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2-[(2-Fluorophenyl)(1*H*-indol-3-yl)methyl]-1*H*-indole

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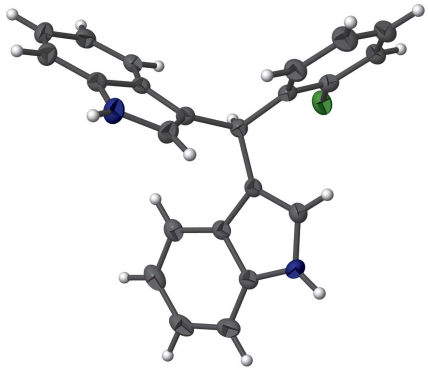
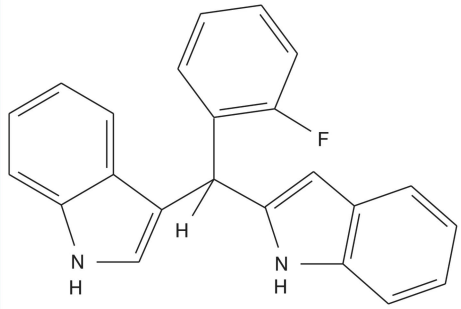
In the title compound, $C_{23}H_{17}FN_2$, the indole ring systems are oriented orthogonally, as indicated by the dihedral angle between them of 88.49 (5)°. The fluorophenyl ring is nearly perpendicular to one of the indole ring systems [dihedral angle = 85.31 (16)°] and twisted by 63.6 (6)° from the other. In the crystal, N—H···π, C—H···π and C—H···F interactions occur, forming a three-dimensional network.

3D view



Chemical scheme



Structure description

Indoles have diverse biological activities and pharmaceutical applications (Diss *et al.*, 2013). In particular, bis(indolyl) methane and its derivatives are known to be important intermediates in organic synthesis and exhibit various physiological properties (Ishikawa *et al.*, 2008). As part of our ongoing research in this area (Walki *et al.*, 2015), the synthesis and crystal structure of the title compound, $C_{23}H_{17}N_2F$, is reported herein (Fig. 1).

Both the indole ring systems are essentially planar with dihedral angles of 1.37 (1) and 2.48 (1)°, between the two fused ring systems N1/C2–C9 and N13/C11/C12/C14–C19, respectively. The indole ring systems are oriented orthogonally with respect to each other, as indicated by the dihedral angle value of 88.49 (5)°. The fluorophenyl ring is nearly perpendicular to one of the indole ring systems [N1/C2–C9; dihedral angle = 85.31 (16)°] whereas it is twisted by 63.6 (6)° from the other indole ring system.

In the crystal, the C5—H5···F26 hydrogen bond (Table 1) links molecules into $R_2^2(18)$ inversion dimers. C23—H23···π chains connect these dimers, forming rectangular columns running down the *b* axis, and these chains are further reinforced by C24—H24···π interactions (Fig. 2 left). Further N1—H1···π and C3—H3···π interactions

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

*Cg*1, *Cg*2, *Cg*3 and *Cg*4 are the centroids of the N1/C2/C7–C9, C2–C7, C14–C19 and C20–C25 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···F26^{i}	0.93	2.51	3.3849 (18)	157
N1—H1···*Cg*4^{ii}	0.86	2.63	3.4390 (14)	157
N13—H13···*Cg*3^{iii}	0.86	2.30	3.1539 (14)	170
C3—H3···*Cg*5^{ii}	0.93	2.96	3.7630 (19)	146
C23—H23···*Cg*4^{iv}	0.93	2.68	3.5022 (17)	147
C24—H24···*Cg*1^{v}	0.93	2.93	3.8376 (17)	166

Symmetry codes: (i) $-x, -y+2, -z+1$; (ii) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (iii) $-x+1, -y+2, -z+1$; (iv) $x, y-1, z$; (v) $-x, -y+1, -z+1$.

