# Synthesis and crystal structure of (1,10-phenanthroline-$\kappa^2N,N'$)[2-(1$H$-pyrazol-1-yl)phenyl-$\kappa^2N^2,C^1$]iridium(III) hexafluoridophosphate with an unknown number of solvent molecules
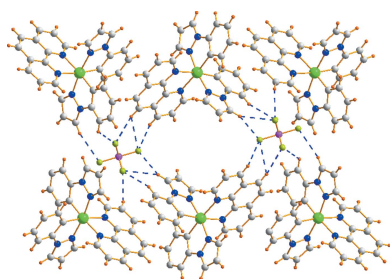
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The cationic complex in the title compound, [Ir(C$_9$H$_7$N$_2$)$_2$(C$_{12}$H$_8$N$_2$)]PF$_6$, comprises two phenylpyrazole (ppz) cyclometallating ligands and one 1,10-phenanthroline (phen) ancillary ligand. The asymmetric unit consists of one [Ir(ppz)$_2$(phen)]$^+$ cation and one [PF$_6$]$^-$ counter-ion. The central Ir$^{III}$ ion is six-coordinated by two N atoms and two C atoms from the two ppz ligands as well as by two N atoms from the phen ligand within a distorted octahedral C$_2$N$_4$ coordination set. In the crystal structure, the [Ir(ppz)$_2$(phen)]$^+$ cations and PF$_6^-$ counter-ions are connected with each other through weak intermolecular C—H$\cdots$F hydrogen bonds. Additional C—H$\cdots\pi$ interactions between the rings of neighbouring cations consolidate the three-dimensional network. Electron density associated with additional disordered solvent molecules inside cavities of the structure was removed with the SQUEEZE procedure in *PLATON* [Spek (2015). *Acta Cryst.* C**71**, 9–18]. The given chemical formula and other crystal data do not take into account the unknown solvent molecule(s). The title compound has a different space-group symmetry (*C*2/*c*) from its solvatomorph (*P*2$_1$/*c*) comprising 1.5CH$_2$Cl$_2$ solvent molecules per ion pair.
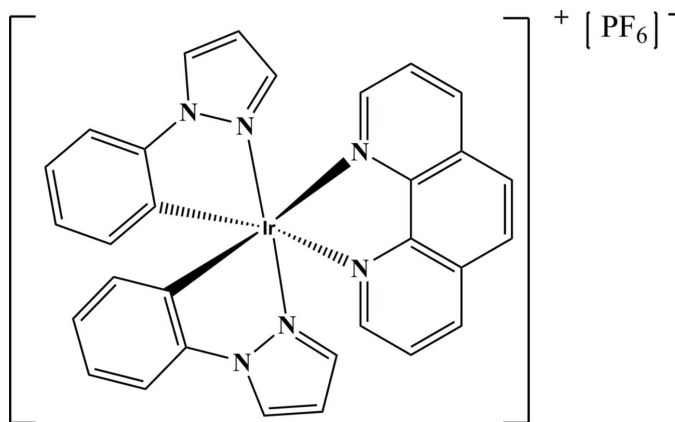
## 1. Chemical context

Cyclometallated iridium(III) complexes have found applications in electroluminescent instruments such as sensors and light-emitting devices and in photocatalysis because of their high emission efficiencies, photo/thermal stabilities and easy tunability of the emission wavelength (Zhao *et al.*, 2010; Shan *et al.*, 2012). In this regard, a variety of cyclometallated iridium complexes have been reported and most of them have potential for the aforementioned applications (Flamigni *et al.*, 2008; Li *et al.*, 2011). The properties of iridium complexes can be tuned by rational design of either the cyclometallating or ancillary ligands (Chen *et al.*, 2010; Goswami *et al.*, 2014; Radwan *et al.*, 2015; Congrave *et al.*, 2017). Among numerous organic conjugate ligands, the cyclometallating ligand 1-phenylpyrazole (ppz) is known for its high triplet energy (Schlegel & Skancke, 1993). Consequently, some bis-cyclometallated Ir$^{III}$ complexes with ppz ligands have been synthesized that exhibit high energy phosphorescence (Sajoto *et al.*, 2005).

On the other hand, ancillary ligands with strong conjugated system such as 1,10-phenanthroline (phen) can also enhance the degree of delocalized $\pi$-electrons of cyclometallated

iridium(III) complex systems through the interaction between the *d* orbitals of the transition metal and the $\pi$-electron orbitals of the organic conjugated system (Liu *et al.*, 2018). This way, the high degree of delocalized $\pi$-electrons can increase the luminescent properties of $Ir^{III}$ complexes (Choy *et al.*, 2014). In this context, we report herein the synthesis and crystal structure of the cyclometallated iridium(III) complex, [Ir(ppz)$_2$(phen)][PF$_6$], which contains an unknown number of solvent molecules.



## 2. Structural commentary

The asymmetric unit of the title complex consists of one [Ir(ppz)$_2$(phen)]$^+$ cation and one PF$_6^-$ counter-ion (Fig. 1). The iridium(III) atom is six-coordinated by four nitrogen atoms and two carbon atoms within an octahedral [N$_4$C$_2$] coordination set. The axial positions are occupied by two nitrogen atoms (N3, N5) from two ppz ligands, while the equatorial plane is composed of two N atoms from the phen ligand (N1, N2) and two C atoms from two ppz ligands (C21, C30).

The bond lengths and angles related to the coordinating carbon and nitrogen atoms are normal and correspond to literature values. The average Ir—C bond length is 2.018 (5) Å, a typical value for the distance between an $Ir^{III}$
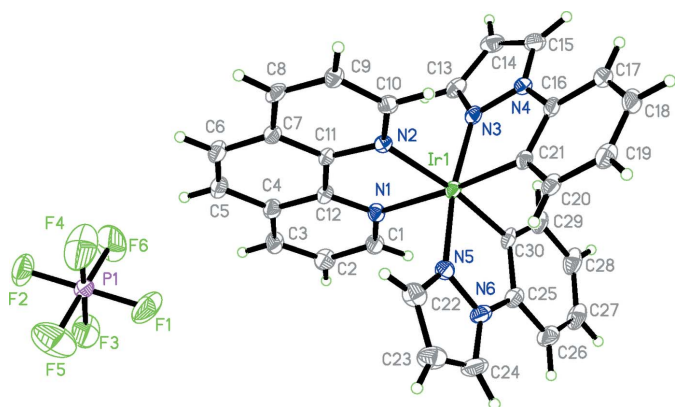
### Table 1
Hydrogen-bond geometry (Å, °).

