

Crystal structure of bis{2-[5-(3,4,5-trimethoxyphenyl)-4*H*-1,2,4-triazol-3-yl]pyridine}palladium(II) bis(trifluoroacetate) trifluoroacetic acid disolvate

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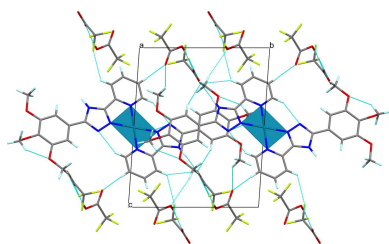
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The new palladium(II) complex, [Pd(C₁₆H₁₆N₄O₃)₂](CF₃COO)₂·2CF₃COOH, crystallizes in the triclinic space group $P\bar{1}$ with the asymmetric unit containing half the cation (Pd^{II} site symmetry C_i), one trifluoroacetate anion and one co-crystallized trifluoroacetic acid molecule. Two neutral chelating 2-[5-(3,4,5-trimethoxyphenyl)-4*H*-1,2,4-triazol-3-yl]pyridine ligands coordinate to the Pd^{II} ion through the triazole-N and pyridine-N atoms in a distorted *trans*-PdN₄ square-planar configuration [Pd–N 1.991 (2), 2.037 (2) Å; *cis* N–Pd–N 79.65 (8), 100.35 (8)°]. The complex cation is quite planar, except for the methoxy groups ($\delta = 0.117$ Å for one of the C atoms). The planar configuration is supported by two intramolecular C–H···N hydrogen bonds. In the crystal, the π – π -stacked cations are arranged in sheets parallel to the *ab* plane that are flanked on both sides by the trifluoroacetic acid–trifluoroacetate anion pairs. Apart from classical N/O–H···O hydrogen-bonding interactions, weak C–H···F/N/O contacts consolidate the three-dimensional architecture. Both trifluoroacetic moieties were found to be disordered over two resolvable positions with a refined occupancy ratio of 0.587 (1):0.413 (17) and 0.530 (6):0.470 (6) for the protonated and deprotonated forms, respectively.

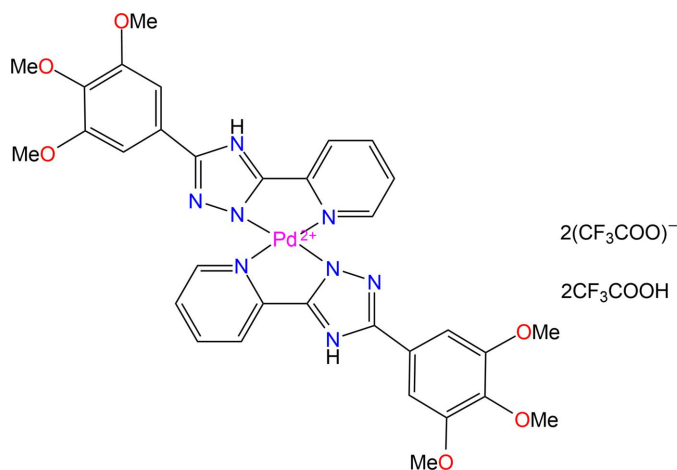
1. Chemical context

Triazoles are five-membered heterocyclic compounds containing three nitrogen atoms and two carbon atoms in the ring. They can exist in different isomeric forms, such as 1,2,3-triazole and 1,2,4-triazole. 1,2,4-Triazole derivatives are of interest in various research fields ranging from medicinal chemistry and pharmaceuticals (Aggarwal & Sumran, 2020; Leenders *et al.*, 2021) to materials science (Farooq, 2020). Versatile coordination behaviour due to the presence of neutral, anionic or cationic nitrogen donors (N-coordination) as well as carbanionic donors (C-coordination) makes 1,2,4-triazoles appealing ligands for the construction of metal complexes with useful functionalities (Song *et al.*, 2019; Feltham *et al.*, 2017; Kumar *et al.*, 2015; Wen *et al.*, 2017). Substitution reactions at the azole ring create a virtually unlimited range of chemical and structural variations to tune the desired characteristics of the resulting complexes.

In our ongoing project exploring the rich potential of 1,2,4-triazoles in coordination and supramolecular chemistry, a number of new metal complexes bearing 3-(pyridin-2-yl)-1,2,4-triazole derivatives as ligands were prepared. The Cu^{II}, Ru^{II}, Pd^{II}, Eu^{III}, Tb^{III} and Pt^{II} compounds revealed promising magnetic (Petrenko *et al.*, 2021), catalytic (Zakharchenko *et al.*, 2019) and luminescent properties (Khomenko *et al.*, 2015,



2023), as well as antiproliferative activity against several human cancer cell lines (Ohorodnik *et al.*, 2022, 2023).



In the present study, the crystal structure of $[\text{Pd}(\text{HL})_2](\text{CF}_3\text{COO})_2 \cdot 2\text{CF}_3\text{COOH}$ (I), where HL is 2-[5-(3,4,5-trimethoxyphenyl)-4*H*-1,2,4-triazol-3-yl]pyridine, is reported. The title compound was isolated in an attempt to recrystallize its neutral precursor PdL_2 from trifluoroacetic acid (TFA). PdL_2 was prepared and studied with IR, UV-Vis, NMR and photoluminescence spectroscopy, as well as MALDI mass spectrometry in solution and solid state but not structurally characterized (Zakharchenko *et al.*, 2016).

2. Structural commentary

The title compound is assembled from discrete $[\text{Pd}(\text{HL})_2]^{2+}$ cations (the Pd^{II} atom is located on a special position with C_i site symmetry), CF_3COO^- anions, and CF_3COOH molecules of crystallization in a 1:2:2 ratio (Fig. 1). Both neutral HL molecules are coordinated to the metal atom as bidentate ligands through the triazole-N2 and pyridine-N1 atoms in a *trans*-configuration. The square-planar N_4 environment of the

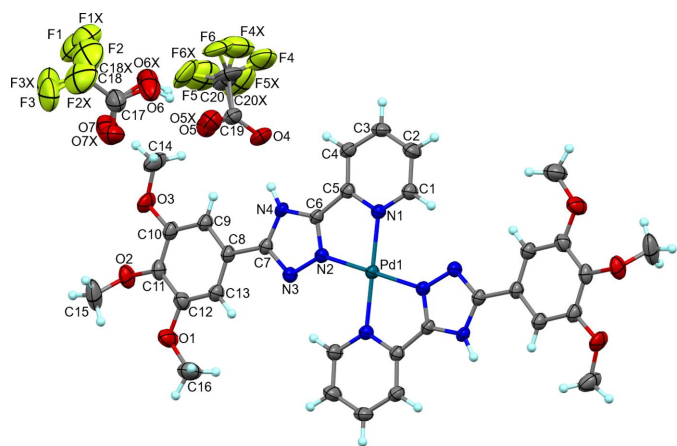


Figure 1
Extended view of the asymmetric unit of (I) with the atom labelling and displacement ellipsoids at the 50% probability level showing the coordination environment of the metal atom.

