6),2,4-trien-3-yl]-3-[bicyclo[4.2.0]octa-1(6),2,4-trien-3-yl-methyl]imidazolium hexafluorophosphate

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In the title compound, $C_{20}H_{19}N_2^+ \cdot PF_6^-$, the two benzocyclo-butene units are essentially planar and they form dihedral angles of 38.0 (2) and 72.7 (2)°, with the central imidazolium ring. In the crystal structure, weak C—H⋯$\pi$ and $\pi$–$\pi$ stacking interactions [centroid–centroid distance = 3.742 (2) Å] contribute to the stability of the crystal structure. The $PF_6^-$ ion is disordered over two positions with site occupancies of 0.869 (9) and 0.131 (9).

## Related literature

For related literature, see: Farona (1996); Kirchhoff & Bruza (1993); Michellys *et al.* (2001); Nemeto & Fukumoto (1998); Zhang *et al.* (2006).
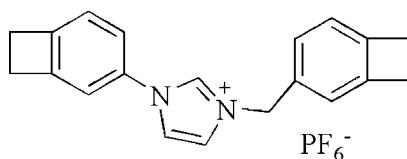


## Experimental

### Crystal data

| | |
|---|---|
| $C_{20}H_{19}N_2^+ \cdot F_6P^-$ | $a = 9.311$ (3) Å |
| $M_r = 432.34$ | $b = 10.138$ (3) Å |
| Triclinic, $P\bar{1}$ | $c = 10.562$ (3) Å |
| $\alpha = 86.82$ (2)° | Mo $K\alpha$ radiation |
| $\beta = 86.44$ (2)° | $\mu = 0.21$ mm$^{-1}$ |
| $\gamma = 73.61$ (2)° | $T = 296$ (2) K |
| $V = 953.9$ (5) Å$^3$ | $0.28 \times 0.25 \times 0.18$ mm |
| $Z = 2$ | |

### Data collection

| | |
|---|---|
| Enraf–Nonius CAD-4 diffractometer | 2238 reflections with $I > 2\sigma(I)$ |
| Absorption correction: none | $R_{int} = 0.006$ |
| 3558 measured reflections | 3 standard reflections |
| 3522 independent reflections | every 300 reflections |
| | intensity decay: 4.6% |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)] = 0.075$ | 81 restraints |
| $wR(F^2) = 0.223$ | H-atom parameters constrained |
| $S = 1.15$ | $\Delta\rho_{max} = 0.64$ e Å$^{-3}$ |
| 3522 reflections | $\Delta\rho_{min} = -0.41$ e Å$^{-3}$ |
| 299 parameters | |

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

$Cg1$ is the centroid of the C3–C8 ring.

| $D$—H⋯$A$ | $D$—H | H⋯$A$ | $D$⋯$A$ | $D$—H⋯$A$ |
|---|---|---|---|---|
| C2—H2B⋯$Cg1^i$ | 0.97 | 3.00 | 3.813 (5) | 143 |

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

Data collection: *DIFRAC* (Gabe & White, 1993); cell refinement: *DIFRAC*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2536).

## References

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# supporting information

# 1-[Bicyclo[4.2.0]octa-1(6),2,4-trien-3-yl]-3-[bicyclo[4.2.0]octa-1(6),2,4-trien-3-ylmethyl]imidazolium hexafluorophosphate

## Fang-Hua Zhu, Jun-Xiao Yang, Zhi-Hua Mao and Ru-Gang Xie

### S1. Comment

Benzocyclobutenes (BCBs) are important building blocks for new polymers and advanced materials (Nemeto & Fukumoto, 1998; Michellys *et al.*, 2001). Most of the reported benzocyclobutene monomers in the literature are bis-BCB-functionalized compounds owing to their highly crosslinked structure and their excellent properties such as the insolubility, low solvent pickup and good thermal stability (Farona, 1996; Zhang *et al.*, 2006; Kirchhoff & Bruza, 1993). We report here the crystal structure of the title compound, a novel bisbenzocyclobutene-terminated imidazolium which was obtained by an anion metathesis between 1-(4-benzocyclobutenyl)-3-(4-benzocyclobutenylmethyl) imidazolium choloride and hexafluorophosphate ammonium.

