IUCrData

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5-Fluoro-3-(1*H*-indol-3-ylmethyl)-1*H*-indole

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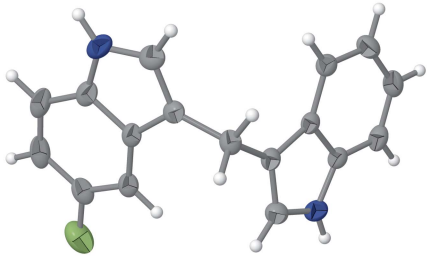
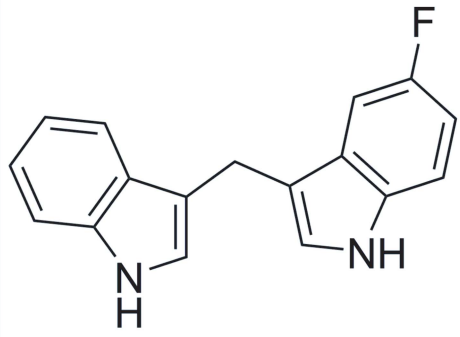
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{17}H_{13}FN_2$, was synthesized as a potential ligand for the construction of metal–organic frameworks. The two indole motifs present two potential coordination modes. It crystallizes in the orthorhombic system with space group $P2_12_12_1$. The dihedral angle between the fused ring systems is 68.77 (10)°. Weak F···H interactions are observed in the crystal.

3D view



Chemical scheme



Structure description

The diarylmethane motif is ubiquitous in natural products, bioactive molecule and medicine (Sakurai *et al.*, 2020). The title compound belongs to the diarylmethane family (Safe *et al.*, 2008), which shows antioxidant, anti-inflammatory and anticancer bioactivities, and can be found in broccoli.

In the present work, we synthesized the title compound as a potential ligand for the construction of metal–organic frameworks. This ligand is expected to be a good candidate for the construction of coordination polymers with diverse structures. The molecular structure is shown in Fig. 1. The title compound crystallizes in the orthorhombic system, space group $P2_12_12_1$. The dihedral angle between the fused ring systems is 68.77 (10)°. Fig. 2 shows the weak F···H and other interactions observed in the crystal Numerical details are given in Table 1.

Synthesis and crystallization

To a dried reaction tube (10 ml) with a magnetic stirring bar were added NHPI (*N*-hydroxyphthalimide ester) ester (0.2 mmol), indole (0.4 mmol) and $Ru(bpy)_2(PF_6)_2$ (3 mol %) successively. Air was then withdrawn and the tube was backfilled with argon three times. Subsequently, degassed DCM (2 ml) was injected into the tube by syringe. Then, the resulting reaction mixture was irradiated at room temperature under blue LEDs (6 W) for 12 h. The reaction progress was monitored by TLC. After the reaction

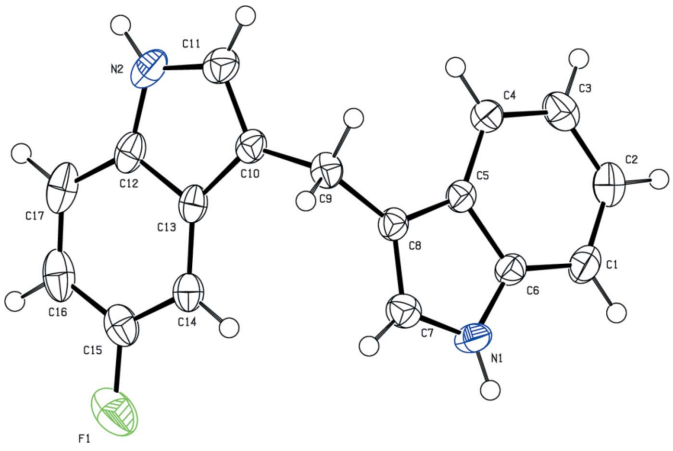


Figure 1
The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

was completed, the reaction mixture was concentrated under reduced pressure, and the residue was purified by column chromatography to afford the title compound. Single crystals of $C_{17}H_{13}FN_2$ were obtained by evaporation after one week. Yield: 47 wt%.