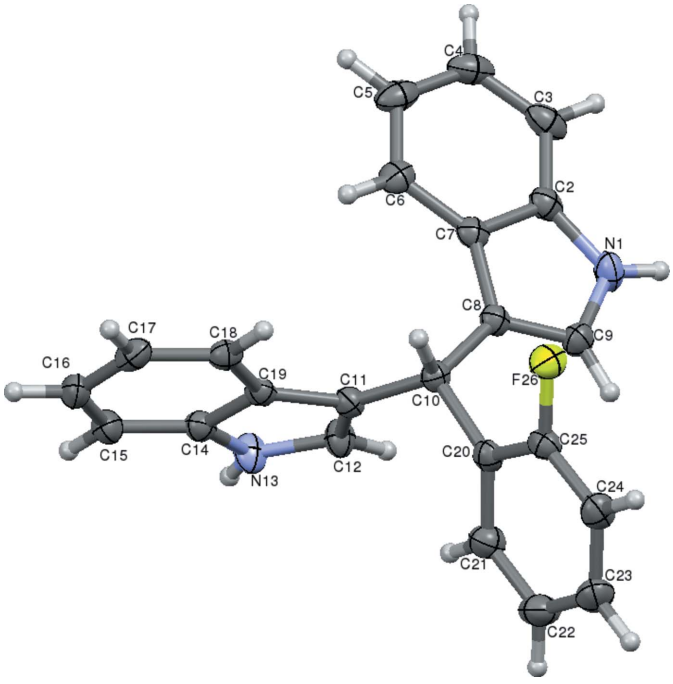


Figure 1
View of the title compound, with displacement ellipsoids for non-H atoms drawn at the 50% probability level.

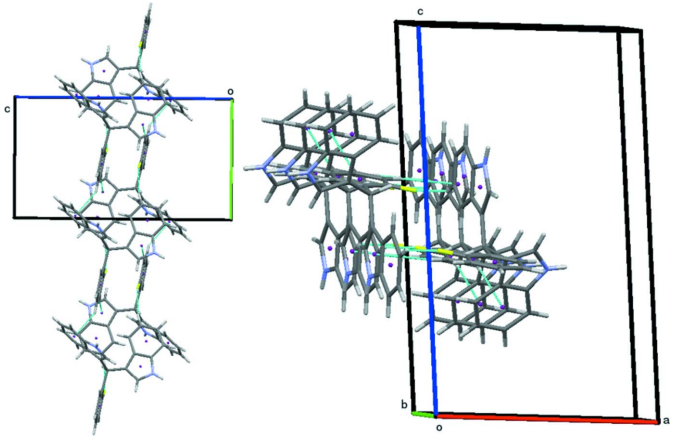


Figure 2
The molecular packing showing $R_2^2(18)$ dimers (left) connected by C—H···π interactions forming rectangular columns (right). Hydrogen bonds are shown as blue lines.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{23}H_{17}FN_2$
M_r	340.39
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, b, c (Å)	10.0822 (5), 9.7969 (4), 17.6310 (8)
β (°)	94.262 (2)
V (Å^3)	1736.67 (14)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.67
Crystal size (mm)	0.29 × 0.27 × 0.25
Data collection	
Diffractometer	Bruker X8 Proteum
Absorption correction	Multi-scan (*SADABS*; Bruker, 2013)
T_{min}, T_{max}	0.823, 0.845
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	7737, 2845, 2480
R_{int}	0.036
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.584
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.038, 0.105, 1.07
No. of reflections	2845
No. of parameters	235
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.23, −0.18

Computer programs: *APEX2* and *SAINT* (Bruker, 2013), *SHELXS97* and *SHELXL97* (Sheldrick, 2008), *Mercury* (Macrae *et al.*, 2008) and *PLATON* (Spek, 2009).

between the molecules results in sheets parallel to the *bc* plane (Fig. 2 right). The sheets are interlinked *via* N13—H13···π interactions along the *a* axis, forming a three-dimensional network (Fig. 3).