Table 1
Selected geometric parameters (\AA , $^\circ$).

Pd1—N2	1.991 (2)	Pd1—N1	2.037 (2)
N2—Pd1—N1 ⁱ	100.35 (8)	N2—Pd1—N1	79.65 (8)

Symmetry code: (i) $-x + 2, -y + 2, -z + 1$.

Table 2
Hydrogen-bond geometry (\AA , $^\circ$).

<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>
O6—H6...O5	0.84	1.73	2.56 (3)	172
O6 ^X —H6 ^X ...F6 ^X	0.86	2.27	2.77 (3)	118
N4—H4...O4	0.86	1.81	2.655 (3)	166
C15—H15A...N3 ⁱⁱ	0.96	2.59	3.334 (5)	134
C15—H15B...O1	0.96	2.35	2.929 (5)	118
C15—H15C...O3 ⁱⁱⁱ	0.96	2.49	3.400 (4)	158
C14—H14A...O7	0.96	2.67	3.492 (17)	144
C4—H4A...F6 ^{iv}	0.93	2.58	3.193 (6)	124
C4—H4A...O4	0.93	2.59	3.425 (4)	150
C9—H9...O4	0.93	2.63	3.510 (4)	158
C2—H2...O7 ^v	0.93	2.44	3.363 (14)	174
C2—H2...O7 ^{Xv}	0.93	2.57	3.498 (19)	178
C1—H1...N3 ⁱ	0.93	2.34	3.146 (3)	145
C16—H16B...F4 ^{vi}	0.96	2.52	3.400 (7)	152

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

Pd^{II} centre is moderately distorted with the two Pd—N distances and two *cis* N—Pd—N angles differing by 0.046 (2) \AA and 20.70 (8) $^\circ$, respectively (Table 1). The $[\text{Pd}(\text{HL})_2]^{2+}$ cation, except for the methoxy groups, is almost planar with the largest deviation from the mean plane being 0.117 \AA (C11). Two intramolecular hydrogen bonds, C1—H1...N3ⁱ and C15—H15B...O1, with an $S(6)$ graph-set motif are observed (Fig. 2, Table 2; symmetry code as given in Table 2) (Etter, 1990). The C—H...N $S(6)$ rings support the planar configuration of the cation.

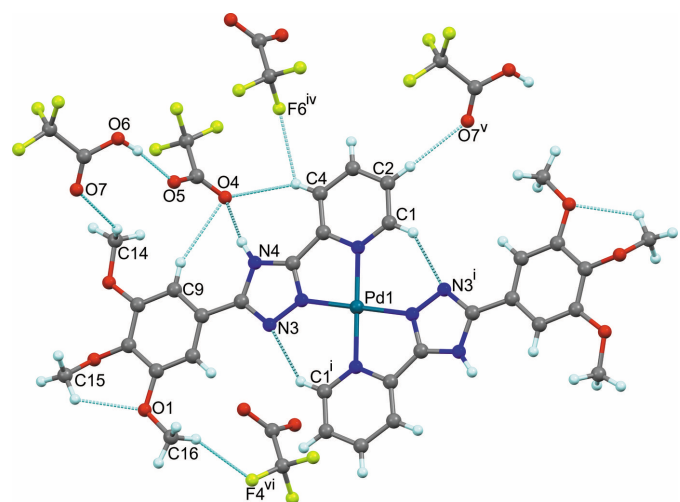


Figure 2
Intramolecular C1—H1...N3ⁱ and C15—H15B...O1 hydrogen-bonding interactions forming rings of $S(6)$ graph-set motif and intermolecular hydrogen bonds involving the trifluoroacetate moieties of (I) (blue dashed lines). Minor disorder components have been omitted for clarity. Symmetry codes as given in Table 2.

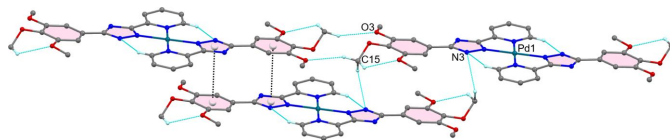


Figure 3

[Pd(HL)₂]²⁺ cations of (I) joined by aromatic stacking between triazole and benzene rings of the centrosymmetrically related ligands (black dashed lines) and intermolecular C15–H15A···N3ⁱⁱⁱ and C15–H15C···O3ⁱⁱⁱ interactions forming rings with R₄⁴(18) and R₂²(12) graph-set motifs (blue dashed lines). H atoms not involved in hydrogen bonding have been omitted for clarity. [Symmetry codes: (ii) $-x + 1, -y + 1, -z + 1$; (iii) $-x + 1, -y, -z + 1$.]

The C–O bond distances for disordered carboxylic [1.177 (7)/1.174 (8), 1.273 (7)/1.275 (8) Å] and carboxylate units [1.223 (4), 1.227 (11)/1.231 (13) Å] unequivocally confirm the molecular and anionic forms of the TFA and TFA[−] anion, respectively.

3. Supramolecular features

The crystal structure is built up of an alternate arrangement of distinct cationic and anionic supramolecular layers oriented in the *ab* plane. In the cationic layer (Fig. 3), the face-to-face aromatic stacking between triazole and benzene rings of the centrosymmetrically related ligands is significantly offset, as evidenced by a centroid-to-centroid distance of 3.566 (2) Å with the interplanar distance and tilt angle being 3.263 Å and 76.26°, respectively. The [Pd(HL)₂]²⁺ cations are additionally intertwined by weak C15–H15A···N3ⁱⁱⁱ and C15–H15C···O3ⁱⁱⁱ hydrogen-bonding interactions (Table 2; symmetry codes as given in Table 2) forming rings of R₄⁴(18) and R₂²(12) graph-set motifs. The closest Pd···Pd separation in the layer exceeds 10 Å.