Refinement

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

Funding information

We thank the Doctoral Funding Project of Longdong University (XYBYZK2224) for financially supporting this work. We also thank the Youth Foundation of Gansu Province (21JR7RM196) for financial support.

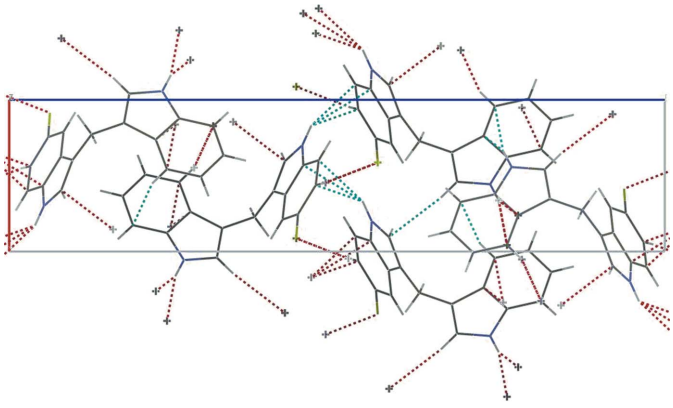


Figure 2
The crystal packing of the title compound. Weak interactions between the molecules are shown as dashed lines.

Table 1
Intermolecular interactions (Å).

Atom1	Atom2	Symm. op. 2	Length	Length − vdW
F1	H16	$-\frac{1}{2}+x, -\frac{1}{2}-y, -z$	2.48	−0.19
C1	H4	$1-x, -\frac{1}{2}+y, \frac{1}{2}-z$	2.88	−0.02
C4	H1	$-x, \frac{1}{2}+y, \frac{1}{2}-z$	2.55	−0.35
C5	H1	$-x, \frac{1}{2}+y, \frac{1}{2}-z$	2.58	−0.32
C11	H7	$1+x, y, z$	2.82	−0.08
C12	H2	$-\frac{1}{2}+x, \frac{1}{2}-y, -z$	2.80	−0.10
C16	H2	$-\frac{1}{2}+x, \frac{1}{2}-y, -z$	2.81	−0.09
C17	H2	$-\frac{1}{2}+x, \frac{1}{2}-y, -z$	2.64	−0.26

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{17}H_{13}FN_2$
M_r	264.29
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	296
a, b, c (Å)	6.0723 (3), 7.8662 (3), 26.2693 (11)
V (Å^3)	1254.78 (9)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.12 × 0.1 × 0.1
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.691, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	12005, 2866, 2528
R_{int}	0.063
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.649
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.047, 0.125, 1.06
No. of reflections	2866
No. of parameters	181
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.53, −0.30
Absolute structure	Flack x determined using 921 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	−0.1 (7)

Computer programs: *APEX2* and *SAINT* (Bruker, 2019), *SHELXT2018/2* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

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full crystallographic data

IUCrData (2023). **8**, x230590 [https://doi.org/10.1107/S2414314623005904]

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5-Fluoro-3-(1*H*-indol-3-ylmethyl)-1*H*-indole

Crystal data

$C_{17}H_{13}FN_2$
M_r = 264.29
Orthorhombic, $P2_12_12_1$
a = 6.0723 (3) Å
b = 7.8662 (3) Å
c = 26.2693 (11) Å
V = 1254.78 (9) Å^3
Z = 4
F(000) = 552
D_x = 1.399 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4629 reflections
θ = 2.7–27.4°
μ = 0.09 mm^{-1}
T = 296 K
Block, colourless
0.12 × 0.1 × 0.1 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.691, T_{max} = 0.746
12005 measured reflections
2866 independent reflections
2528 reflections with $I > 2\sigma(I)$
R_{int} = 0.063
θ_{max} = 27.5°, θ_{min} = 2.7°
h = −7→7
k = −9→10
l = −34→29