The two benzocyclobutene units are essentially planar. The plane of the C1—C8 and C13—C20 benzocyclobutene units form dihedral angles of 38.0 (2) and 72.7 (2)°, respectively, with the central imidazolium ring.

A combination of intermolecular $\pi$-$\pi$ and C—H···$\pi$ interactions provide packing forces in the crystal structure of the title compound. A $\pi$-$\pi$ interaction between C13—C15/C18—C20 benzene ring and its symmetry- related counterpart at (-*x*, 1 - *y*, -*z*), with their centroids separated by 3.742 (2) Å, plays an important part in the connection of two adjacent molecules. In addition, a weak C—H···$\pi$ interaction between C2—H2B group and C3—C8 benzene ring at (1 - *x*, -1 - *y*, 1 - *z*) contributes to the crystal packing (Table 1).

### S2. Experimental

4-(*N*-imidazolyl)benzocyclobutene (5 mmol, 850 mg) and 4-cholomethylbenzocyclobutene (5 mmol, 760 mg) were placed in a two-necked round-bottomed flask under a nitrogen atmosphere, and the mixture was heated at 353 K for 3 h. After the reaction was completed, the resulting 1-(4-benzocyclobutenyl)-3-(4-benzocyclobutenylmethyl)imidazolium chloride, [BBMI][Cl$^-$], was obtained in 85% yield (1.381 g). [BBMI][Cl$^-$] was dissolved in deioned water, and NH$_4$PF$_6$ was added to replace Cl$^-$ ions by PF$_6^-$ ions, to obtain the title compound as a white solid (yield 1.331 g). Colourless crystals of the title compound were obtained by recrystallization of the solid from acetone and ethanol (1:1 *v/v*). $^1$H NMR (400 MHz, CDCl$_3$,): $\delta$ 8.84 (s, 1H), 7.41 (t, 1H), 7.31 (m, 2H), 7.27 (2 d, 1H), 7.22 (d, 1H), 7.18 (d, J = 8.0 Hz, 1H), 7.15 (s, 1H), 7.09 (d, J = 7.6 Hz, 1H), 5.37 (s, 2H), 3.21 (s, 4H), 3.17 (s, 4H); $^{13}$C NMR (100 MHz, CDCl$_3$,): $\delta$ 148.58, 148.04, 7.65, 147.21, 133.71, 133.42, 130.78, 128.20, 124.45, 123.53, 122.77, 121.88, 121.46, 117.09, 54.52, 29.47, 29.32 p.p.m.

### S3. Refinement

H atoms were positioned geometrically and refined in the riding model approximation with C—H = 0.93 or 0.97 Å and $U_{iso}$(H) = 1.2$U_{eq}$(C). Four F atoms of the PF$_6^-$ ion are disordered over two positions (F3,F4,F5,F6/F3A,F4A,F5A,F6A) with refined occupancies of 0.869 (9) and 0.131 (9). The disordered F atoms were restrained to be coplanar, with the F···F distances restrained to be equal. The displacement parameters of disordered F atoms were restrained to be approximately