Within the anionic layer, the TFA molecule acts as a proton donor in hydrogen bonding towards the TFA[−] anion (O6–H6···O5; Fig. 2). The TFA–TFA[−] pairs stack on both sides of the cationic layers and create a three-dimensional C/N–H···F/O hydrogen-bonded network (Fig. 4). C4–H4A···O4, N4–H4···O4, C9–H9···O4 and

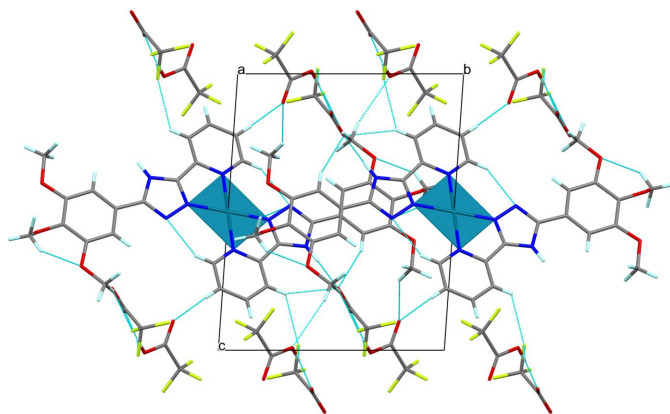


Figure 4

Fragment of the crystal packing of (I) viewed along the *a* axis showing the alternate arrangement of cationic and anionic supramolecular layers interacting through numerous C/N–H···F/O contacts. Minor disorder components have been omitted for clarity.

C14–H14A···O7 interactions between the cation and anion generate interconnected rings exhibiting R₂¹(7) and R₃³(13) graph-set motifs (Fig. 2).

4. Database survey

More than 1400 crystal structures of metal complexes featuring the 3-(pyridin-2-yl)-1,2,4-triazole backbone having various substituents in the rings are found in the Cambridge Structural Database (CSD, Version 5.45, update of November 2023; Groom *et al.*, 2016) with the nuclearity up to 24 metal (Co) centres (BIBHUS; Yao *et al.*, 2018). The only solid-state structure comprising HL, the Re^I carbonyl [ReBr(HL)(CO)₃]·CH₃OH (GAMTOG; Kharlova *et al.*, 2017), differs from (I) in the position of the acidic NH function in the triazole ring. Of nine palladium compounds with 3-(pyridin-2-yl)-1,2,4-triazole derivatives, eight were reported by our research group. In the Pd^{II} complexes, the substituted 3-(pyridin-2-yl)-1,2,4-triazole ligands in the neutral or anionic form coordinate to the metal atom through the pyridine-N and either triazole-N1 (TOFXUK, TOFYAR, TOGNEL, TOGNIP; Zakharchenko *et al.*, 2019) or triazole-N4 atoms (CAMSUI; Zakharchenko *et al.*, 2021a). Another example of the N4 protonation is found in hydrogen bis[2-[3-(pyridin-2-yl)-1,2,4-triazol-1-yl]propanoate] (CIPCUA; Gallagher *et al.*, 2007), which is a co-crystal of a neutral molecule and a zwitterion with a protonated N4 atom. Most similar, but not isomorphous, to the title compound is [Pd(HL')₂](CF₃COO)₂·4CF₃COOH with the neutral ligand HL' having a phenyl group instead of the trimethoxyphenyl substituent in (I), which also crystallizes in the triclinic space group *P* $\bar{1}$ (KEFKUF; Zakharchenko *et al.*, 2021b).

5. Synthesis and crystallization

The initial complex PdL₂ was synthesized according to the previously published method (Zakharchenko *et al.*, 2016). X-ray quality crystals of the title compound were obtained by recrystallization of PdL₂ from TFA. The compound was characterized by IR and ¹H NMR spectroscopy; it starts to decompose above 548 K. FT-IR (KBr pellet), ν (cm^{−1}): 3434br, 3104, 3110, 3010, 2948, 2926, 2850, 1776, 1676, 1638, 1618, 1596, 1490s, 1470, 1430, 1292, 1196s, 1178s, 1130vs, 1036, 1006, 842, 796, 726, 704, 598, 568, 524.

The IR spectrum of (I) (Fig. 5) is dominated by peaks associated with trifluoroacetic moieties, which are absent in the spectrum of PdL₂ (Zakharchenko *et al.*, 2016). TFA molecules are detected by an intense broad band due to ν (O–H) vibration centred at about 3430 cm^{−1} and a smaller band at 1776 cm^{−1} ascribed to ν (C=O) stretching. Two medium intensity bands observed at 1676 and 1430 cm^{−1} are assigned to ν_{as} (COO) and ν_s (COO) stretching modes of the TFA[−] anion, respectively. As expected, major absorption peaks at 1196, 1178 and 1130 cm^{−1} are present in the C–F stretching region (1110–1220 cm^{−1}). ν (C=N) and ν (C=C) stretching frequencies of the 1,2,4-triazole ligand in the range 1638–1596 cm^{−1} cannot be easily distinguished. Several bands

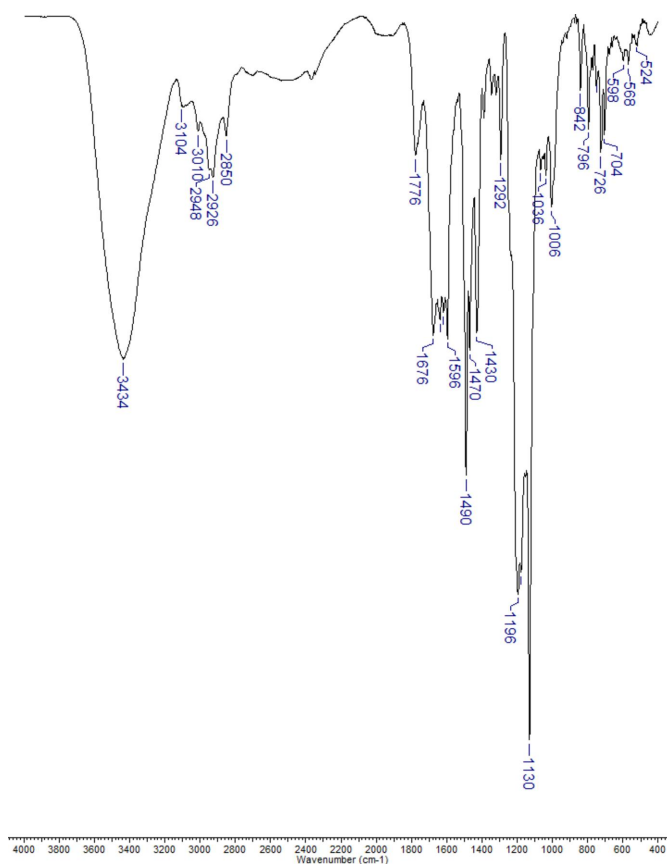


Figure 5
IR spectrum of (I) in the 4000–400 cm⁻¹ range.

observed above and below 3000 cm⁻¹ are assigned to aromatic and methyl group $\nu(\text{C-H})$ vibrations, respectively. A low intensity broad absorption at 3104 cm⁻¹ can be ascribed to $\nu(\text{N-H})$ stretching of the hydrogen-bonded N4H group of the triazole ring.