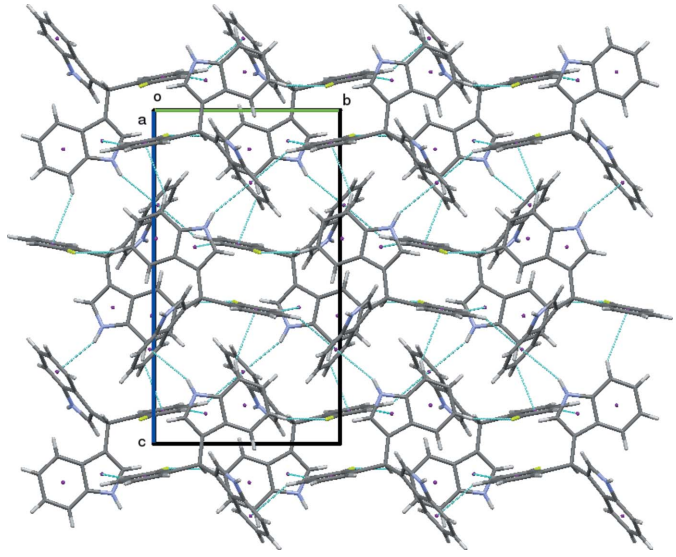


Figure 3
Packing of the molecules viewed along the *a* axis.

Synthesis and crystallization

To a flask containing 5 ml of glacial acetic acid, indole (2 mmol, 0.23 g) was added under stirring until all the indole had dissolved. Then 2-fluorobenzaldehyde (1 mmol, 0.14 g) was added under vigorous stirring. The reaction mixture was allowed to stir over 4 to 6 h, during which time the reaction solution turned from light yellow to light pink to dark red colour. The product was detected by TLC (100% CH_2Cl_2). After the completion of the reaction, the reaction mixture was added to ice-cold water. The product that separated out from the reaction mixture was filtered and washed with water. The crude product was further purified by recrystallization using methanol as solvent to obtain orange blocks of the title compound in 81% yield, m.p. = 178–180°C.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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full crystallographic data

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Crystal data

$C_{23}H_{17}FN_2$
M_r = 340.39
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 10.0822 (5) Å
b = 9.7969 (4) Å
c = 17.6310 (8) Å
β = 94.262 (2)°
V = 1736.67 (14) Å^3
Z = 4
$F(000)$ = 712
D_x = 1.302 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 2845 reflections
θ = 4.4–64.2°
μ = 0.67 mm^{-1}
T = 296 K
Block, orange
0.29 × 0.27 × 0.25 mm

Data collection

Bruker X8 Proteum diffractometer
Radiation source: Rotating Anode
Graphite monochromator
Detector resolution: 18.4 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2013)
T_{min} = 0.823, T_{max} = 0.845
7737 measured reflections
2845 independent reflections
2480 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 64.2°, θ_{min} = 4.4°
h = −11→11
k = −10→11
l = −20→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.105
S = 1.07
2845 reflections
235 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0587P)^2 + 0.3295P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Special details