isotropic.
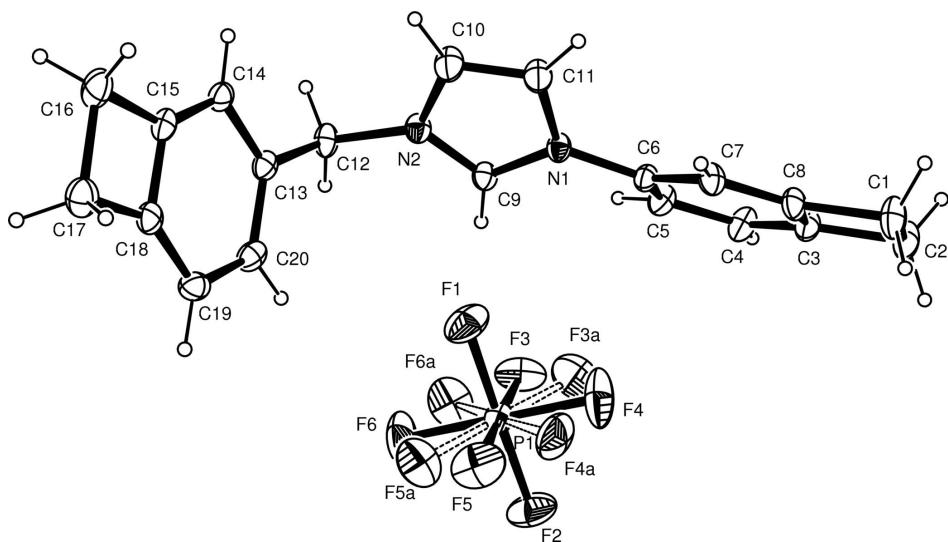


**Figure 1**

The molecular structure of the title compound, showing 30% probability displacement ellipsoids and the atomic numbering.

## 1-[Bicyclo[4.2.0]octa-1(6),2,4-trien-3-yl]-3-[bicyclo[4.2.0]octa- 1(6),2,4-trien-3-ylmethyl]imidazolium hexafluorophosphate

*Crystal data*
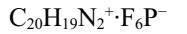
| | |
|---|---|
| $C_{20}H_{19}N_2^+ \cdot F_6P^-$ | $Z = 2$ |
| $M_r = 432.34$ | $F(000) = 444$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.505$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $a = 9.311$ (3) Å | Cell parameters from 20 reflections |
| $b = 10.138$ (3) Å | $\theta = 4.9$–9.1° |
| $c = 10.562$ (3) Å | $\mu = 0.21$ mm$^{-1}$ |
| $\alpha = 86.82$ (2)° | $T = 296$ K |
| $\beta = 86.44$ (2)° | Block, colourless |
| $\gamma = 73.61$ (2)° | $0.28 \times 0.25 \times 0.18$ mm |
| $V = 953.9$ (5) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Enraf–Nonius CAD-4 diffractometer | $R_{int} = 0.006$ |
| | $\theta_{max} = 25.5°$, $\theta_{min} = 1.9°$ |
| Radiation source: fine-focus sealed tube | $h = -10 \rightarrow 11$ |
| Graphite monochromator | $k = -2 \rightarrow 12$ |
| $\omega/2\theta$ scans | $l = -12 \rightarrow 12$ |
| 3558 measured reflections | 3 standard reflections every 300 reflections |
| 3522 independent reflections | intensity decay: 4.6% |
| 2238 reflections with $I > 2\sigma(I)$ | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | |
| $R[F^2 > 2\sigma(F^2)] = 0.075$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.223$ | |
| $S = 1.15$ | H-atom parameters constrained |
| 3522 reflections | $w = 1/[\sigma^2(F_o^2) + (0.1349P)^2]$ |
| 299 parameters | where $P = (F_o^2 + 2F_c^2)/3$ |
| 81 restraints | $(\Delta/\sigma)_{max} = 0.001$ |
| Primary atom site location: structure-invariant direct methods | $\Delta\rho_{max} = 0.64$ e Å$^{-3}$ |
| | $\Delta\rho_{min} = -0.41$ e Å$^{-3}$ |