*Cg*4 and *Cg*9 are the centroids of rings N3/N4/C15–C13 and C16–C21, respectively.

| $D$—H$\cdots A$ | $D$—H | H$\cdots A$ | $D\cdots A$ | $D$—H$\cdots A$ |
|---|---|---|---|---|
| C2—H2A$\cdots$F6$^i$ | 0.93 | 2.57 | 3.184 (7) | 124 |
| C9—H9A$\cdots$F3$^{ii}$ | 0.93 | 2.49 | 3.024 (7) | 117 |
| C17—H17A$\cdots$F4$^{iii}$ | 0.93 | 2.36 | 2.977 (8) | 124 |
| C23—H23A$\cdots$F2$^{iv}$ | 0.93 | 2.53 | 3.383 (8) | 152 |
| C24—H24A$\cdots$F1$^v$ | 0.93 | 2.48 | 3.368 (8) | 161 |
| C26—H26A$\cdots$F5$^v$ | 0.93 | 2.47 | 3.348 (9) | 158 |
| C6—H6A$\cdots$Cg9$^{vi}$ | 0.93 | 2.58 | 3.501 (6) | 173 |
| C29—H29A$\cdots$Cg4 | 0.93 | 2.98 | 3.688 (7) | 134 |

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

and a C atom originating from a ppz ligand (Adamovich *et al.*, 2019). There are two different Ir—N bond types in the cation of the title compound: the average Ir—N$_{C^\wedge N}$ (C^N refers to the ppz ligand) bond length is 2.023 (2) Å, whereas the value for the Ir—N$_{N^\wedge N}$ (N^N refers to the phen ligand) bond is much longer at 2.141 (8) Å. The bond angles around the $Ir^{III}$ atom involving *cis*-arranged ligand atoms deviate clearly from 90° and range from 78.06 (15)° (the bite angle of the phen ligand) to 99.24 (17)°, except for C21—Ir1—C30 with a value of 89.44 (19)°, which correspond to a relatively low distortion from an ideal octahedral coordination polyhedron. The bond angles along the axes of the pseudo-octahedral coordination figure are 171.64 (16), 173.09 (18) and 173.97 (17)° for N3—Ir—N5, C30—Ir—N2 and C21—Ir—N1, respectively.

## 3. Supramolecular features

In the crystal structure, the complex cations are linked to the PF$_6^-$ counter-ions by six C—H$\cdots$F interactions (Table 1, Fig. 2), leading to the formation of a three-dimensional supramolecular network. In addition, there are also C—H$\cdots\pi$ interactions between the [Ir(ppz)$_2$(phen)]$^+$ cations, involving



**Figure 1**
The structures of the molecular entities in the title compound, with displacement ellipsoids drawn at the 30% probability level. H atoms are represented by spheres of arbitrary radius.

**Figure 2**
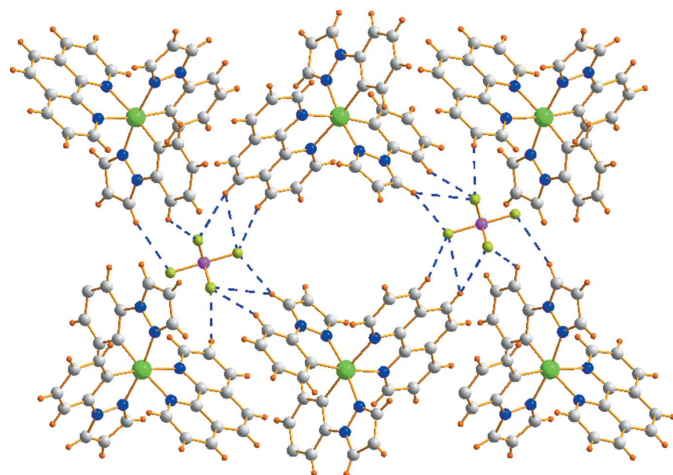C—H$\cdots$F hydrogen bonding interactions between complex cations and counter-ions (shown as dashed lines).

**Figure 3**
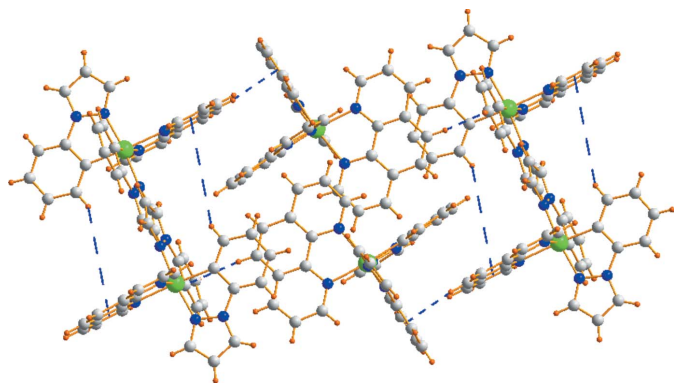C—H···$\pi$ interactions in the title structure (shown as dashed lines).

the centroids of one of the pyrazole rings and of a phenyl ring (Table 1, Fig. 3). As can be seen in Fig. 4, the packing of the components leads to voids that are large enough to host solvent molecules of an unknown nature.

## 4. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.39, update November 2017; Groom *et al.*, 2016) for complexes containing the iridium(III) ion with ppz ligand fragments yielded 36 hits. Among the 36 structures, only four contain auxiliary phen ligands or derivatives thereof. From these, one compound (JUPTIZ; Howarth *et al.*, 2015) matches the title compound, but crystallizes in the space group $P2_1/c$ and contains two molecular ion pairs in the asymmetric unit in
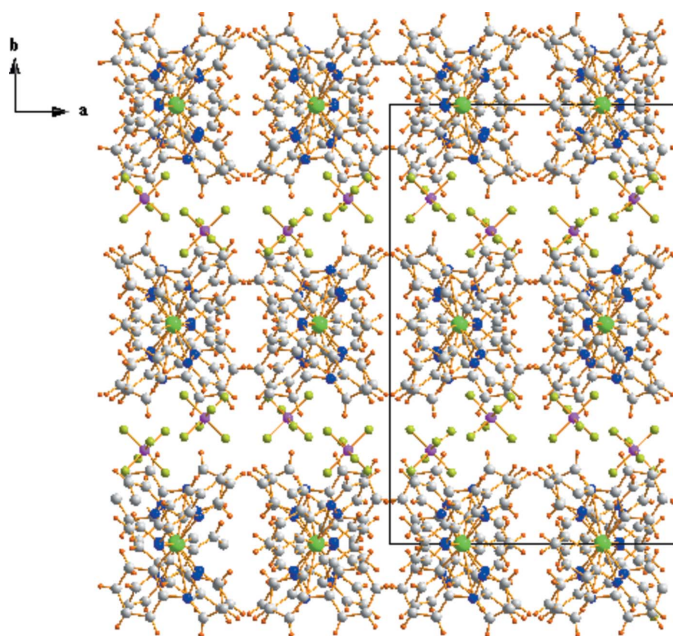