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R-factors wR and all goodnesses 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 observed criterion of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating -R-factor-obs 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
F26	0.00536 (8)	0.57139 (8)	0.42643 (5)	0.0316 (3)
N1	0.24195 (13)	0.72377 (13)	0.64897 (7)	0.0302 (4)
N13	0.52338 (12)	0.93784 (13)	0.38606 (7)	0.0292 (4)
C2	0.20504 (14)	0.85811 (15)	0.64054 (9)	0.0260 (4)
C3	0.18320 (15)	0.95771 (17)	0.69494 (10)	0.0334 (5)
C4	0.15144 (15)	1.08692 (17)	0.66901 (11)	0.0358 (5)
C5	0.14059 (15)	1.11795 (16)	0.59132 (11)	0.0343 (5)
C6	0.16105 (14)	1.01950 (15)	0.53726 (9)	0.0271 (4)
C7	0.19387 (13)	0.88639 (14)	0.56189 (8)	0.0227 (4)
C8	0.22413 (13)	0.76180 (14)	0.52312 (8)	0.0218 (4)
C9	0.25164 (15)	0.66702 (15)	0.57843 (9)	0.0270 (4)
C10	0.22554 (14)	0.74567 (14)	0.43815 (8)	0.0220 (4)
C11	0.33021 (14)	0.83498 (14)	0.40622 (8)	0.0228 (4)
C12	0.46077 (14)	0.84799 (15)	0.43141 (9)	0.0276 (5)
C14	0.43318 (14)	0.98676 (14)	0.33069 (8)	0.0242 (4)
C15	0.44803 (16)	1.08041 (15)	0.27228 (9)	0.0295 (5)
C16	0.33645 (16)	1.11427 (15)	0.22625 (9)	0.0304 (5)
C17	0.21197 (16)	1.05838 (15)	0.23816 (9)	0.0294 (4)
C18	0.19700 (14)	0.96412 (14)	0.29521 (8)	0.0244 (4)
C19	0.30918 (14)	0.92501 (14)	0.34225 (8)	0.0215 (4)
C20	0.23638 (14)	0.59460 (14)	0.41749 (8)	0.0232 (4)
C21	0.35517 (16)	0.52949 (16)	0.40449 (9)	0.0310 (5)
C22	0.35997 (17)	0.39047 (17)	0.38958 (10)	0.0369 (5)
C23	0.24583 (17)	0.31272 (16)	0.38742 (10)	0.0349 (5)
C24	0.12536 (16)	0.37375 (16)	0.39988 (9)	0.0300 (5)
C25	0.12406 (14)	0.51209 (15)	0.41452 (8)	0.0246 (4)
H1	0.25670	0.68190	0.69160	0.0360*
H3	0.18990	0.93750	0.74660	0.0400*
H4	0.13680	1.15540	0.70400	0.0430*
H5	0.11920	1.20650	0.57580	0.0410*
H6	0.15330	1.04080	0.48570	0.0330*
H9	0.27390	0.57650	0.56970	0.0320*
H10	0.13910	0.77760	0.41590	0.0260*
H12	0.50150	0.80260	0.47320	0.0330*
H13	0.60610	0.95990	0.39160	0.0350*