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

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| N1 | 0.3258 (3) | −0.0608 (3) | 0.3056 (3) | 0.0388 (7) | |
| N2 | 0.1779 (3) | 0.1062 (3) | 0.1997 (3) | 0.0412 (7) | |
| C1 | 0.7078 (6) | −0.4108 (5) | 0.5742 (4) | 0.0654 (13) | |
| H1A | 0.6556 | −0.4310 | 0.6520 | 0.078* | |
| H1B | 0.7911 | −0.3758 | 0.5917 | 0.078* | |
| C2 | 0.7481 (5) | −0.5286 (5) | 0.4784 (4) | 0.0658 (13) | |
| H2A | 0.8522 | −0.5547 | 0.4476 | 0.079* | |
| H2B | 0.7152 | −0.6086 | 0.5066 | 0.079* | |
| C3 | 0.6426 (4) | −0.4283 (4) | 0.3899 (4) | 0.0486 (10) | |
| C4 | 0.5802 (5) | −0.4138 (4) | 0.2742 (4) | 0.0534 (11) | |
| H4 | 0.6062 | −0.4838 | 0.2166 | 0.064* | |
| C5 | 0.4756 (4) | −0.2881 (4) | 0.2472 (3) | 0.0477 (10) | |
| H5 | 0.4297 | −0.2731 | 0.1700 | 0.057* | |
| C6 | 0.4397 (4) | −0.1854 (4) | 0.3348 (3) | 0.0385 (8) | |
| C7 | 0.5044 (4) | −0.2001 (4) | 0.4520 (3) | 0.0452 (9) | |
| H7 | 0.4809 | −0.1304 | 0.5099 | 0.054* | |
| C8 | 0.6057 (5) | −0.3256 (4) | 0.4753 (3) | 0.0474 (9) | |
| C9 | 0.2990 (4) | 0.0017 (3) | 0.1915 (3) | 0.0381 (8) | |
| H9 | 0.3572 | −0.0247 | 0.1175 | 0.046* | |
| C10 | 0.1233 (5) | 0.1122 (4) | 0.3233 (4) | 0.0583 (12) | |
| H10 | 0.0378 | 0.1759 | 0.3552 | 0.070* | |
| C11 | 0.2151 (5) | 0.0095 (4) | 0.3902 (4) | 0.0525 (10) | |
| H11 | 0.2059 | −0.0104 | 0.4768 | 0.063* | |
| C12 | 0.1133 (4) | 0.2028 (4) | 0.0937 (3) | 0.0457 (9) | |
| H12A | 0.0101 | 0.2026 | 0.0854 | 0.055* | |