**Figure 4**
A packing diagram of the title compound viewed along the *c* axis, showing the porous structure with different cavities for the unknown solvent molecules.

contrast to the title compound, which crystallizes in space group $C2/c$ with one ion pair in the asymmetric unit. Bond lengths and angles in the corresponding $[Ir(ppz)_2(phen)]^+$ cations are very similar. In both structure refinements, the contributions of solvent molecules were not considered; for JUPTIZ, 1.5 $CH_2Cl_2$ solvent molecules were estimated per ion pair, but in the title structure the number and nature of solvent molecule(s) remains unknown. Hence JUPTIZ is a solvatomorph of the title compound. The three other structures comprise derivatives of the phen ligand, *viz.* JUPTEV/ JUPTAR (Howarth *et al.*, 2015) and DUCWOZ (Shan *et al.*, 2012).

## 5. Synthesis and crystallization

The organometallated iridium(III) dimer, $[Ir(\mu\text{-}Cl)(ppz)_2]_2$ (ppz = 1-phenylpyrazole), was prepared according to a literature protocol (Kwon *et al.*, 2005) by heating $IrCl_3 \cdot 3H_2O$ (1 equiv.) and 1-phenylpyrazole (2.3 equiv.) in a mixed solution of 2-ethoxyethanol and water (*v*/*v* = 3:1) at 408 K.

The title compound was synthesized from the reaction of $[Ir(\mu\text{-}Cl)(ppz)_2]_2$ and 1,10-phenanthroline in a mixed solution of dichloromethane ($CH_2Cl_2$) and methanol (MeOH) (*v*/*v* = 2:1) at 358 K with $KPF_6$ as a source for the $PF_6^-$ counter-ion. The mixture was dried under vacuum and separated by column chromatography on silica gel with $CH_2Cl_2$/petroleum ether (*v*/*v* = 4:1) as eluent. A pure product of the cyclometalated iridium(III) complex was obtained as a dark-yellow solid. Elemental analysis for $C_{30}H_{22}F_6IrN_6P$ (calculated; found): C (44.83; 45.26); H (2.76, 2.73); N (10.46, 10.39)%.

Single crystals of the title compound were grown by inter-diffusion reaction between *n*-hexane and a dichloromethane solution of the pure solid with $CH_2Cl_2$/hexane (*v*/*v* = 1/1) as buffer solution at room temperature for 7 d (Nie *et al.*, 2019). In should be noted that the dichloromethane sesquisolvate of $[Ir(ppz)_2(phen)](PF_6)$ (JUPTIZ) was obtained by reacting $[Ir(\mu\text{-}Cl)(ppz)_2]_2$ with 1,10-phenanthroline under microwave irradiation for 30 min. at 373 K (Howarth *et al.*, 2015).

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Carbon-bound H atoms were placed in calculated positions (C—H = 0.93 Å) and were included in the refinement in the riding-model approximation, with $U_{iso}(H)$ set to $1.2U_{eq}(C)$. The contribution of the missing solvent molecules to the diffraction pattern was subtracted from the reflection data by the SQUEEZE method (Spek, 2015) as implemented in *PLATON* (Spek, 2020). The solvent-accessible volume in the structure of the title compound as calculated by *PLATON* is 1136.1 Å$^3$ (17.7%).

**Table 2**
Experimental details.

| | |
|---|---|
| **Crystal data** | |
| Chemical formula | $[Ir(C_9H_7N_2)_2(C_{12}H_8N_2)]PF_6$ |
| $M_r$ | 803.70 |
| Crystal system, space group | Monoclinic, $C2/c$ |
| Temperature (K) | 293 |
| $a, b, c$ (Å) | 14.976 (3), 22.818 (5), 18.850 (4) |
| $\beta$ (°) | 95.98 (3) |
| $V$ (Å$^3$) | 6406 (2) |
| $Z$ | 8 |
| Radiation type | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 4.28 |
| Crystal size (mm) | $0.25 \times 0.22 \times 0.20$ |
| | |
| **Data collection** | |
| Diffractometer | Bruker APEXII CCD |
| Absorption correction | Multi-scan (*SADABS*; Krause *et al.*, 2015) |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 29484, 6280, 5077 |
| $R_{int}$ | 0.039 |
| $(\sin \theta/\lambda)_{max}$ (Å$^{-1}$) | 0.618 |
| | |
| **Refinement** | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.039, 0.094, 1.11 |
| No. of reflections | 6280 |
| No. of parameters | 397 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 1.25, −0.96 |

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*), *PLATON* (Spek, 2020) and *DIAMOND* (Brandenburg & Putz, 2016), *publCIF* (Westrip, 2010).

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# Synthesis and crystal structure of (1,10-phenanthroline-$\kappa^2N$,$N′$)[2-(1H-pyrazol-1-yl)phenyl-$\kappa^2N^2$,$C^1$]iridium(III) hexafluoridophosphate with an unknown number of solvent molecules

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**Computing details**

Data collection: *APEX3* (Bruker, 2016); cell refinement: *SAINT* (Bruker, 2016); data reduction: *SAINT* (Bruker, 2016); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015b); molecular graphics: *PLATON* (Spek, 2020) and *DIAMOND* (Brandenburg & Putz, 2016); software used to prepare material for publication: *publCIF* (Westrip, 2010).