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.125
S = 1.06
2866 reflections
181 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.051P)^2 + 0.4902P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}
Absolute structure: Flack x determined using 921 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: −0.1 (7)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. The H atoms were included in calculated positions and treated as riding atoms: C–H = 0.93- 0.98 Å, with U_{iso}(H) = 1.2Ueq (C), U_{iso}(H) = 1.2Ueq (N).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
F1	0.0864 (4)	−0.1701 (2)	0.06215 (8)	0.0567 (6)
N1	−0.0566 (4)	0.2923 (3)	0.23358 (10)	0.0325 (6)
H1	−0.163457	0.235536	0.246712	0.039*
N2	0.7008 (4)	0.3284 (3)	0.05286 (9)	0.0366 (6)
H2	0.829218	0.336765	0.039262	0.044*
C6	0.1230 (5)	0.3505 (3)	0.25954 (11)	0.0268 (6)
C5	0.2620 (5)	0.4322 (3)	0.22362 (10)	0.0240 (6)
C8	0.1535 (5)	0.4238 (3)	0.17516 (10)	0.0255 (6)
C14	0.2106 (5)	0.1028 (3)	0.08297 (10)	0.0289 (6)
H14	0.081609	0.123845	0.100979	0.035*
C13	0.3747 (5)	0.2253 (3)	0.07888 (10)	0.0264 (6)
C10	0.3965 (5)	0.3978 (3)	0.09673 (10)	0.0264 (6)
C9	0.2277 (5)	0.4995 (3)	0.12576 (11)	0.0285 (6)
H9A	0.288541	0.611254	0.132574	0.034*
H9B	0.099716	0.514911	0.104165	0.034*
C4	0.4575 (5)	0.5076 (4)	0.24071 (11)	0.0293 (6)
H4	0.552343	0.561101	0.217991	0.035*
C12	0.5690 (5)	0.1875 (4)	0.05142 (10)	0.0316 (6)
C17	0.6017 (6)	0.0300 (4)	0.02794 (11)	0.0394 (8)
H17	0.730172	0.006624	0.009970	0.047*
C7	−0.0405 (5)	0.3383 (4)	0.18325 (11)	0.0312 (6)
H7	−0.145343	0.315036	0.158400	0.037*
C3	0.5069 (6)	0.5007 (4)	0.29232 (12)	0.0371 (7)
H3	0.636564	0.549724	0.304113	0.045*
C11	0.5953 (5)	0.4543 (4)	0.07946 (11)	0.0334 (7)
H11	0.651263	0.562800	0.084954	0.040*
C15	0.2462 (6)	−0.0498 (4)	0.05952 (11)	0.0345 (7)
C1	0.1734 (6)	0.3440 (4)	0.31123 (11)	0.0344 (7)
H1A	0.081012	0.289653	0.334300	0.041*
C16	0.4374 (7)	−0.0887 (4)	0.03240 (12)	0.0419 (8)
H16	0.453295	−0.195077	0.017363	0.050*
C2	0.3651 (6)	0.4213 (4)	0.32682 (12)	0.0374 (7)
H2A	0.401270	0.420605	0.361224	0.045*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0776 (17)	0.0382 (10)	0.0542 (13)	−0.0067 (11)	−0.0121 (12)	−0.0048 (9)
N1	0.0292 (14)	0.0317 (12)	0.0366 (13)	−0.0075 (10)	0.0077 (11)	0.0018 (11)
N2	0.0284 (13)	0.0539 (16)	0.0276 (12)	0.0014 (12)	0.0064 (11)	0.0075 (11)
C6	0.0278 (15)	0.0242 (12)	0.0285 (13)	−0.0002 (11)	0.0056 (12)	−0.0006 (11)
C5	0.0270 (14)	0.0189 (11)	0.0262 (13)	0.0034 (10)	0.0038 (11)	−0.0006 (10)
C8	0.0283 (14)	0.0229 (12)	0.0253 (13)	0.0017 (11)	0.0028 (11)	−0.0011 (10)
C14	0.0341 (16)	0.0307 (14)	0.0220 (13)	0.0051 (12)	−0.0021 (12)	0.0010 (10)
C13	0.0307 (15)	0.0314 (13)	0.0171 (11)	0.0064 (11)	−0.0015 (11)	0.0028 (10)