H15	0.53040	1.11870	0.26470	0.0350*
H16	0.34400	1.17550	0.18650	0.0360*
H17	0.13790	1.08520	0.20710	0.0350*
H18	0.11390	0.92710	0.30240	0.0290*
H21	0.43320	0.58030	0.40580	0.0370*
H22	0.44070	0.34960	0.38100	0.0440*
H23	0.24960	0.21960	0.37760	0.0420*
H24	0.04740	0.32280	0.39840	0.0360*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F26	0.0230 (4)	0.0280 (4)	0.0438 (6)	−0.0020 (3)	0.0034 (4)	−0.0013 (4)
N1	0.0418 (8)	0.0289 (7)	0.0201 (7)	−0.0057 (5)	0.0031 (6)	0.0031 (5)
N13	0.0192 (6)	0.0338 (7)	0.0345 (8)	−0.0060 (5)	0.0005 (5)	0.0060 (6)
C2	0.0235 (7)	0.0278 (7)	0.0273 (8)	−0.0067 (6)	0.0059 (6)	−0.0036 (6)
C3	0.0283 (8)	0.0424 (9)	0.0307 (9)	−0.0117 (7)	0.0099 (7)	−0.0111 (7)
C4	0.0244 (8)	0.0356 (9)	0.0487 (11)	−0.0057 (7)	0.0109 (7)	−0.0194 (8)
C5	0.0216 (7)	0.0265 (8)	0.0550 (11)	0.0008 (6)	0.0036 (7)	−0.0081 (7)
C6	0.0190 (7)	0.0257 (7)	0.0363 (9)	−0.0011 (6)	0.0000 (6)	−0.0012 (7)
C7	0.0170 (7)	0.0246 (7)	0.0266 (8)	−0.0040 (5)	0.0029 (6)	−0.0025 (6)
C8	0.0196 (7)	0.0220 (7)	0.0241 (8)	−0.0039 (5)	0.0031 (6)	−0.0015 (6)
C9	0.0322 (8)	0.0218 (7)	0.0273 (8)	−0.0025 (6)	0.0045 (6)	−0.0006 (6)
C10	0.0213 (7)	0.0220 (7)	0.0226 (8)	−0.0016 (5)	0.0013 (6)	−0.0002 (6)
C11	0.0232 (7)	0.0237 (7)	0.0216 (7)	−0.0018 (6)	0.0031 (6)	−0.0010 (6)
C12	0.0261 (8)	0.0305 (8)	0.0258 (8)	−0.0027 (6)	−0.0011 (6)	0.0053 (6)
C14	0.0247 (7)	0.0240 (7)	0.0243 (8)	0.0000 (6)	0.0039 (6)	−0.0020 (6)
C15	0.0298 (8)	0.0270 (8)	0.0329 (9)	−0.0012 (6)	0.0098 (7)	0.0015 (7)
C16	0.0405 (9)	0.0243 (7)	0.0270 (8)	0.0026 (6)	0.0076 (7)	0.0046 (6)
C17	0.0355 (8)	0.0238 (7)	0.0279 (8)	0.0057 (6)	−0.0035 (7)	−0.0013 (6)
C18	0.0251 (7)	0.0221 (7)	0.0258 (8)	−0.0002 (6)	0.0013 (6)	−0.0039 (6)
C19	0.0235 (7)	0.0205 (7)	0.0210 (7)	−0.0006 (5)	0.0044 (6)	−0.0045 (6)
C20	0.0273 (8)	0.0246 (7)	0.0177 (7)	−0.0019 (6)	0.0027 (6)	−0.0009 (6)
C21	0.0290 (8)	0.0302 (8)	0.0344 (9)	−0.0023 (6)	0.0070 (7)	−0.0048 (7)
C22	0.0353 (9)	0.0331 (8)	0.0435 (10)	0.0045 (7)	0.0114 (8)	−0.0071 (7)
C23	0.0456 (10)	0.0256 (8)	0.0342 (9)	−0.0003 (7)	0.0085 (7)	−0.0066 (7)
C24	0.0341 (8)	0.0263 (8)	0.0299 (8)	−0.0072 (6)	0.0037 (7)	−0.0046 (6)
C25	0.0239 (7)	0.0285 (8)	0.0215 (8)	0.0008 (6)	0.0024 (6)	−0.0009 (6)

Geometric parameters (Å, º)

F26—C25	1.3606 (16)	C17—C18	1.382 (2)
N1—C2	1.3728 (19)	C18—C19	1.405 (2)
N1—C9	1.372 (2)	C20—C25	1.389 (2)
N13—C12	1.3739 (19)	C20—C21	1.391 (2)
N13—C14	1.3703 (19)	C21—C22	1.389 (2)
N1—H1	0.8600	C22—C23	1.378 (2)
N13—H13	0.8600	C23—C24	1.386 (2)