**(1,10-Phenanthroline-$\kappa^2N$,$N′$)[2-(1H-pyrazol-1-yl)phenyl-$\kappa^2N^2$,$C^1$]iridium(III) hexafluoridophosphate**

*Crystal data*

| | |
|---|---|
| [Ir(C$_9$H$_7$N$_2$)$_2$(C$_{12}$H$_8$N$_2$)]PF$_6$ | $F(000) = 3120$ |
| $M_r = 803.70$ | $D_x = 1.667$ Mg m$^{-3}$ |
| Monoclinic, $C2/c$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $a = 14.976$ (3) Å | Cell parameters from 10866 reflections |
| $b = 22.818$ (5) Å | $\theta = 3.0–26.0°$ |
| $c = 18.850$ (4) Å | $\mu = 4.28$ mm$^{-1}$ |
| $\beta = 95.98$ (3)° | $T = 293$ K |
| $V = 6406$ (2) Å$^3$ | Block, red |
| $Z = 8$ | $0.25 \times 0.22 \times 0.20$ mm |

*Data collection*

| | |
|---|---|
| Bruker APEXII CCD diffractometer | 6280 independent reflections |
| | 5077 reflections with $I > 2\sigma(I)$ |
| phi and $\omega$ scans | $R_{int} = 0.039$ |
| Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015) | $\theta_{max} = 26.0°$, $\theta_{min} = 3.0°$ |
| | $h = -15 \rightarrow 18$ |
| | $k = -28 \rightarrow 25$ |
| 29484 measured reflections | $l = -23 \rightarrow 23$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | |
| $R[F^2 > 2\sigma(F^2)] = 0.039$ | H-atom parameters constrained |
| $wR(F^2) = 0.094$ | $w = 1/[\sigma^2(F_o^2) + (0.0321P)^2 + 39.6215P]$ |
| $S = 1.11$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 6280 reflections | $(\Delta/\sigma)_{max} < 0.001$ |
| 397 parameters | $\Delta\rho_{max} = 1.25$ e Å$^{-3}$ |
| 0 restraints | $\Delta\rho_{min} = -0.96$ e Å$^{-3}$ |

*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.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

|  | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Ir1 | 0.25544 (2) | −0.00046 (2) | 0.00566 (2) | 0.02570 (9) |
| N1 | 0.3343 (3) | 0.06189 (18) | 0.0705 (2) | 0.0289 (10) |
| N2 | 0.1946 (3) | −0.00380 (17) | 0.1030 (2) | 0.0254 (9) |
| N3 | 0.3270 (3) | −0.07374 (19) | 0.0323 (2) | 0.0289 (10) |
| N4 | 0.2938 (3) | −0.12375 (18) | −0.0004 (2) | 0.0317 (10) |
| N5 | 0.1815 (3) | 0.06810 (19) | −0.0356 (2) | 0.0323 (10) |
| N6 | 0.2036 (3) | 0.08734 (19) | −0.1000 (2) | 0.0362 (11) |
| C1 | 0.4018 (4) | 0.0938 (2) | 0.0528 (3) | 0.0362 (13) |
| H1A | 0.4237 | 0.0871 | 0.0091 | 0.043* |
| C2 | 0.4418 (4) | 0.1377 (3) | 0.0976 (3) | 0.0416 (14) |
| H2A | 0.4888 | 0.1598 | 0.0831 | 0.050* |
| C3 | 0.4118 (4) | 0.1478 (2) | 0.1623 (3) | 0.0397 (14) |
| H3A | 0.4386 | 0.1764 | 0.1926 | 0.048* |
| C4 | 0.3391 (4) | 0.1142 (2) | 0.1827 (3) | 0.0348 (12) |
| C5 | 0.3015 (4) | 0.1213 (3) | 0.2491 (3) | 0.0408 (14) |
| H5A | 0.3250 | 0.1498 | 0.2811 | 0.049* |
| C6 | 0.2328 (4) | 0.0877 (2) | 0.2662 (3) | 0.0378 (13) |
| H6A | 0.2101 | 0.0930 | 0.3099 | 0.045* |
| C7 | 0.1946 (3) | 0.0440 (2) | 0.2182 (3) | 0.0296 (11) |
| C8 | 0.1236 (4) | 0.0068 (2) | 0.2333 (3) | 0.0334 (12) |
| H8A | 0.0989 | 0.0101 | 0.2764 | 0.040* |
| C9 | 0.0914 (4) | −0.0341 (2) | 0.1843 (3) | 0.0326 (12) |
| H9A | 0.0450 | −0.0589 | 0.1940 | 0.039* |
| C10 | 0.1287 (3) | −0.0384 (2) | 0.1192 (3) | 0.0300 (11) |
| H10A | 0.1063 | −0.0665 | 0.0863 | 0.036* |
| C11 | 0.2288 (3) | 0.0364 (2) | 0.1529 (2) | 0.0264 (11) |
| C12 | 0.3025 (3) | 0.0718 (2) | 0.1349 (3) | 0.0277 (11) |
| C13 | 0.4046 (4) | −0.0891 (3) | 0.0694 (3) | 0.0367 (13) |
| H13A | 0.4422 | −0.0638 | 0.0973 | 0.044* |
| C14 | 0.4207 (4) | −0.1483 (3) | 0.0602 (3) | 0.0445 (15) |
| H14A | 0.4698 | −0.1697 | 0.0805 | 0.053* |
| C15 | 0.3503 (4) | −0.1691 (3) | 0.0154 (3) | 0.0417 (14) |
| H15A | 0.3426 | −0.2073 | −0.0011 | 0.050* |
| C16 | 0.2090 (4) | −0.1185 (2) | −0.0414 (3) | 0.0302 (12) |
| C17 | 0.1642 (4) | −0.1663 (3) | −0.0724 (3) | 0.0422 (14) |
| H17A | 0.1892 | −0.2036 | −0.0679 | 0.051* |
| C18 | 0.0812 (4) | −0.1576 (3) | −0.1106 (3) | 0.0465 (16) |
| H18A | 0.0498 | −0.1892 | −0.1320 | 0.056* |
| C19 | 0.0454 (4) | −0.1023 (3) | −0.1166 (3) | 0.0426 (14) |