Due to very poor solubility of the title compound in organic solvents, it was not possible to obtain its satisfactory ¹H NMR spectrum in CDCl₃. Only the protons of the methoxy groups are distinctly observed as two singlets in a 2:1 ratio at 4.01 and 3.96 ppm while the aromatic protons in the 10–7 ppm range were indistinguishable from the background. The presence of TFA molecules and trace amounts of water in the solvent leads to significant broadening of the N4H signal to the point of disappearing in the spectrum. On the contrary, in the ¹H NMR spectrum of the free HL ligand in CDCl₃ the acidic N2-bound proton appears as a broadened singlet at 13.31 ppm (Zakharchenko *et al.*, 2016).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. Both trifluoroacetic moieties were found to be disordered over two resolvable positions with a refined occupancy ratio of 0.587 (1):0.413 (17) and 0.530 (6):0.470 (6) for the protonated and deprotonated forms, respectively. The disorder was restrained using SIMU and RIGU commands in *SHELXL* for the ten resulting atoms

Table 3
Experimental details.

Crystal data	
Chemical formula	[Pd(C ₁₆ H ₁₆ N ₄ O ₃) ₂](C ₂ F ₃ O ₂) ₂ ·2C ₂ HF ₃ O ₂
<i>M_r</i>	1185.15
Crystal system, space group	Triclinic, <i>P</i> $\bar{1}$
Temperature (K)	293
<i>a</i> , <i>b</i> , <i>c</i> (Å)	8.6173 (4), 10.6265 (6), 13.1312 (4)
α , β , γ (°)	93.384 (4), 98.121 (3), 94.090 (4)
<i>V</i> (Å ³)	1184.46 (9)
<i>Z</i>	1
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	0.51
Crystal size (mm)	0.4 × 0.3 × 0.3
Data collection	
Diffractometer	Xcalibur, Eos
Absorption correction	Multi-scan (<i>CrysAlis PRO</i> ; Rigaku OD, 2019)
<i>T_{min}</i> , <i>T_{max}</i>	0.968, 1.000
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	9462, 5010, 4615
<i>R_{int}</i>	0.026
($\sin \theta/\lambda$) _{max} (Å ⁻¹)	0.633
Refinement	
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.042, 0.095, 1.04
No. of reflections	5010
No. of parameters	444
No. of restraints	382
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ⁻³)	0.39, -0.45

Computer programs: *CrysAlis PRO* (Rigaku OD, 2019), *SHELXT2014/5* (Sheldrick, 2015a), *SHELXL2018/3* (Sheldrick, 2015b), *Mercury* (Macrae *et al.*, 2020) and *OLEX2* (Dolomanov *et al.*, 2009).

except for C19 and O4 of the trifluoroacetic anion and twelve resulting atoms except for C17 of the trifluoroacetic acid. The four-atom C–COO fragments were restrained to be nearly planar by a FLAT command. Bond distances in the disordered fragments were restrained by the SAME command to be similar in length. Anisotropic displacement parameters were employed for the non-hydrogen atoms. Anisotropic displacement parameters for pairs of the disordered atoms were constrained to be the same. The H atom bound to O was found in difference-Fourier maps, C/N-bound H atoms were included in calculated positions and refined using a riding model with isotropic displacement parameters based on those of the parent atom [C–H = 0.93 Å, N/O–H = 0.86 Å, *U*_{iso}(H) = 1.2*U*_{eq}C for CH, NH and OH; C–H = 0.96 Å, *U*_{iso}(H) = 1.5*U*_{eq}C for CH₃]. Idealised methyl groups were refined as rotating groups.

Acknowledgements

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supporting information

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Crystal structure of bis{2-[5-(3,4,5-trimethoxyphenyl)-4*H*-1,2,4-triazol-3-yl]pyridine}palladium(II) bis(trifluoroacetate) trifluoroacetic acid disolvate

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Computing details

Bis{2-[5-(3,4,5-trimethoxyphenyl)-4*H*-1,2,4-triazol-3-yl]pyridine}palladium(II) bis(trifluoroacetate) trifluoroacetic acid disolvate

Crystal data

[Pd(C₁₆H₁₆N₄O₃)₂](C₂F₃O₂)₂·2C₂HF₃O₂

M_r = 1185.15

Triclinic, *P* $\bar{1}$

a = 8.6173 (4) Å

b = 10.6265 (6) Å

c = 13.1312 (4) Å

α = 93.384 (4)°

β = 98.121 (3)°

γ = 94.090 (4)°

V = 1184.46 (9) Å³

Z = 1

F(000) = 596

D_x = 1.662 Mg m⁻³

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 3996 reflections

θ = 1.6–28.8°

μ = 0.51 mm⁻¹

T = 293 K

Irregular, clear light yellow

0.4 × 0.3 × 0.3 mm

Data collection

Xcalibur, Eos

diffractometer

φ and ω scans

Absorption correction: multi-scan

(CrysAlisPro; Rigaku OD, 2019)

T_{min} = 0.968, *T_{max}* = 1.000

9462 measured reflections

5010 independent reflections

4615 reflections with *I* > 2σ(*I*)

R_{int} = 0.026

θ_{\max} = 26.7°, θ_{\min} = 1.9°

h = -10→10

k = -13→13

l = -16→16

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2σ(*F*²)] = 0.042

wR(*F*²) = 0.095

S = 1.04

5010 reflections

444 parameters

382 restraints

Primary atom site location: dual

Hydrogen site location: mixed

H-atom parameters constrained

w = 1/[σ²(*F_o*²) + (0.0395*P*)² + 0.5877*P*]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} < 0.001