C10	0.0286 (15)	0.0310 (13)	0.0196 (12)	0.0003 (11)	−0.0006 (11)	0.0040 (10)
C9	0.0337 (15)	0.0263 (12)	0.0256 (13)	0.0023 (12)	−0.0015 (12)	0.0007 (11)
C4	0.0296 (15)	0.0283 (13)	0.0299 (14)	−0.0019 (12)	0.0038 (12)	−0.0016 (11)
C12	0.0325 (16)	0.0433 (16)	0.0191 (12)	0.0083 (13)	0.0004 (12)	0.0052 (11)
C17	0.0423 (19)	0.0527 (19)	0.0233 (14)	0.0218 (16)	0.0028 (14)	0.0028 (13)
C7	0.0293 (15)	0.0312 (14)	0.0329 (15)	−0.0005 (12)	−0.0013 (12)	−0.0012 (12)
C3	0.0363 (18)	0.0389 (16)	0.0361 (16)	−0.0004 (14)	−0.0076 (14)	−0.0065 (13)
C11	0.0344 (17)	0.0390 (16)	0.0268 (14)	−0.0031 (13)	−0.0008 (13)	0.0070 (12)
C15	0.0465 (19)	0.0304 (14)	0.0265 (14)	0.0038 (13)	−0.0086 (14)	0.0013 (11)
C1	0.0425 (18)	0.0313 (14)	0.0294 (15)	0.0008 (13)	0.0117 (13)	0.0009 (12)
C16	0.063 (2)	0.0365 (16)	0.0256 (14)	0.0183 (16)	−0.0052 (16)	−0.0024 (12)
C2	0.0442 (19)	0.0417 (16)	0.0262 (14)	0.0063 (15)	−0.0012 (14)	−0.0030 (13)

Geometric parameters (Å, º)

F1—C15	1.357 (4)	C10—C9	1.507 (4)
N1—H1	0.8600	C10—C11	1.364 (4)
N1—C6	1.365 (4)	C9—H9A	0.9700
N1—C7	1.374 (4)	C9—H9B	0.9700
N2—H2	0.8600	C4—H4	0.9300
N2—C12	1.368 (4)	C4—C3	1.390 (4)
N2—C11	1.371 (4)	C12—C17	1.398 (4)
C6—C5	1.420 (4)	C17—H17	0.9300
C6—C1	1.393 (4)	C17—C16	1.372 (5)
C5—C8	1.435 (4)	C7—H7	0.9300
C5—C4	1.401 (4)	C3—H3	0.9300
C8—C9	1.497 (4)	C3—C2	1.398 (5)
C8—C7	1.373 (4)	C11—H11	0.9300
C14—H14	0.9300	C15—C16	1.396 (5)
C14—C13	1.391 (4)	C1—H1A	0.9300
C14—C15	1.366 (4)	C1—C2	1.375 (5)
C13—C10	1.442 (4)	C16—H16	0.9300
C13—C12	1.415 (4)	C2—H2A	0.9300
C6—N1—H1	125.2	C5—C4—H4	120.7
C6—N1—C7	109.6 (2)	C3—C4—C5	118.6 (3)
C7—N1—H1	125.2	C3—C4—H4	120.7
C12—N2—H2	125.5	N2—C12—C13	107.7 (3)
C12—N2—C11	109.0 (3)	N2—C12—C17	130.3 (3)
C11—N2—H2	125.5	C17—C12—C13	122.0 (3)
N1—C6—C5	107.1 (2)	C12—C17—H17	121.2
N1—C6—C1	130.6 (3)	C16—C17—C12	117.5 (3)
C1—C6—C5	122.3 (3)	C16—C17—H17	121.2
C6—C5—C8	107.2 (2)	N1—C7—H7	125.1
C4—C5—C6	118.8 (2)	C8—C7—N1	109.8 (3)
C4—C5—C8	133.9 (3)	C8—C7—H7	125.1
C5—C8—C9	127.7 (3)	C4—C3—H3	119.4
C7—C8—C5	106.2 (2)	C4—C3—C2	121.2 (3)