| | | | |
|---|---|---|---|
| H19A | −0.0103 | −0.0966 | −0.1424 | 0.051* |
| C20 | 0.0912 (4) | −0.0547 (2) | −0.0845 (3) | 0.0332 (12) |
| H20A | 0.0653 | −0.0176 | −0.0887 | 0.040* |
| C21 | 0.1757 (3) | −0.0614 (2) | −0.0458 (3) | 0.0287 (11) |
| C22 | 0.1125 (4) | 0.0995 (3) | −0.0200 (3) | 0.0428 (14) |
| H22A | 0.0840 | 0.0954 | 0.0213 | 0.051* |
| C23 | 0.0886 (4) | 0.1395 (3) | −0.0738 (4) | 0.0528 (17) |
| H23A | 0.0421 | 0.1666 | −0.0757 | 0.063* |
| C24 | 0.1476 (5) | 0.1307 (3) | −0.1235 (3) | 0.0515 (17) |
| H24A | 0.1487 | 0.1511 | −0.1661 | 0.062* |
| C25 | 0.2783 (4) | 0.0599 (2) | −0.1271 (3) | 0.0358 (13) |
| C26 | 0.3100 (4) | 0.0767 (3) | −0.1898 (3) | 0.0478 (16) |
| H26A | 0.2833 | 0.1077 | −0.2163 | 0.057* |
| C27 | 0.3814 (5) | 0.0474 (3) | −0.2130 (3) | 0.0514 (17) |
| H27A | 0.4039 | 0.0586 | −0.2551 | 0.062* |
| C28 | 0.4198 (5) | 0.0012 (3) | −0.1737 (3) | 0.0514 (17) |
| H28A | 0.4680 | −0.0188 | −0.1896 | 0.062* |
| C29 | 0.3863 (4) | −0.0159 (3) | −0.1097 (3) | 0.0371 (13) |
| H29A | 0.4128 | −0.0472 | −0.0839 | 0.045* |
| C30 | 0.3144 (4) | 0.0132 (2) | −0.0845 (3) | 0.0308 (12) |
| P1 | 0.35528 (11) | 0.28676 (7) | 0.33205 (8) | 0.0404 (4) |
| F1 | 0.3359 (4) | 0.2677 (2) | 0.2511 (2) | 0.105 (2) |
| F2 | 0.3773 (4) | 0.3061 (2) | 0.4122 (2) | 0.0877 (15) |
| F3 | 0.4239 (4) | 0.3339 (2) | 0.3101 (3) | 0.0978 (17) |
| F4 | 0.2835 (5) | 0.2436 (3) | 0.3520 (4) | 0.152 (3) |
| F5 | 0.2829 (4) | 0.3381 (3) | 0.3221 (4) | 0.140 (2) |
| F6 | 0.4276 (5) | 0.2399 (3) | 0.3409 (3) | 0.147 (3) |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Ir1 | 0.02768 (12) | 0.02890 (14) | 0.02103 (12) | −0.00258 (8) | 0.00499 (8) | 0.00069 (8) |
| N1 | 0.029 (2) | 0.029 (2) | 0.028 (2) | −0.0065 (19) | 0.0014 (19) | −0.0035 (18) |
| N2 | 0.026 (2) | 0.026 (2) | 0.024 (2) | −0.0003 (17) | 0.0036 (17) | 0.0014 (17) |
| N3 | 0.033 (2) | 0.034 (2) | 0.022 (2) | −0.0015 (19) | 0.0107 (19) | −0.0002 (18) |
| N4 | 0.042 (3) | 0.029 (2) | 0.025 (2) | −0.001 (2) | 0.008 (2) | 0.0023 (18) |
| N5 | 0.036 (3) | 0.029 (2) | 0.031 (2) | −0.005 (2) | 0.002 (2) | −0.0001 (19) |
| N6 | 0.042 (3) | 0.037 (3) | 0.029 (2) | −0.006 (2) | 0.000 (2) | 0.010 (2) |
| C1 | 0.040 (3) | 0.037 (3) | 0.032 (3) | −0.010 (3) | 0.004 (2) | −0.001 (2) |
| C2 | 0.037 (3) | 0.046 (4) | 0.042 (4) | −0.012 (3) | 0.005 (3) | 0.000 (3) |
| C3 | 0.040 (3) | 0.042 (3) | 0.035 (3) | −0.010 (3) | −0.005 (3) | −0.008 (3) |
| C4 | 0.039 (3) | 0.037 (3) | 0.028 (3) | −0.005 (2) | 0.000 (2) | −0.003 (2) |
| C5 | 0.053 (4) | 0.043 (3) | 0.026 (3) | −0.005 (3) | 0.002 (3) | −0.004 (2) |
| C6 | 0.043 (3) | 0.046 (3) | 0.026 (3) | −0.004 (3) | 0.008 (2) | −0.003 (2) |
| C7 | 0.033 (3) | 0.031 (3) | 0.024 (3) | 0.004 (2) | 0.003 (2) | 0.001 (2) |
| C8 | 0.037 (3) | 0.041 (3) | 0.023 (3) | 0.002 (2) | 0.013 (2) | 0.007 (2) |
| C9 | 0.035 (3) | 0.037 (3) | 0.027 (3) | 0.000 (2) | 0.009 (2) | 0.001 (2) |
| C10 | 0.031 (3) | 0.030 (3) | 0.029 (3) | −0.003 (2) | 0.007 (2) | 0.003 (2) |