Δρ_{max} = 0.39 e Å⁻³

Δρ_{min} = -0.45 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Pd1	1.000000	1.000000	0.500000	0.02885 (10)	
C19	0.8894 (4)	0.4302 (3)	0.1535 (2)	0.0488 (8)	
C20	0.9655 (9)	0.3470 (9)	0.0791 (6)	0.081 (2)	0.530 (6)
F4	1.1096 (6)	0.3858 (7)	0.0705 (5)	0.109 (2)	0.530 (6)
F5	0.9508 (14)	0.2294 (9)	0.1005 (8)	0.139 (3)	0.530 (6)
F6	0.8973 (9)	0.3527 (8)	−0.0170 (4)	0.108 (2)	0.530 (6)
O5	0.7452 (12)	0.425 (3)	0.140 (2)	0.066 (4)	0.530 (6)
C20X	0.9676 (14)	0.3301 (10)	0.0963 (10)	0.081 (2)	0.470 (6)
F4X	0.9952 (15)	0.3563 (10)	0.0080 (8)	0.134 (3)	0.470 (6)
F5X	1.0921 (8)	0.2898 (7)	0.1463 (6)	0.110 (3)	0.470 (6)
F6X	0.8690 (10)	0.2246 (8)	0.0722 (7)	0.107 (3)	0.470 (6)
O5X	0.7498 (15)	0.441 (3)	0.123 (3)	0.074 (6)	0.470 (6)
O4	0.9779 (3)	0.4896 (2)	0.22379 (16)	0.0580 (6)	
C17	0.4530 (4)	0.2377 (4)	0.0206 (3)	0.0643 (10)	
C18	0.3492 (9)	0.1637 (9)	−0.0678 (6)	0.095 (2)	0.587 (17)
F1	0.3053 (15)	0.2296 (12)	−0.1475 (7)	0.136 (3)	0.587 (17)
F2	0.4262 (13)	0.0721 (12)	−0.1043 (11)	0.143 (3)	0.587 (17)
F3	0.2162 (9)	0.1158 (12)	−0.0410 (7)	0.118 (3)	0.587 (17)
O7	0.442 (2)	0.2093 (17)	0.1046 (6)	0.100 (5)	0.587 (17)
O6	0.5404 (17)	0.3257 (15)	−0.0093 (13)	0.094 (5)	0.587 (17)
H6	0.613798	0.359574	0.035314	0.113*	0.587 (17)
C18X	0.3438 (13)	0.1690 (10)	−0.0676 (7)	0.101 (3)	0.413 (17)
F1X	0.4166 (17)	0.1293 (19)	−0.1438 (10)	0.125 (4)	0.413 (17)
F2X	0.2443 (16)	0.2475 (14)	−0.1086 (13)	0.132 (4)	0.413 (17)
F3X	0.278 (2)	0.0643 (15)	−0.0378 (12)	0.141 (4)	0.413 (17)
O7X	0.428 (3)	0.241 (2)	0.1062 (8)	0.078 (4)	0.413 (17)
O6X	0.574 (2)	0.288 (2)	−0.0120 (18)	0.081 (5)	0.413 (17)
H6X	0.646340	0.324285	0.033784	0.097*	0.413 (17)
O1	0.5903 (3)	0.3676 (2)	0.69590 (17)	0.0607 (7)	
O2	0.6094 (3)	0.15652 (19)	0.57418 (17)	0.0506 (5)	
O3	0.7086 (3)	0.16746 (19)	0.39566 (16)	0.0543 (6)	
N4	0.9103 (3)	0.6437 (2)	0.37512 (15)	0.0306 (5)	
H4	0.916011	0.589624	0.324689	0.037*	
N3	0.8561 (3)	0.7280 (2)	0.52237 (16)	0.0329 (5)	
N2	0.9308 (3)	0.81692 (19)	0.47212 (15)	0.0300 (5)	
N1	1.0785 (3)	0.9649 (2)	0.36266 (15)	0.0311 (5)	
C10	0.7119 (3)	0.2797 (3)	0.4517 (2)	0.0386 (6)	
C15	0.4529 (4)	0.1298 (4)	0.5844 (4)	0.0795 (13)	
H15A	0.387704	0.144900	0.521519	0.119*	

H15B	0.426681	0.183175	0.640017	0.119*
H15C	0.435939	0.042788	0.598890	0.119*
C7	0.8445 (3)	0.6231 (2)	0.46214 (19)	0.0303 (6)
C12	0.6508 (3)	0.3838 (3)	0.6071 (2)	0.0409 (7)
C14	0.7558 (5)	0.1699 (3)	0.2963 (2)	0.0638 (10)
H14A	0.687992	0.219861	0.253864	0.096*
H14B	0.749120	0.085221	0.265300	0.096*
H14C	0.862275	0.206258	0.302706	0.096*
C4	1.0995 (3)	0.8044 (3)	0.2315 (2)	0.0388 (7)
H4A	1.077270	0.721198	0.204347	0.047*
C9	0.7732 (3)	0.3943 (3)	0.4228 (2)	0.0365 (6)
H9	0.813638	0.398525	0.360914	0.044*
C2	1.2150 (4)	1.0120 (3)	0.2234 (2)	0.0497 (8)
H2	1.272046	1.071011	0.190832	0.060*
C13	0.7108 (3)	0.4976 (3)	0.5783 (2)	0.0382 (6)
H13	0.709298	0.571194	0.620054	0.046*
C1	1.1601 (4)	1.0467 (3)	0.3140 (2)	0.0403 (7)
H1	1.180841	1.129773	0.341708	0.048*
C3	1.1847 (4)	0.8902 (3)	0.1821 (2)	0.0492 (8)
H3	1.221162	0.865472	0.121268	0.059*
C5	1.0485 (3)	0.8437 (2)	0.32096 (18)	0.0293 (5)
C11	0.6520 (3)	0.2734 (3)	0.5440 (2)	0.0384 (6)
C8	0.7734 (3)	0.5023 (2)	0.4872 (2)	0.0322 (6)
C16	0.5794 (5)	0.4776 (4)	0.7613 (3)	0.0674 (11)
H16A	0.519452	0.536517	0.723050	0.101*
H16B	0.683090	0.516035	0.786165	0.101*
H16C	0.528455	0.454170	0.818594	0.101*
C6	0.9639 (3)	0.7659 (2)	0.38500 (18)	0.0292 (5)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd1	0.04096 (18)	0.01711 (15)	0.02989 (16)	-0.00198 (11)	0.01369 (12)	-0.00184 (10)
C19	0.064 (2)	0.0396 (18)	0.0431 (17)	-0.0077 (16)	0.0198 (16)	-0.0102 (14)
C20	0.110 (4)	0.077 (4)	0.058 (4)	0.005 (4)	0.035 (4)	-0.034 (4)
F4	0.074 (3)	0.144 (5)	0.105 (4)	0.008 (3)	0.032 (3)	-0.064 (4)
F5	0.189 (8)	0.086 (5)	0.153 (7)	0.043 (5)	0.057 (6)	-0.014 (5)
F6	0.111 (5)	0.153 (5)	0.052 (3)	-0.007 (4)	0.021 (3)	-0.057 (3)
O5	0.059 (5)	0.079 (9)	0.057 (6)	-0.014 (4)	0.013 (4)	-0.010 (5)
C20X	0.108 (5)	0.063 (5)	0.070 (5)	0.000 (4)	0.023 (4)	-0.028 (4)
F4X	0.187 (8)	0.130 (5)	0.101 (6)	0.022 (6)	0.079 (6)	-0.014 (5)
F5X	0.103 (4)	0.089 (5)	0.133 (5)	0.049 (4)	0.001 (4)	-0.046 (4)
F6X	0.124 (6)	0.067 (4)	0.115 (5)	-0.011 (4)	0.001 (4)	-0.058 (3)
O5X	0.076 (7)	0.072 (9)	0.073 (12)	-0.012 (5)	0.021 (5)	-0.015 (8)
O4	0.0708 (16)	0.0521 (15)	0.0483 (13)	-0.0024 (12)	0.0127 (11)	-0.0217 (11)
C17	0.053 (2)	0.075 (3)	0.063 (2)	-0.0078 (19)	0.0090 (18)	0.004 (2)
C18	0.079 (5)	0.117 (5)	0.083 (5)	-0.024 (4)	0.013 (4)	-0.019 (4)
F1	0.124 (6)	0.186 (7)	0.083 (4)	-0.025 (5)	-0.024 (4)	0.010 (5)