C2—C3	1.397 (2)	C24—C25	1.380 (2)
C2—C7	1.410 (2)	C3—H3	0.9300
C3—C4	1.376 (2)	C4—H4	0.9300
C4—C5	1.399 (3)	C5—H5	0.9300
C5—C6	1.382 (2)	C6—H6	0.9300
C6—C7	1.406 (2)	C9—H9	0.9300
C7—C8	1.4426 (19)	C10—H10	0.9800
C8—C9	1.360 (2)	C12—H12	0.9300
C8—C10	1.508 (2)	C15—H15	0.9300
C10—C11	1.511 (2)	C16—H16	0.9300
C10—C20	1.5300 (19)	C17—H17	0.9300
C11—C12	1.364 (2)	C18—H18	0.9300
C11—C19	1.435 (2)	C21—H21	0.9300
C14—C19	1.417 (2)	C22—H22	0.9300
C14—C15	1.396 (2)	C23—H23	0.9300
C15—C16	1.378 (2)	C24—H24	0.9300
C16—C17	1.399 (2)		

C2—N1—C9	109.10 (12)	C10—C20—C21	124.09 (13)
C12—N13—C14	109.23 (12)	C20—C21—C22	121.57 (15)
C2—N1—H1	125.00	C21—C22—C23	120.43 (16)
C9—N1—H1	125.00	C22—C23—C24	119.80 (15)
C12—N13—H13	125.00	C23—C24—C25	118.29 (14)
C14—N13—H13	125.00	F26—C25—C24	117.97 (13)
N1—C2—C7	107.22 (13)	C20—C25—C24	124.02 (14)
C3—C2—C7	122.18 (14)	F26—C25—C20	118.01 (12)
N1—C2—C3	130.58 (15)	C2—C3—H3	121.00
C2—C3—C4	117.34 (16)	C4—C3—H3	121.00
C3—C4—C5	121.64 (16)	C3—C4—H4	119.00
C4—C5—C6	121.24 (15)	C5—C4—H4	119.00
C5—C6—C7	118.55 (15)	C4—C5—H5	119.00
C2—C7—C8	107.18 (12)	C6—C5—H5	119.00
C6—C7—C8	133.75 (13)	C5—C6—H6	121.00
C2—C7—C6	119.05 (13)	C7—C6—H6	121.00
C9—C8—C10	128.64 (13)	N1—C9—H9	125.00
C7—C8—C9	106.08 (13)	C8—C9—H9	125.00
C7—C8—C10	125.28 (12)	C8—C10—H10	107.00
N1—C9—C8	110.41 (13)	C11—C10—H10	107.00
C8—C10—C20	110.13 (11)	C20—C10—H10	107.00
C11—C10—C20	114.02 (12)	N13—C12—H12	125.00
C8—C10—C11	111.58 (11)	C11—C12—H12	125.00
C10—C11—C12	128.07 (13)	C14—C15—H15	121.00
C10—C11—C19	125.60 (12)	C16—C15—H15	121.00
C12—C11—C19	106.33 (13)	C15—C16—H16	119.00
N13—C12—C11	110.15 (13)	C17—C16—H16	119.00
N13—C14—C15	130.82 (14)	C16—C17—H17	119.00
C15—C14—C19	122.07 (13)	C18—C17—H17	119.00
N13—C14—C19	107.10 (12)	C17—C18—H18	121.00

C14—C15—C16	117.73 (14)	C19—C18—H18	120.00
C15—C16—C17	121.30 (14)	C20—C21—H21	119.00
C16—C17—C18	121.22 (14)	C22—C21—H21	119.00
C17—C18—C19	119.02 (13)	C21—C22—H22	120.00
C14—C19—C18	118.60 (13)	C23—C22—H22	120.00
C11—C19—C14	107.16 (12)	C22—C23—H23	120.00
C11—C19—C18	134.18 (13)	C24—C23—H23	120.00
C10—C20—C25	119.96 (12)	C23—C24—H24	121.00
C21—C20—C25	115.88 (13)	C25—C24—H24	121.00