| C11 | 0.031 (3) | 0.029 (3) | 0.019 (2) | 0.000 (2) | 0.000 (2) | −0.001 (2) |
|-----|-----------|-----------|-----------|-----------|-----------|------------|
| C12 | 0.027 (3) | 0.034 (3) | 0.022 (3) | 0.001 (2) | 0.001 (2) | −0.001 (2) |
| C13 | 0.029 (3) | 0.052 (4) | 0.029 (3) | 0.000 (3) | 0.001 (2) | 0.007 (3) |
| C14 | 0.047 (4) | 0.045 (4) | 0.042 (4) | 0.014 (3) | 0.008 (3) | 0.014 (3) |
| C15 | 0.055 (4) | 0.034 (3) | 0.038 (3) | 0.007 (3) | 0.014 (3) | 0.008 (3) |
| C16 | 0.039 (3) | 0.033 (3) | 0.019 (3) | −0.005 (2) | 0.006 (2) | −0.002 (2) |
| C17 | 0.065 (4) | 0.035 (3) | 0.027 (3) | −0.008 (3) | 0.009 (3) | 0.000 (2) |
| C18 | 0.059 (4) | 0.046 (4) | 0.033 (3) | −0.028 (3) | 0.000 (3) | −0.005 (3) |
| C19 | 0.041 (3) | 0.057 (4) | 0.029 (3) | −0.014 (3) | 0.000 (3) | 0.002 (3) |
| C20 | 0.034 (3) | 0.043 (3) | 0.022 (3) | −0.008 (2) | 0.005 (2) | 0.001 (2) |
| C21 | 0.034 (3) | 0.035 (3) | 0.019 (2) | −0.009 (2) | 0.010 (2) | 0.000 (2) |
| C22 | 0.033 (3) | 0.044 (3) | 0.051 (4) | 0.010 (3) | 0.004 (3) | −0.001 (3) |
| C23 | 0.042 (4) | 0.051 (4) | 0.063 (5) | 0.007 (3) | −0.009 (3) | 0.011 (3) |
| C24 | 0.062 (4) | 0.045 (4) | 0.044 (4) | 0.001 (3) | −0.011 (3) | 0.018 (3) |
| C25 | 0.038 (3) | 0.041 (3) | 0.028 (3) | −0.007 (3) | 0.001 (2) | 0.002 (2) |
| C26 | 0.061 (4) | 0.047 (4) | 0.035 (3) | −0.019 (3) | 0.004 (3) | 0.011 (3) |
| C27 | 0.058 (4) | 0.071 (5) | 0.028 (3) | −0.026 (4) | 0.016 (3) | −0.002 (3) |
| C28 | 0.051 (4) | 0.072 (5) | 0.033 (3) | −0.017 (3) | 0.015 (3) | −0.009 (3) |
| C29 | 0.038 (3) | 0.050 (3) | 0.025 (3) | −0.008 (3) | 0.012 (2) | −0.005 (2) |
| C30 | 0.032 (3) | 0.044 (3) | 0.017 (2) | −0.012 (2) | 0.004 (2) | −0.004 (2) |
| P1 | 0.0506 (9) | 0.0377 (8) | 0.0325 (8) | −0.0028 (7) | 0.0030 (7) | 0.0021 (6) |
| F1 | 0.163 (5) | 0.105 (4) | 0.041 (3) | −0.067 (4) | −0.014 (3) | 0.005 (2) |
| F2 | 0.127 (4) | 0.095 (3) | 0.041 (2) | −0.017 (3) | 0.009 (2) | −0.019 (2) |
| F3 | 0.128 (4) | 0.083 (3) | 0.086 (3) | −0.054 (3) | 0.027 (3) | −0.018 (3) |
| F4 | 0.198 (7) | 0.158 (6) | 0.116 (5) | −0.125 (5) | 0.084 (5) | −0.035 (4) |
| F5 | 0.103 (4) | 0.108 (4) | 0.200 (7) | 0.049 (4) | −0.023 (4) | 0.011 (4) |
| F6 | 0.183 (7) | 0.146 (5) | 0.104 (5) | 0.135 (5) | −0.024 (4) | −0.014 (4) |

*Geometric parameters (Å, º)*

| | | | |
|-----------|-----------|-----------|-----------|
| Ir1—C21 | 2.016 (5) | C11—C12 | 1.437 (7) |
| Ir1—C30 | 2.019 (5) | C13—C14 | 1.388 (8) |
| Ir1—N3 | 2.021 (4) | C13—H13A | 0.9300 |
| Ir1—N5 | 2.025 (4) | C14—C15 | 1.365 (9) |
| Ir1—N2 | 2.133 (4) | C14—H14A | 0.9300 |
| Ir1—N1 | 2.148 (4) | C15—H15A | 0.9300 |
| N1—C1 | 1.317 (7) | C16—C17 | 1.378 (7) |
| N1—C12 | 1.367 (6) | C16—C21 | 1.394 (7) |
| N2—C10 | 1.324 (6) | C17—C18 | 1.383 (9) |
| N2—C11 | 1.374 (6) | C17—H17A | 0.9300 |
| N3—C13 | 1.339 (7) | C18—C19 | 1.370 (9) |
| N3—N4 | 1.366 (6) | C18—H18A | 0.9300 |
| N4—C15 | 1.349 (7) | C19—C20 | 1.390 (8) |
| N4—C16 | 1.420 (7) | C19—H19A | 0.9300 |
| N5—C22 | 1.315 (7) | C20—C21 | 1.401 (7) |
| N5—N6 | 1.364 (6) | C20—H20A | 0.9300 |
| N6—C24 | 1.341 (7) | C22—C23 | 1.383 (8) |
| N6—C25 | 1.422 (7) | C22—H22A | 0.9300 |

| | | | |
|---|---|---|---|
| C1—C2 | 1.402 (8) | C23—C24 | 1.369 (9) |
| C1—H1A | 0.9300 | C23—H23A | 0.9300 |
| C2—C3 | 1.364 (8) | C24—H24A | 0.9300 |
| C2—H2A | 0.9300 | C25—C26 | 1.374 (8) |
| C3—C4 | 1.418 (8) | C25—C30 | 1.408 (8) |
| C3—H3A | 0.9300 | C26—C27 | 1.372 (9) |
| C4—C12 | 1.395 (7) | C26—H26A | 0.9300 |
| C4—C5 | 1.434 (8) | C27—C28 | 1.378 (9) |
| C5—C6 | 1.350 (8) | C27—H27A | 0.9300 |
| C5—H5A | 0.9300 | C28—C29 | 1.409 (8) |
| C6—C7 | 1.425 (7) | C28—H28A | 0.9300 |
| C6—H6A | 0.9300 | C29—C30 | 1.391 (8) |
| C7—C11 | 1.393 (7) | C29—H29A | 0.9300 |
| C7—C8 | 1.413 (7) | P1—F6 | 1.520 (5) |
| C8—C9 | 1.364 (7) | P1—F4 | 1.534 (5) |
| C8—H8A | 0.9300 | P1—F3 | 1.572 (5) |
| C9—C10 | 1.404 (7) | P1—F2 | 1.575 (4) |
| C9—H9A | 0.9300 | P1—F1 | 1.584 (5) |
| C10—H10A | 0.9300 | P1—F5 | 1.593 (5) |
| | | | |
| C21—Ir1—C30 | 89.44 (19) | N3—C13—C14 | 110.0 (5) |
| C21—Ir1—N3 | 79.7 (2) | N3—C13—H13A | 125.0 |
| C30—Ir1—N3 | 94.0 (2) | C14—C13—H13A | 125.0 |
| C21—Ir1—N5 | 94.3 (2) | C15—C14—C13 | 106.3 (5) |
| C30—Ir1—N5 | 80.0 (2) | C15—C14—H14A | 126.8 |
| N3—Ir1—N5 | 171.64 (16) | C13—C14—H14A | 126.8 |
| C21—Ir1—N2 | 96.00 (17) | N4—C15—C14 | 107.3 (5) |
| C30—Ir1—N2 | 173.09 (18) | N4—C15—H15A | 126.3 |
| N3—Ir1—N2 | 91.16 (15) | C14—C15—H15A | 126.3 |
| N5—Ir1—N2 | 95.29 (16) | C17—C16—C21 | 123.9 (5) |
| C21—Ir1—N1 | 173.97 (17) | C17—C16—N4 | 122.2 (5) |
| C30—Ir1—N1 | 96.56 (18) | C21—C16—N4 | 114.0 (4) |
| N3—Ir1—N1 | 99.24 (17) | C16—C17—C18 | 118.6 (6) |
| N5—Ir1—N1 | 87.33 (17) | C16—C17—H17A | 120.7 |
| N2—Ir1—N1 | 78.06 (15) | C18—C17—H17A | 120.7 |
| C1—N1—C12 | 118.8 (4) | C19—C18—C17 | 119.8 (5) |
| C1—N1—Ir1 | 127.8 (4) | C19—C18—H18A | 120.1 |
| C12—N1—Ir1 | 113.1 (3) | C17—C18—H18A | 120.1 |
| C10—N2—C11 | 118.2 (4) | C18—C19—C20 | 120.9 (6) |
| C10—N2—Ir1 | 128.0 (3) | C18—C19—H19A | 119.6 |
| C11—N2—Ir1 | 113.8 (3) | C20—C19—H19A | 119.6 |
| C13—N3—N4 | 105.8 (4) | C19—C20—C21 | 121.2 (5) |
| C13—N3—Ir1 | 139.0 (4) | C19—C20—H20A | 119.4 |
| N4—N3—Ir1 | 114.8 (3) | C21—C20—H20A | 119.4 |
| C15—N4—N3 | 110.5 (5) | C16—C21—C20 | 115.7 (5) |
| C15—N4—C16 | 133.6 (5) | C16—C21—Ir1 | 115.2 (4) |
| N3—N4—C16 | 116.0 (4) | C20—C21—Ir1 | 129.2 (4) |
| C22—N5—N6 | 107.0 (5) | N5—C22—C23 | 110.2 (6) |