F2	0.151 (6)	0.131 (7)	0.132 (7)	-0.006 (5)	0.001 (5)	-0.058 (5)
F3	0.068 (4)	0.142 (7)	0.131 (4)	-0.045 (4)	0.004 (3)	-0.010 (5)
O7	0.099 (7)	0.123 (11)	0.075 (6)	-0.035 (7)	0.006 (5)	0.040 (6)
O6	0.097 (8)	0.116 (9)	0.059 (5)	-0.046 (7)	-0.002 (5)	0.022 (6)
C18X	0.086 (6)	0.122 (6)	0.088 (6)	-0.023 (6)	0.010 (5)	-0.019 (5)
F1X	0.125 (6)	0.151 (9)	0.089 (6)	-0.018 (7)	0.017 (5)	-0.042 (6)
F2X	0.087 (6)	0.176 (7)	0.115 (7)	0.012 (6)	-0.031 (5)	-0.022 (6)
F3X	0.132 (8)	0.129 (8)	0.147 (7)	-0.064 (6)	0.017 (7)	-0.008 (6)
O7X	0.099 (8)	0.076 (8)	0.065 (7)	-0.007 (6)	0.039 (6)	-0.012 (5)
O6X	0.062 (6)	0.116 (12)	0.057 (6)	-0.028 (7)	0.013 (5)	-0.013 (7)
O1	0.0919 (18)	0.0384 (13)	0.0582 (14)	-0.0097 (12)	0.0406 (13)	0.0027 (10)
O2	0.0538 (13)	0.0281 (11)	0.0716 (15)	-0.0050 (10)	0.0154 (11)	0.0121 (10)
O3	0.0882 (17)	0.0208 (11)	0.0522 (13)	-0.0098 (11)	0.0160 (11)	-0.0074 (9)
N4	0.0412 (13)	0.0206 (11)	0.0288 (11)	-0.0052 (9)	0.0084 (9)	-0.0058 (8)
N3	0.0463 (13)	0.0196 (11)	0.0345 (11)	-0.0037 (9)	0.0148 (10)	0.0010 (9)
N2	0.0410 (13)	0.0181 (11)	0.0323 (11)	-0.0024 (9)	0.0139 (9)	-0.0017 (8)
N1	0.0429 (13)	0.0206 (11)	0.0311 (11)	-0.0022 (9)	0.0131 (9)	-0.0012 (8)
C10	0.0488 (17)	0.0232 (14)	0.0419 (15)	-0.0044 (12)	0.0057 (13)	-0.0013 (11)
C15	0.054 (2)	0.042 (2)	0.143 (4)	-0.0146 (17)	0.026 (2)	0.006 (2)
C7	0.0371 (15)	0.0202 (13)	0.0332 (13)	-0.0012 (11)	0.0068 (11)	-0.0010 (10)
C12	0.0465 (17)	0.0322 (16)	0.0460 (16)	-0.0022 (13)	0.0162 (13)	0.0044 (13)
C14	0.105 (3)	0.0382 (19)	0.0460 (19)	-0.0013 (19)	0.0120 (18)	-0.0093 (15)
C4	0.0528 (18)	0.0307 (15)	0.0322 (14)	-0.0040 (13)	0.0113 (12)	-0.0064 (11)
C9	0.0494 (17)	0.0244 (14)	0.0351 (14)	-0.0046 (12)	0.0087 (12)	-0.0005 (11)
C2	0.071 (2)	0.0403 (18)	0.0423 (16)	-0.0079 (16)	0.0301 (15)	0.0003 (13)
C13	0.0506 (17)	0.0238 (14)	0.0407 (15)	-0.0027 (12)	0.0135 (13)	-0.0026 (11)
C1	0.0583 (19)	0.0241 (14)	0.0403 (15)	-0.0054 (13)	0.0197 (13)	-0.0011 (11)
C3	0.070 (2)	0.0453 (19)	0.0349 (15)	-0.0060 (16)	0.0272 (15)	-0.0070 (13)
C5	0.0341 (14)	0.0248 (13)	0.0289 (12)	0.0010 (11)	0.0055 (10)	0.0003 (10)
C11	0.0420 (16)	0.0239 (14)	0.0488 (16)	-0.0052 (12)	0.0067 (13)	0.0067 (12)
C8	0.0391 (15)	0.0216 (13)	0.0352 (14)	-0.0029 (11)	0.0061 (11)	0.0011 (10)
C16	0.093 (3)	0.057 (2)	0.059 (2)	0.001 (2)	0.040 (2)	-0.0033 (18)
C6	0.0369 (14)	0.0209 (13)	0.0291 (12)	-0.0016 (10)	0.0071 (10)	-0.0033 (10)