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| | | | | | |
|---|---|---|---|---|---|
| H12B | 0.1688 | 0.1715 | 0.0150 | 0.055* | |
| C13 | 0.1181 (4) | 0.3462 (4) | 0.1145 (3) | 0.0394 (8) | |
| C14 | −0.0027 (4) | 0.4358 (4) | 0.1797 (3) | 0.0414 (9) | |
| H14 | −0.0882 | 0.4103 | 0.2072 | 0.050* | |
| C15 | 0.0126 (4) | 0.5639 (4) | 0.2006 (3) | 0.0446 (9) | |
| C16 | −0.0632 (5) | 0.7005 (4) | 0.2603 (5) | 0.0611 (12) | |
| H16A | −0.0769 | 0.6945 | 0.3520 | 0.073* | |
| H16B | −0.1550 | 0.7522 | 0.2215 | 0.073* | |
| C17 | 0.0771 (5) | 0.7460 (4) | 0.2122 (4) | 0.0613 (12) | |
| H17A | 0.0569 | 0.8218 | 0.1496 | 0.074* | |
| H17B | 0.1360 | 0.7622 | 0.2793 | 0.074* | |
| C18 | 0.1361 (4) | 0.6058 (4) | 0.1564 (3) | 0.0448 (9) | |
| C19 | 0.2544 (4) | 0.5185 (4) | 0.0891 (4) | 0.0494 (10) | |
| H19 | 0.3375 | 0.5464 | 0.0583 | 0.059* | |
| C20 | 0.2429 (4) | 0.3886 (4) | 0.0702 (3) | 0.0465 (10) | |
| H20 | 0.3209 | 0.3271 | 0.0264 | 0.056* | |
| P1 | 0.67915 (11) | 0.11989 (10) | 0.17994 (9) | 0.0466 (4) | |
| F1 | 0.5204 (3) | 0.1791 (4) | 0.2485 (3) | 0.0890 (10) | |
| F2 | 0.8367 (3) | 0.0567 (3) | 0.1101 (3) | 0.0964 (11) | |
| F3 | 0.6267 (4) | 0.0006 (4) | 0.1205 (3) | 0.0908 (17) | 0.869 (9) |
| F4 | 0.7321 (5) | 0.0220 (4) | 0.2990 (3) | 0.109 (2) | 0.869 (9) |
| F5 | 0.7340 (4) | 0.2334 (4) | 0.2388 (5) | 0.112 (2) | 0.869 (9) |
| F6 | 0.6275 (5) | 0.2124 (5) | 0.0586 (4) | 0.120 (2) | 0.869 (9) |
| F3A | 0.6793 (7) | −0.018 (2) | 0.2185 (12) | 0.127 (14) | 0.131 (9) |
| F4A | 0.7527 (10) | 0.1314 (12) | 0.3022 (15) | 0.090 (10) | 0.131 (9) |
| F5A | 0.6805 (7) | 0.260 (2) | 0.1369 (12) | 0.103 (11) | 0.131 (9) |
| F6A | 0.6104 (10) | 0.1040 (12) | 0.0613 (16) | 0.093 (10) | 0.131 (9) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0443 (17) | 0.0364 (15) | 0.0320 (15) | −0.0048 (13) | −0.0036 (12) | −0.0025 (12) |
| N2 | 0.0480 (18) | 0.0395 (16) | 0.0332 (15) | −0.0068 (14) | −0.0038 (13) | −0.0028 (12) |
| C1 | 0.075 (3) | 0.066 (3) | 0.047 (2) | −0.005 (2) | −0.023 (2) | 0.011 (2) |
| C2 | 0.064 (3) | 0.065 (3) | 0.056 (3) | 0.002 (2) | −0.013 (2) | 0.012 (2) |
| C3 | 0.046 (2) | 0.051 (2) | 0.042 (2) | −0.0017 (18) | −0.0069 (16) | 0.0010 (17) |
| C4 | 0.050 (2) | 0.055 (2) | 0.046 (2) | 0.0046 (18) | −0.0117 (17) | −0.0145 (18) |
| C5 | 0.049 (2) | 0.055 (2) | 0.0348 (19) | −0.0043 (18) | −0.0110 (16) | −0.0087 (17) |
| C6 | 0.0361 (18) | 0.0414 (19) | 0.0340 (17) | −0.0041 (15) | −0.0050 (14) | 0.0004 (14) |
| C7 | 0.057 (2) | 0.048 (2) | 0.0287 (17) | −0.0099 (18) | −0.0066 (16) | −0.0024 (15) |
| C8 | 0.055 (2) | 0.050 (2) | 0.0331 (18) | −0.0094 (18) | −0.0107 (16) | 0.0070 (16) |
| C9 | 0.044 (2) | 0.0407 (19) | 0.0286 (16) | −0.0088 (16) | −0.0053 (14) | −0.0017 (14) |
| C10 | 0.068 (3) | 0.049 (2) | 0.042 (2) | 0.008 (2) | 0.0080 (19) | −0.0024 (18) |
| C11 | 0.061 (3) | 0.050 (2) | 0.0356 (19) | 0.0015 (19) | 0.0081 (18) | −0.0034 (16) |
| C12 | 0.052 (2) | 0.046 (2) | 0.0351 (18) | −0.0040 (17) | −0.0135 (16) | −0.0021 (16) |
| C13 | 0.0406 (19) | 0.0434 (19) | 0.0305 (17) | −0.0041 (15) | −0.0117 (14) | 0.0019 (14) |
| C14 | 0.0363 (19) | 0.043 (2) | 0.0417 (19) | −0.0050 (15) | −0.0094 (15) | 0.0040 (15) |
| C15 | 0.041 (2) | 0.043 (2) | 0.044 (2) | −0.0004 (16) | −0.0123 (16) | 0.0019 (16) |