C9—N1—C2—C3	−179.64 (15)	C8—C10—C20—C25	80.00 (16)
C9—N1—C2—C7	−1.13 (16)	C11—C10—C20—C21	29.6 (2)
C2—N1—C9—C8	1.08 (17)	C11—C10—C20—C25	−153.68 (13)
C14—N13—C12—C11	0.86 (17)	C10—C11—C12—N13	178.93 (13)
C12—N13—C14—C15	179.23 (15)	C19—C11—C12—N13	−1.51 (17)
C12—N13—C14—C19	0.18 (16)	C10—C11—C19—C14	−178.85 (13)
N1—C2—C3—C4	177.56 (15)	C10—C11—C19—C18	4.2 (3)
C7—C2—C3—C4	−0.8 (2)	C12—C11—C19—C14	1.58 (16)
N1—C2—C7—C6	−177.98 (12)	C12—C11—C19—C18	−175.37 (16)
N1—C2—C7—C8	0.77 (15)	N13—C14—C15—C16	−177.53 (15)
C3—C2—C7—C6	0.7 (2)	C19—C14—C15—C16	1.4 (2)
C3—C2—C7—C8	179.44 (13)	N13—C14—C19—C11	−1.08 (15)
C2—C3—C4—C5	0.3 (2)	N13—C14—C19—C18	176.43 (12)
C3—C4—C5—C6	0.2 (2)	C15—C14—C19—C11	179.76 (13)
C4—C5—C6—C7	−0.3 (2)	C15—C14—C19—C18	−2.7 (2)
C5—C6—C7—C2	−0.2 (2)	C14—C15—C16—C17	0.9 (2)
C5—C6—C7—C8	−178.49 (15)	C15—C16—C17—C18	−1.9 (2)
C2—C7—C8—C9	−0.14 (15)	C16—C17—C18—C19	0.5 (2)
C2—C7—C8—C10	−179.35 (13)	C17—C18—C19—C11	178.41 (15)
C6—C7—C8—C9	178.35 (15)	C17—C18—C19—C14	1.7 (2)
C6—C7—C8—C10	−0.9 (2)	C10—C20—C21—C22	176.78 (15)
C7—C8—C9—N1	−0.56 (16)	C25—C20—C21—C22	−0.1 (2)
C10—C8—C9—N1	178.62 (13)	C10—C20—C25—F26	3.4 (2)
C7—C8—C10—C11	62.75 (17)	C10—C20—C25—C24	−176.90 (14)
C7—C8—C10—C20	−169.58 (12)	C21—C20—C25—F26	−179.62 (13)
C9—C8—C10—C11	−116.29 (16)	C21—C20—C25—C24	0.1 (2)
C9—C8—C10—C20	11.4 (2)	C20—C21—C22—C23	−0.1 (3)
C8—C10—C11—C12	49.0 (2)	C21—C22—C23—C24	0.3 (3)
C8—C10—C11—C19	−130.47 (14)	C22—C23—C24—C25	−0.3 (2)
C20—C10—C11—C12	−76.54 (19)	C23—C24—C25—F26	179.77 (14)
C20—C10—C11—C19	103.98 (16)	C23—C24—C25—C20	0.1 (2)
C8—C10—C20—C21	−96.71 (16)		

Hydrogen-bond geometry (Å, º)

*Cg*1, *Cg*2, *Cg*3 and *Cg*4 are the centroids of the N1/C2/C7–C9, C2–C7, C14–C19 and C20–C25 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···F26^{i}	0.93	2.51	3.3849 (18)	157

N1—H1···*Cg*4^{ii}	0.86	2.63	3.4390 (14)	157
N13—H13···*Cg*3^{iii}	0.86	2.30	3.1539 (14)	170
C3—H3···*Cg*5^{ii}	0.93	2.96	3.7630 (19)	146
C23—H23···*Cg*4^{iv}	0.93	2.68	3.5022 (17)	147
C24—H24···*Cg*1^{v}	0.93	2.93	3.8376 (17)	166

Symmetry codes: (i) $-x$, $-y+2$, $-z+1$; (ii) x, $-y+3/2$, $z+1/2$; (iii) $-x+1$, $-y+2$, $-z+1$; (iv) x, $y-1$, z; (v) $-x$, $-y+1$, $-z+1$.