Geometric parameters (Å, °)

Pd1—N2	1.991 (2)	N4—H4	0.8600
Pd1—N2 ⁱ	1.991 (2)	N4—C7	1.367 (3)
Pd1—N1	2.037 (2)	N4—C6	1.340 (3)
Pd1—N1 ⁱ	2.037 (2)	N3—N2	1.360 (3)
C19—C20	1.526 (9)	N3—C7	1.317 (3)
C19—O5	1.227 (11)	N2—C6	1.313 (3)
C19—C20X	1.517 (11)	N1—C1	1.328 (3)
C19—O5X	1.231 (13)	N1—C5	1.362 (3)
C19—O4	1.223 (4)	C10—C9	1.388 (4)
C20—F4	1.303 (7)	C10—C11	1.385 (4)
C20—F5	1.298 (7)	C15—H15A	0.9600
C20—F6	1.321 (7)	C15—H15B	0.9600

C20X—F4X	1.258 (12)	C15—H15C	0.9600
C20X—F5X	1.290 (12)	C7—C8	1.458 (3)
C20X—F6X	1.351 (11)	C12—C13	1.376 (4)
C17—C18	1.504 (7)	C12—C11	1.397 (4)
C17—O7	1.177 (7)	C14—H14A	0.9600
C17—O6	1.273 (7)	C14—H14B	0.9600
C17—C18X	1.501 (8)	C14—H14C	0.9600
C17—O7X	1.174 (8)	C4—H4A	0.9300
C17—O6X	1.275 (8)	C4—C3	1.378 (4)
C18—F1	1.320 (6)	C4—C5	1.365 (3)
C18—F2	1.321 (6)	C9—H9	0.9300
C18—F3	1.323 (6)	C9—C8	1.384 (4)
O6—H6	0.8428	C2—H2	0.9300
C18X—F1X	1.319 (7)	C2—C1	1.380 (4)
C18X—F2X	1.321 (7)	C2—C3	1.366 (4)
C18X—F3X	1.319 (7)	C13—H13	0.9300
O6X—H6X	0.8555	C13—C8	1.383 (4)
O1—C12	1.357 (3)	C1—H1	0.9300
O1—C16	1.425 (4)	C3—H3	0.9300
O2—C15	1.385 (4)	C5—C6	1.446 (3)
O2—C11	1.367 (3)	C16—H16A	0.9600
O3—C10	1.360 (3)	C16—H16B	0.9600
O3—C14	1.421 (4)	C16—H16C	0.9600
N2—Pd1—N2 ⁱ	180.00 (12)	O3—C10—C9	124.2 (3)
N2—Pd1—N1 ⁱ	100.35 (8)	O3—C10—C11	115.4 (2)
N2 ⁱ —Pd1—N1	100.35 (8)	C11—C10—C9	120.4 (3)
N2 ⁱ —Pd1—N1 ⁱ	79.65 (8)	O2—C15—H15A	109.5
N2—Pd1—N1	79.65 (8)	O2—C15—H15B	109.5
N1 ⁱ —Pd1—N1	180.00 (12)	O2—C15—H15C	109.5
O5—C19—C20	116.4 (14)	H15A—C15—H15B	109.5
O5X—C19—C20X	116.7 (16)	H15A—C15—H15C	109.5
O4—C19—C20	116.6 (4)	H15B—C15—H15C	109.5
O4—C19—O5	126.9 (14)	N4—C7—C8	125.5 (2)
O4—C19—C20X	113.6 (6)	N3—C7—N4	110.5 (2)
O4—C19—O5X	129.6 (17)	N3—C7—C8	123.9 (2)
F4—C20—C19	114.3 (6)	O1—C12—C13	124.8 (3)
F4—C20—F6	100.3 (7)	O1—C12—C11	115.1 (3)
F5—C20—C19	110.6 (7)	C13—C12—C11	120.0 (2)
F5—C20—F4	112.8 (8)	O3—C14—H14A	109.5
F5—C20—F6	107.0 (9)	O3—C14—H14B	109.5
F6—C20—C19	111.1 (6)	O3—C14—H14C	109.5
F4X—C20X—C19	115.2 (10)	H14A—C14—H14B	109.5
F4X—C20X—F5X	108.9 (11)	H14A—C14—H14C	109.5
F4X—C20X—F6X	100.6 (11)	H14B—C14—H14C	109.5
F5X—C20X—C19	116.1 (9)	C3—C4—H4A	120.6
F5X—C20X—F6X	103.9 (9)	C5—C4—H4A	120.6
F6X—C20X—C19	110.5 (9)	C5—C4—C3	118.8 (3)