| C16 | 0.058 (3) | 0.043 (2) | 0.075 (3) | −0.0011 (19) | −0.008 (2) | −0.008 (2) |
|-----|-----------|-----------|-----------|--------------|------------|------------|
| C17 | 0.063 (3) | 0.050 (2) | 0.071 (3) | −0.015 (2) | −0.014 (2) | −0.002 (2) |
| C18 | 0.045 (2) | 0.047 (2) | 0.0414 (19) | −0.0100 (17) | −0.0131 (16) | 0.0077 (16) |
| C19 | 0.042 (2) | 0.064 (3) | 0.044 (2) | −0.0179 (19) | −0.0051 (17) | 0.0037 (18) |
| C20 | 0.040 (2) | 0.056 (2) | 0.0373 (19) | −0.0024 (17) | −0.0069 (16) | −0.0042 (17) |
| P1 | 0.0469 (6) | 0.0481 (6) | 0.0409 (6) | −0.0061 (5) | −0.0024 (4) | −0.0045 (4) |
| F1 | 0.0595 (17) | 0.125 (2) | 0.0650 (17) | 0.0038 (16) | 0.0102 (13) | −0.0246 (17) |
| F2 | 0.0592 (18) | 0.098 (2) | 0.127 (3) | −0.0126 (16) | 0.0273 (18) | −0.042 (2) |
| F3 | 0.093 (3) | 0.122 (4) | 0.078 (3) | −0.060 (3) | 0.027 (2) | −0.048 (3) |
| F4 | 0.165 (4) | 0.074 (3) | 0.054 (2) | 0.023 (3) | −0.021 (2) | 0.0058 (18) |
| F5 | 0.105 (3) | 0.082 (3) | 0.158 (5) | −0.031 (2) | 0.003 (3) | −0.061 (3) |
| F6 | 0.151 (4) | 0.106 (4) | 0.060 (2) | 0.024 (3) | 0.002 (2) | 0.034 (2) |
| F3A | 0.122 (19) | 0.094 (16) | 0.18 (2) | −0.059 (14) | −0.017 (16) | 0.033 (16) |
| F4A | 0.077 (13) | 0.105 (19) | 0.079 (13) | −0.006 (12) | −0.049 (11) | 0.013 (12) |
| F5A | 0.140 (19) | 0.086 (15) | 0.091 (17) | −0.050 (13) | 0.001 (14) | 0.004 (13) |
| F6A | 0.145 (18) | 0.095 (17) | 0.052 (12) | −0.048 (14) | −0.024 (11) | −0.012 (12) |

## Geometric parameters (Å, º)

| | | | |
|-----|-----------|-----------|-----------|
| N1—C9 | 1.334 (4) | C12—H12B | 0.97 |
| N1—C11 | 1.382 (4) | C13—C20 | 1.395 (5) |
| N1—C6 | 1.435 (4) | C13—C14 | 1.400 (5) |
| N2—C9 | 1.313 (4) | C14—C15 | 1.378 (5) |
| N2—C10 | 1.370 (5) | C14—H14 | 0.93 |
| N2—C12 | 1.484 (4) | C15—C18 | 1.381 (6) |
| C1—C8 | 1.517 (5) | C15—C16 | 1.518 (5) |
| C1—C2 | 1.558 (6) | C16—C17 | 1.552 (7) |
| C1—H1A | 0.97 | C16—H16A | 0.97 |
| C1—H1B | 0.97 | C16—H16B | 0.97 |
| C2—C3 | 1.522 (5) | C17—C18 | 1.510 (5) |
| C2—H2A | 0.97 | C17—H17A | 0.97 |
| C2—H2B | 0.97 | C17—H17B | 0.97 |
| C3—C4 | 1.368 (5) | C18—C19 | 1.387 (5) |
| C3—C8 | 1.371 (5) | C19—C20 | 1.378 (5) |
| C4—C5 | 1.395 (5) | C19—H19 | 0.93 |
| C4—H4 | 0.93 | C20—H20 | 0.93 |
| C5—C6 | 1.386 (5) | P1—F3A | 1.43 (3) |
| C5—H5 | 0.93 | P1—F5A | 1.47 (2) |
| C6—C7 | 1.393 (5) | P1—F6A | 1.48 (2) |
| C7—C8 | 1.373 (5) | P1—F4A | 1.524 (18) |
| C7—H7 | 0.93 | P1—F5 | 1.556 (4) |
| C9—H9 | 0.93 | P1—F6 | 1.563 (4) |
| C10—C11 | 1.343 (5) | P1—F4 | 1.572 (4) |
| C10—H10 | 0.93 | P1—F1 | 1.576 (3) |
| C11—H11 | 0.93 | P1—F2 | 1.579 (3) |
| C12—C13 | 1.497 (5) | P1—F3 | 1.597 (4) |
| C12—H12A | 0.97 | | |