C7—C8—C9	126.0 (3)	C2—C3—H3	119.4
C13—C14—H14	121.3	N2—C11—H11	124.8
C15—C14—H14	121.3	C10—C11—N2	110.4 (3)
C15—C14—C13	117.4 (3)	C10—C11—H11	124.8
C14—C13—C10	133.9 (3)	F1—C15—C14	118.4 (3)
C14—C13—C12	119.4 (3)	F1—C15—C16	117.9 (3)
C12—C13—C10	106.7 (3)	C14—C15—C16	123.7 (3)
C13—C10—C9	127.0 (3)	C6—C1—H1A	121.3
C11—C10—C13	106.2 (3)	C2—C1—C6	117.4 (3)
C11—C10—C9	126.7 (3)	C2—C1—H1A	121.3
C8—C9—C10	115.6 (2)	C17—C16—C15	119.9 (3)
C8—C9—H9A	108.4	C17—C16—H16	120.0
C8—C9—H9B	108.4	C15—C16—H16	120.0
C10—C9—H9A	108.4	C3—C2—H2A	119.1
C10—C9—H9B	108.4	C1—C2—C3	121.7 (3)
H9A—C9—H9B	107.4	C1—C2—H2A	119.1

F1—C15—C16—C17	178.8 (3)	C13—C10—C11—N2	−1.1 (3)
N1—C6—C5—C8	1.5 (3)	C13—C12—C17—C16	0.0 (4)
N1—C6—C5—C4	178.5 (2)	C10—C13—C12—N2	0.3 (3)
N1—C6—C1—C2	−177.1 (3)	C10—C13—C12—C17	−179.0 (3)
N2—C12—C17—C16	−179.2 (3)	C9—C8—C7—N1	−177.8 (3)
C6—N1—C7—C8	1.6 (3)	C9—C10—C11—N2	−177.5 (3)
C6—C5—C8—C9	176.6 (3)	C4—C5—C8—C9	0.2 (5)
C6—C5—C8—C7	−0.5 (3)	C4—C5—C8—C7	−176.9 (3)
C6—C5—C4—C3	−0.6 (4)	C4—C3—C2—C1	1.2 (5)
C6—C1—C2—C3	−1.2 (5)	C12—N2—C11—C10	1.3 (3)
C5—C6—C1—C2	0.2 (4)	C12—C13—C10—C9	176.9 (3)
C5—C8—C9—C10	81.7 (4)	C12—C13—C10—C11	0.4 (3)
C5—C8—C7—N1	−0.6 (3)	C12—C17—C16—C15	0.1 (4)
C5—C4—C3—C2	−0.3 (5)	C7—N1—C6—C5	−1.9 (3)
C8—C5—C4—C3	175.5 (3)	C7—N1—C6—C1	175.8 (3)
C14—C13—C10—C9	−2.1 (5)	C7—C8—C9—C10	−101.7 (3)
C14—C13—C10—C11	−178.6 (3)	C11—N2—C12—C13	−1.0 (3)
C14—C13—C12—N2	179.5 (2)	C11—N2—C12—C17	178.3 (3)
C14—C13—C12—C17	0.2 (4)	C11—C10—C9—C8	−126.1 (3)
C14—C15—C16—C17	−0.4 (5)	C15—C14—C13—C10	178.5 (3)
C13—C14—C15—F1	−178.7 (2)	C15—C14—C13—C12	−0.4 (4)
C13—C14—C15—C16	0.6 (4)	C1—C6—C5—C8	−176.4 (3)
C13—C10—C9—C8	58.2 (4)	C1—C6—C5—C4	0.6 (4)