| | | | |
|---|---|---|---|
| C22—N5—Ir1 | 138.2 (4) | N5—C22—H22A | 124.9 |
| N6—N5—Ir1 | 114.6 (3) | C23—C22—H22A | 124.9 |
| C24—N6—N5 | 109.3 (5) | C24—C23—C22 | 105.6 (6) |
| C24—N6—C25 | 133.9 (5) | C24—C23—H23A | 127.2 |
| N5—N6—C25 | 116.8 (4) | C22—C23—H23A | 127.2 |
| N1—C1—C2 | 122.2 (5) | N6—C24—C23 | 107.9 (5) |
| N1—C1—H1A | 118.9 | N6—C24—H24A | 126.0 |
| C2—C1—H1A | 118.9 | C23—C24—H24A | 126.0 |
| C3—C2—C1 | 120.0 (5) | C26—C25—C30 | 123.7 (6) |
| C3—C2—H2A | 120.0 | C26—C25—N6 | 122.8 (5) |
| C1—C2—H2A | 120.0 | C30—C25—N6 | 113.4 (5) |
| C2—C3—C4 | 119.0 (5) | C27—C26—C25 | 119.2 (6) |
| C2—C3—H3A | 120.5 | C27—C26—H26A | 120.4 |
| C4—C3—H3A | 120.5 | C25—C26—H26A | 120.4 |
| C12—C4—C3 | 117.5 (5) | C26—C27—C28 | 119.9 (6) |
| C12—C4—C5 | 118.6 (5) | C26—C27—H27A | 120.1 |
| C3—C4—C5 | 123.9 (5) | C28—C27—H27A | 120.1 |
| C6—C5—C4 | 121.5 (5) | C27—C28—C29 | 120.4 (6) |
| C6—C5—H5A | 119.2 | C27—C28—H28A | 119.8 |
| C4—C5—H5A | 119.2 | C29—C28—H28A | 119.8 |
| C5—C6—C7 | 120.7 (5) | C30—C29—C28 | 121.1 (6) |
| C5—C6—H6A | 119.6 | C30—C29—H29A | 119.4 |
| C7—C6—H6A | 119.6 | C28—C29—H29A | 119.4 |
| C11—C7—C8 | 117.1 (5) | C29—C30—C25 | 115.6 (5) |
| C11—C7—C6 | 119.3 (5) | C29—C30—Ir1 | 129.5 (4) |
| C8—C7—C6 | 123.6 (5) | C25—C30—Ir1 | 114.9 (4) |
| C9—C8—C7 | 119.7 (5) | F6—P1—F4 | 91.8 (5) |
| C9—C8—H8A | 120.2 | F6—P1—F3 | 91.8 (4) |
| C7—C8—H8A | 120.2 | F4—P1—F3 | 176.4 (4) |
| C8—C9—C10 | 119.6 (5) | F6—P1—F2 | 91.0 (3) |
| C8—C9—H9A | 120.2 | F4—P1—F2 | 91.5 (3) |
| C10—C9—H9A | 120.2 | F3—P1—F2 | 89.1 (3) |
| N2—C10—C9 | 122.3 (5) | F6—P1—F1 | 88.2 (3) |
| N2—C10—H10A | 118.9 | F4—P1—F1 | 89.8 (3) |
| C9—C10—H10A | 118.9 | F3—P1—F1 | 89.6 (3) |
| N2—C11—C7 | 122.9 (5) | F2—P1—F1 | 178.4 (3) |
| N2—C11—C12 | 117.0 (4) | F6—P1—F5 | 177.3 (4) |
| C7—C11—C12 | 120.0 (4) | F4—P1—F5 | 90.9 (4) |
| N1—C12—C4 | 122.5 (5) | F3—P1—F5 | 85.5 (3) |
| N1—C12—C11 | 117.7 (4) | F2—P1—F5 | 88.8 (3) |
| C4—C12—C11 | 119.8 (4) | F1—P1—F5 | 92.0 (4) |
| | | | |
| C13—N3—N4—C15 | −0.8 (5) | C7—C11—C12—C4 | −0.5 (7) |
| Ir1—N3—N4—C15 | 174.3 (3) | N4—N3—C13—C14 | 0.2 (6) |
| C13—N3—N4—C16 | 177.8 (4) | Ir1—N3—C13—C14 | −173.0 (4) |
| Ir1—N3—N4—C16 | −7.1 (5) | N3—C13—C14—C15 | 0.4 (6) |
| C22—N5—N6—C24 | 0.0 (6) | N3—N4—C15—C14 | 1.0 (6) |
| Ir1—N5—N6—C24 | 177.0 (4) | C16—N4—C15—C14 | −177.2 (5) |