| | | | |
|---|---|---|---|
| C9—N1—C11 | 107.5 (3) | C15—C14—H14 | 122.0 |
| C9—N1—C6 | 126.8 (3) | C13—C14—H14 | 122.0 |
| C11—N1—C6 | 125.4 (3) | C14—C15—C18 | 123.0 (3) |
| C9—N2—C10 | 108.2 (3) | C14—C15—C16 | 144.1 (4) |
| C9—N2—C12 | 125.8 (3) | C18—C15—C16 | 93.0 (3) |
| C10—N2—C12 | 125.9 (3) | C15—C16—C17 | 86.7 (3) |
| C8—C1—C2 | 86.7 (3) | C15—C16—H16A | 114.2 |
| C8—C1—H1A | 114.2 | C17—C16—H16A | 114.2 |
| C2—C1—H1A | 114.2 | C15—C16—H16B | 114.2 |
| C8—C1—H1B | 114.2 | C17—C16—H16B | 114.2 |
| C2—C1—H1B | 114.2 | H16A—C16—H16B | 111.4 |
| H1A—C1—H1B | 111.4 | C18—C17—C16 | 86.8 (3) |
| C3—C2—C1 | 86.3 (3) | C18—C17—H17A | 114.2 |
| C3—C2—H2A | 114.3 | C16—C17—H17A | 114.2 |
| C1—C2—H2A | 114.3 | C18—C17—H17B | 114.2 |
| C3—C2—H2B | 114.3 | C16—C17—H17B | 114.2 |
| C1—C2—H2B | 114.3 | H17A—C17—H17B | 111.3 |
| H2A—C2—H2B | 111.4 | C15—C18—C19 | 121.1 (4) |
| C4—C3—C8 | 122.4 (3) | C15—C18—C17 | 93.5 (3) |
| C4—C3—C2 | 144.0 (4) | C19—C18—C17 | 145.3 (4) |
| C8—C3—C2 | 93.5 (3) | C20—C19—C18 | 116.7 (4) |
| C3—C4—C5 | 116.3 (3) | C20—C19—H19 | 121.6 |
| C3—C4—H4 | 121.8 | C18—C19—H19 | 121.6 |
| C5—C4—H4 | 121.8 | C19—C20—C13 | 122.2 (3) |
| C6—C5—C4 | 120.5 (3) | C19—C20—H20 | 118.9 |
| C6—C5—H5 | 119.8 | C13—C20—H20 | 118.9 |
| C4—C5—H5 | 119.8 | F3A—P1—F5A | 178.4 (5) |
| C5—C6—C7 | 123.0 (3) | F3A—P1—F6A | 88.8 (6) |
| C5—C6—N1 | 118.5 (3) | F5A—P1—F6A | 90.0 (6) |
| C7—C6—N1 | 118.5 (3) | F3A—P1—F4A | 89.6 (6) |
| C8—C7—C6 | 114.7 (3) | F5A—P1—F4A | 91.5 (6) |
| C8—C7—H7 | 122.7 | F6A—P1—F4A | 178.2 (6) |
| C6—C7—H7 | 122.7 | F5—P1—F6 | 92.2 (3) |
| C3—C8—C7 | 123.1 (3) | F5—P1—F4 | 89.7 (3) |
| C3—C8—C1 | 93.5 (3) | F6—P1—F4 | 177.9 (2) |
| C7—C8—C1 | 143.4 (3) | F3A—P1—F1 | 91.1 (2) |
| N2—C9—N1 | 109.6 (3) | F5A—P1—F1 | 90.1 (2) |
| N2—C9—H9 | 125.2 | F6A—P1—F1 | 91.5 (2) |
| N1—C9—H9 | 125.2 | F4A—P1—F1 | 89.5 (2) |
| C11—C10—N2 | 107.8 (3) | F5—P1—F1 | 89.43 (19) |
| C11—C10—H10 | 126.1 | F6—P1—F1 | 90.82 (19) |
| N2—C10—H10 | 126.1 | F4—P1—F1 | 90.17 (19) |
| C10—C11—N1 | 106.8 (3) | F3A—P1—F2 | 87.5 (2) |
| C10—C11—H11 | 126.6 | F5A—P1—F2 | 91.3 (2) |
| N1—C11—H11 | 126.6 | F6A—P1—F2 | 87.4 (2) |
| N2—C12—C13 | 111.8 (3) | F4A—P1—F2 | 91.5 (2) |
| N2—C12—H12A | 109.3 | F5—P1—F2 | 92.30 (19) |
| C13—C12—H12A | 109.3 | F6—P1—F2 | 89.4 (2) |