O7—C17—C18	117.9 (10)	C10—C9—H9	120.4
O7—C17—O6	129.6 (11)	C8—C9—C10	119.1 (2)
O6—C17—C18	112.5 (9)	C8—C9—H9	120.4
O7X—C17—C18X	123.2 (13)	C1—C2—H2	120.3
O7X—C17—O6X	126.8 (15)	C3—C2—H2	120.3
O6X—C17—C18X	110.0 (12)	C3—C2—C1	119.4 (3)
F1—C18—C17	114.7 (7)	C12—C13—H13	120.1
F1—C18—F2	105.2 (8)	C12—C13—C8	119.8 (3)
F1—C18—F3	104.8 (8)	C8—C13—H13	120.1
F2—C18—C17	109.5 (7)	N1—C1—C2	121.9 (3)
F2—C18—F3	109.9 (8)	N1—C1—H1	119.0
F3—C18—C17	112.4 (6)	C2—C1—H1	119.0
C17—O6—H6	116.1	C4—C3—H3	120.3
F1X—C18X—C17	113.1 (9)	C2—C3—C4	119.4 (3)
F1X—C18X—F2X	105.1 (10)	C2—C3—H3	120.3
F1X—C18X—F3X	103.9 (10)	N1—C5—C4	122.1 (2)
F2X—C18X—C17	109.2 (8)	N1—C5—C6	111.5 (2)
F3X—C18X—C17	110.4 (9)	C4—C5—C6	126.3 (2)
F3X—C18X—F2X	114.9 (12)	O2—C11—C10	117.9 (3)
C17—O6X—H6X	116.5	O2—C11—C12	122.1 (3)
C12—O1—C16	117.7 (2)	C10—C11—C12	119.6 (2)
C11—O2—C15	117.5 (3)	C9—C8—C7	120.7 (2)
C10—O3—C14	117.7 (2)	C13—C8—C7	118.3 (2)
C7—N4—H4	127.3	C13—C8—C9	120.9 (2)
C6—N4—H4	127.3	O1—C16—H16A	109.5
C6—N4—C7	105.5 (2)	O1—C16—H16B	109.5
C7—N3—N2	105.36 (19)	O1—C16—H16C	109.5
N3—N2—Pd1	135.44 (16)	H16A—C16—H16B	109.5
C6—N2—Pd1	114.75 (16)	H16A—C16—H16C	109.5
C6—N2—N3	109.8 (2)	H16B—C16—H16C	109.5
C1—N1—Pd1	126.23 (18)	N4—C6—C5	132.4 (2)
C1—N1—C5	118.4 (2)	N2—C6—N4	108.8 (2)
C5—N1—Pd1	115.29 (16)	N2—C6—C5	118.8 (2)
Pd1—N2—C6—N4	-179.45 (16)	N3—C7—C8—C9	-176.4 (3)
Pd1—N2—C6—C5	1.7 (3)	N3—C7—C8—C13	1.6 (4)
Pd1—N1—C1—C2	176.7 (2)	N2—N3—C7—N4	0.1 (3)
Pd1—N1—C5—C4	-177.2 (2)	N2—N3—C7—C8	179.1 (2)
Pd1—N1—C5—C6	0.6 (3)	N1—C5—C6—N4	179.9 (3)
O5—C19—C20—F4	156.2 (17)	N1—C5—C6—N2	-1.5 (3)
O5—C19—C20—F5	-75.1 (18)	C10—C9—C8—C7	176.6 (3)
O5—C19—C20—F6	43.6 (18)	C10—C9—C8—C13	-1.3 (4)
O5X—C19—C20X—F4X	73 (2)	C15—O2—C11—C10	-118.1 (4)
O5X—C19—C20X—F5X	-158 (2)	C15—O2—C11—C12	68.1 (4)
O5X—C19—C20X—F6X	-40 (2)	C7—N4—C6—N2	-0.8 (3)
O4—C19—C20—F4	-27.5 (8)	C7—N4—C6—C5	177.9 (3)
O4—C19—C20—F5	101.2 (8)	C7—N3—N2—Pd1	179.9 (2)
O4—C19—C20—F6	-140.1 (6)	C7—N3—N2—C6	-0.6 (3)

O4—C19—C20X—F4X	-106.0 (11)	C12—C13—C8—C7	-176.4 (3)
O4—C19—C20X—F5X	22.9 (12)	C12—C13—C8—C9	1.6 (4)
O4—C19—C20X—F6X	140.8 (9)	C14—O3—C10—C9	-6.8 (5)
O7—C17—C18—F1	147.3 (16)	C14—O3—C10—C11	174.6 (3)
O7—C17—C18—F2	-94.6 (16)	C4—C5—C6—N4	-2.4 (5)
O7—C17—C18—F3	27.8 (18)	C4—C5—C6—N2	176.2 (3)
O6—C17—C18—F1	-31.6 (16)	C9—C10—C11—O2	-172.8 (3)
O6—C17—C18—F2	86.4 (15)	C9—C10—C11—C12	1.2 (5)
O6—C17—C18—F3	-151.2 (15)	C13—C12—C11—O2	172.9 (3)
O7X—C17—C18X—F1X	-151.8 (19)	C13—C12—C11—C10	-0.8 (5)
O7X—C17—C18X—F2X	91.5 (19)	C1—N1—C5—C4	0.1 (4)
O7X—C17—C18X—F3X	-36 (2)	C1—N1—C5—C6	177.9 (2)
O6X—C17—C18X—F1X	27 (2)	C1—C2—C3—C4	0.2 (5)
O6X—C17—C18X—F2X	-89.8 (19)	C3—C4—C5—N1	0.2 (4)
O6X—C17—C18X—F3X	142.9 (19)	C3—C4—C5—C6	-177.2 (3)
O1—C12—C13—C8	178.3 (3)	C3—C2—C1—N1	0.1 (5)
O1—C12—C11—O2	-6.0 (4)	C5—N1—C1—C2	-0.3 (4)
O1—C12—C11—C10	-179.7 (3)	C5—C4—C3—C2	-0.3 (5)
O3—C10—C9—C8	-178.6 (3)	C11—C10—C9—C8	-0.1 (4)
O3—C10—C11—O2	5.8 (4)	C11—C12—C13—C8	-0.5 (5)
O3—C10—C11—C12	179.8 (3)	C16—O1—C12—C13	3.9 (5)
N4—C7—C8—C9	2.5 (4)	C16—O1—C12—C11	-177.2 (3)
N4—C7—C8—C13	-179.5 (3)	C6—N4—C7—N3	0.4 (3)
N3—N2—C6—N4	0.9 (3)	C6—N4—C7—C8	-178.5 (2)
N3—N2—C6—C5	-178.0 (2)		

Symmetry code: (i) $-x+2, -y+2, -z+1$.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O6—H6 \cdots F6	0.84	2.63	3.085 (15)	115
O6—H6 \cdots O5	0.84	1.73	2.56 (3)	172
O6X—H6X \cdots F6X	0.86	2.27	2.77 (3)	118
O6X—H6X \cdots O5X	0.86	1.76	2.58 (4)	160
N4—H4 \cdots O4	0.86	1.81	2.655 (3)	166
C15—H15A \cdots N3 ⁱⁱ	0.96	2.59	3.334 (5)	134
C15—H15B \cdots O1	0.96	2.35	2.929 (5)	118
C15—H15C \cdots O3 ⁱⁱⁱ	0.96	2.49	3.400 (4)	158
C14—H14A \cdots O7	0.96	2.67	3.492 (17)	144
C4—H4A \cdots F6 ^{iv}	0.93	2.58	3.193 (6)	124
C4—H4A \cdots O4	0.93	2.59	3.425 (4)	150
C9—H9 \cdots O4	0.93	2.63	3.510 (4)	158
C2—H2 \cdots O7 ^v	0.93	2.44	3.363 (14)	174
C2—H2 \cdots O7X ^v	0.93	2.57	3.498 (19)	178
C1—H1 \cdots N3 ⁱ	0.93	2.34	3.146 (3)	145
C16—H16B \cdots F4 ^{vi}	0.96	2.52	3.400 (7)	152

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