| | | | |
|---|---|---|---|
| C22—N5—N6—C25 | 178.9 (5) | C13—C14—C15—N4 | −0.9 (6) |
| Ir1—N5—N6—C25 | −4.1 (6) | C15—N4—C16—C17 | 3.6 (9) |
| C12—N1—C1—C2 | −0.2 (8) | N3—N4—C16—C17 | −174.6 (5) |
| Ir1—N1—C1—C2 | 173.0 (4) | C15—N4—C16—C21 | −177.5 (5) |
| N1—C1—C2—C3 | 0.9 (9) | N3—N4—C16—C21 | 4.3 (6) |
| C1—C2—C3—C4 | −1.0 (9) | C21—C16—C17—C18 | 0.2 (8) |
| C2—C3—C4—C12 | 0.4 (8) | N4—C16—C17—C18 | 179.0 (5) |
| C2—C3—C4—C5 | −179.5 (6) | C16—C17—C18—C19 | −0.1 (8) |
| C12—C4—C5—C6 | 0.8 (9) | C17—C18—C19—C20 | −0.3 (9) |
| C3—C4—C5—C6 | −179.3 (6) | C18—C19—C20—C21 | 0.8 (8) |
| C4—C5—C6—C7 | −0.6 (9) | C17—C16—C21—C20 | 0.3 (7) |
| C5—C6—C7—C11 | −0.1 (8) | N4—C16—C21—C20 | −178.6 (4) |
| C5—C6—C7—C8 | 179.0 (5) | C17—C16—C21—Ir1 | 179.4 (4) |
| C11—C7—C8—C9 | −0.5 (8) | N4—C16—C21—Ir1 | 0.5 (5) |
| C6—C7—C8—C9 | −179.6 (5) | C19—C20—C21—C16 | −0.8 (7) |
| C7—C8—C9—C10 | −0.5 (8) | C19—C20—C21—Ir1 | −179.8 (4) |
| C11—N2—C10—C9 | 1.9 (7) | N6—N5—C22—C23 | 0.1 (7) |
| Ir1—N2—C10—C9 | −176.7 (4) | Ir1—N5—C22—C23 | −175.8 (4) |
| C8—C9—C10—N2 | −0.2 (8) | N5—C22—C23—C24 | −0.1 (7) |
| C10—N2—C11—C7 | −3.0 (7) | N5—N6—C24—C23 | −0.1 (7) |
| Ir1—N2—C11—C7 | 175.8 (4) | C25—N6—C24—C23 | −178.8 (6) |
| C10—N2—C11—C12 | 177.8 (4) | C22—C23—C24—N6 | 0.1 (7) |
| Ir1—N2—C11—C12 | −3.4 (5) | C24—N6—C25—C26 | 1.4 (10) |
| C8—C7—C11—N2 | 2.3 (7) | N5—N6—C25—C26 | −177.2 (5) |
| C6—C7—C11—N2 | −178.5 (5) | C24—N6—C25—C30 | −176.9 (6) |
| C8—C7—C11—C12 | −178.5 (5) | N5—N6—C25—C30 | 4.5 (7) |
| C6—C7—C11—C12 | 0.7 (8) | C30—C25—C26—C27 | −0.7 (9) |
| C1—N1—C12—C4 | −0.5 (8) | N6—C25—C26—C27 | −178.9 (5) |
| Ir1—N1—C12—C4 | −174.6 (4) | C25—C26—C27—C28 | 0.7 (9) |
| C1—N1—C12—C11 | 180.0 (5) | C26—C27—C28—C29 | −0.3 (9) |
| Ir1—N1—C12—C11 | 5.9 (6) | C27—C28—C29—C30 | −0.2 (9) |
| C3—C4—C12—N1 | 0.3 (8) | C28—C29—C30—C25 | 0.2 (8) |
| C5—C4—C12—N1 | −179.8 (5) | C28—C29—C30—Ir1 | −178.3 (4) |
| C3—C4—C12—C11 | 179.9 (5) | C26—C25—C30—C29 | 0.2 (8) |
| C5—C4—C12—C11 | −0.2 (8) | N6—C25—C30—C29 | 178.5 (5) |
| N2—C11—C12—N1 | −1.7 (7) | C26—C25—C30—Ir1 | 179.0 (4) |
| C7—C11—C12—N1 | 179.1 (5) | N6—C25—C30—Ir1 | −2.7 (6) |
| N2—C11—C12—C4 | 178.7 (5) | | |

*Hydrogen-bond geometry (Å, °)*

*Cg*4 and *Cg*9 are the centroids of rings N3/N4/C15–C13 and C16–C21, respectively.

| D—H···A | D—H | H···A | D···A | D—H···A |
|---|---|---|---|---|
| C2—H2A···F6[i] | 0.93 | 2.57 | 3.184 (7) | 124 |
| C9—H9A···F3[ii] | 0.93 | 2.49 | 3.024 (7) | 117 |
| C17—H17A···F4[iii] | 0.93 | 2.36 | 2.977 (8) | 124 |
| C23—H23A···F2[iv] | 0.93 | 2.53 | 3.383 (8) | 152 |
| C24—H24A···F1[v] | 0.93 | 2.48 | 3.368 (8) | 161 |

| | | | | |
|---|---|---|---|---|
| C26—H26*A*···F5$^{v}$ | 0.93 | 2.47 | 3.348 (9) | 158 |
| C6—H6*A*···*Cg*9$^{vi}$ | 0.93 | 2.58 | 3.501 (6) | 173 |
| C29—H29*A*···*Cg*4 | 0.93 | 2.98 | 3.688 (7) | 134 |

Symmetry codes: (i) −*x*+1, *y*, −*z*+1/2; (ii) −*x*+1/2, *y*−1/2, −*z*+1/2; (iii) *x*, −*y*, *z*−1/2; (iv) *x*−1/2, −*y*+1/2, *z*−1/2; (v) −*x*+1/2, −*y*+1/2, −*z*; (vi) −*x*+1/2, *y*+1/2, −*z*+1/2.