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|---|---|---|---|
| N2—C12—H12B | 109.3 | F4—P1—F2 | 89.5 (2) |
| C13—C12—H12B | 109.3 | F1—P1—F2 | 178.25 (18) |
| H12A—C12—H12B | 107.9 | F5—P1—F3 | 178.5 (2) |
| C20—C13—C14 | 120.9 (3) | F6—P1—F3 | 88.8 (3) |
| C20—C13—C12 | 119.8 (3) | F4—P1—F3 | 89.3 (2) |
| C14—C13—C12 | 119.3 (3) | F1—P1—F3 | 91.61 (18) |
| C15—C14—C13 | 116.0 (4) | F2—P1—F3 | 86.66 (17) |
| | | | |
| C8—C1—C2—C3 | 0.8 (3) | C12—N2—C10—C11 | −178.3 (4) |
| C1—C2—C3—C4 | −177.7 (7) | N2—C10—C11—N1 | −0.8 (5) |
| C1—C2—C3—C8 | −0.9 (4) | C9—N1—C11—C10 | 0.7 (5) |
| C8—C3—C4—C5 | −0.3 (7) | C6—N1—C11—C10 | −174.1 (4) |
| C2—C3—C4—C5 | 176.0 (6) | C9—N2—C12—C13 | −115.5 (4) |
| C3—C4—C5—C6 | 0.3 (7) | C10—N2—C12—C13 | 63.2 (5) |
| C4—C5—C6—C7 | 0.4 (7) | N2—C12—C13—C20 | 89.8 (4) |
| C4—C5—C6—N1 | −176.8 (4) | N2—C12—C13—C14 | −89.5 (4) |
| C9—N1—C6—C5 | −35.6 (6) | C20—C13—C14—C15 | −2.4 (5) |
| C11—N1—C6—C5 | 138.2 (4) | C12—C13—C14—C15 | 177.0 (3) |
| C9—N1—C6—C7 | 147.2 (4) | C13—C14—C15—C18 | 2.5 (5) |
| C11—N1—C6—C7 | −39.0 (5) | C13—C14—C15—C16 | −178.8 (5) |
| C5—C6—C7—C8 | −0.9 (6) | C14—C15—C16—C17 | −179.7 (5) |
| N1—C6—C7—C8 | 176.2 (3) | C18—C15—C16—C17 | −0.8 (3) |
| C4—C3—C8—C7 | −0.3 (7) | C15—C16—C17—C18 | 0.8 (3) |
| C2—C3—C8—C7 | −178.1 (4) | C14—C15—C18—C19 | −1.0 (6) |
| C4—C3—C8—C1 | 178.7 (4) | C16—C15—C18—C19 | 179.7 (3) |
| C2—C3—C8—C1 | 0.9 (4) | C14—C15—C18—C17 | −179.9 (3) |
| C6—C7—C8—C3 | 0.9 (6) | C16—C15—C18—C17 | 0.9 (3) |
| C6—C7—C8—C1 | −177.5 (6) | C16—C17—C18—C15 | −0.9 (3) |
| C2—C1—C8—C3 | −0.9 (4) | C16—C17—C18—C19 | −179.2 (6) |
| C2—C1—C8—C7 | 177.7 (6) | C15—C18—C19—C20 | −0.7 (5) |
| C10—N2—C9—N1 | −0.2 (4) | C17—C18—C19—C20 | 177.3 (5) |
| C12—N2—C9—N1 | 178.7 (3) | C18—C19—C20—C13 | 0.8 (5) |
| C11—N1—C9—N2 | −0.3 (4) | C14—C13—C20—C19 | 0.8 (5) |
| C6—N1—C9—N2 | 174.4 (3) | C12—C13—C20—C19 | −178.6 (3) |
| C9—N2—C10—C11 | 0.6 (5) | | |

*Hydrogen-bond geometry (Å, º)*

| $D$—H⋯$A$ | $D$—H | H⋯$A$ | $D$⋯$A$ | $D$—H⋯$A$ |
|---|---|---|---|---|
| C2—H2$B$⋯$Cg$1[i] | 0.97 | 3.00 | 3.813 (5) | 143 |

Symmetry code: (i) −x+1, −y−1, −z+1.