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# 5-[(*E*)-4-Fluorobenzylidene]-8-(4-fluorophenyl)-2-hydroxy-9-phenyl-3,10-diazahexacyclo[10.7.1.1$^{3,7}$.0$^{2,11}$.0$^{7,11}$.0$^{16,20}$]henicosa-1(20),12,14,16,18-pentaen-6-one

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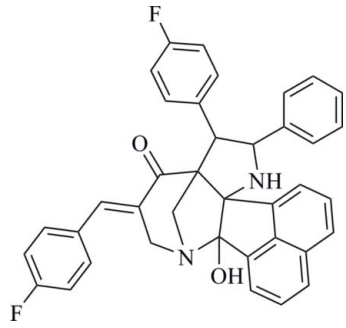
Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.044; $wR$ factor = 0.115; data-to-parameter ratio = 14.6.

In the title compound, $C_{38}H_{28}F_2N_2O_2$, the piperidine ring adopts a chair conformation and the pyrrolidine ring adopts an envelope conformation with the spiro C atom as the flap atom. The naphthalene ring system makes dihedral angles of 39.89 (8), 35.33 (8) and 46.45 (8)° with the two fluoro-substituted benzene rings and the phenyl ring, respectively, while the dihedral angle between the two fluoro-substituted benzene rings is 75.21 (10)°. An intramolecular O—H···N hydrogen bond generates an $S$(5) ring. In the crystal, molecules are connected by C—H···O hydrogen bonds, forming supramolecular chains propagating along the $c$-axis direction. Weak C—H···$\pi$ interactions further consolidate the structure.

## Related literature

For further details of 1,3-dipolar cycloaddition, see: Suresh Kumar *et al.* (2011); Jayashankaran *et al.* (2005); Manian *et al.* (2006); Williams & Fegley (1992). For ring conformations, see: Cremer & Pople (1975).




## Experimental

### Crystal data

$C_{38}H_{28}F_2N_2O_2$
$M_r$ = 582.62
Triclinic, $P\overline{1}$
$a$ = 9.3269 (3) Å
$b$ = 11.8635 (4) Å
$c$ = 14.2095 (4) Å
$\alpha$ = 75.904 (2)°
$\beta$ = 74.726 (2)°
$\gamma$ = 77.627 (2)°
$V$ = 1452.01 (8) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 296 K
0.39 × 0.25 × 0.15 mm

### Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min}$ = 0.965, $T_{max}$ = 0.987
16981 measured reflections
5915 independent reflections
4221 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.033

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.115
$S$ = 1.03
5915 reflections
405 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.17 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.20 e Å$^{-3}$

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

*Cg*5, *Cg*6, *Cg*7 and *Cg*9 are the centroids of the C1–C5/C10, C5–C10, C22–C27 and C34–C39 rings, respectively.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O2—H1*O2*···N2 | 0.90 (2) | 1.97 (2) | 2.636 (2) | 129.6 (17) |
| C13—H13*A*···O2$^{i}$ | 0.98 | 2.53 | 3.499 (2) | 171 |
| C20—H20*B*···O2$^{i}$ | 0.97 | 2.55 | 3.522 (2) | 179 |
| C23—H23*A*···O1$^{ii}$ | 0.93 | 2.52 | 3.410 (2) | 162 |
| C35—H35*A*···O2$^{i}$ | 0.93 | 2.46 | 3.383 (2) | 174 |
| C30—H30*A*···*Cg*5$^{iii}$ | 0.93 | 2.94 | 3.836 (2) | 168 |
| C31—H31*A*···*Cg*6$^{iii}$ | 0.93 | 2.91 | 3.609 (3) | 133 |
| C38—H38*A*···*Cg*7$^{iv}$ | 0.93 | 2.92 | 3.836 (2) | 168 |
| C7—H7*A*···*Cg*9$^{v}$ | 0.93 | 2.85 | 3.400 (3) | 119 |

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6426).

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# supplementary materials

*Acta Cryst.* (2011). E**67**, o2879-o2880 [ **doi:10.1107/S1600536811040633** ]

# 5-[(*E*)-4-Fluorobenzylidene]-8-(4-fluorophenyl)-2-hydroxy-9-phenyl-3,10-diazahexacyclo[10.7.1.1$^{3,7}$.0$^{2,11}$.0$^{7,11}$.0$^{16,20}$\]henicosa-1(20),12,14,16,18-pentaen-6-one

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## Comment

1,3-Dipolar cycloaddition provides a facile route for the synthesis of many dispiroheterocyclic systems through the cycloaddition reaction of azomethine ylides with the definite dipolarophiles (Suresh Kumar *et al.*, 2011; Jayashankaran *et al.*, 2005; Manian *et al.*, 2006). This method is widely used as one of the key steps for the synthesis of natural products such as alkaloids and pharmacologically important compounds (Williams & Fegley, 1992). The significance of these heterocycles prompted us to investigate the crystal structure determination of the title compound and report the results in this paper.

The asymmetric unit of the title compound is shown in Fig. 1. The piperidine (N1/C15/C17–C20) ring adopts a chair conformation [Q = 0.6152 (17) Å; θ = 39.34 (16)° and φ = 298.6 (2)°; Cremer & Pople, 1975] and the pyrrolidine (N2/C13–C16) ring adopts an envelope conformation with the spiro C14 atom as the flap atom (displacement -0.230 (2) Å) and with puckering parameters, Q = 0.3614 (17) Å; and φ = 252.3 (3)°. The naphthalene (C1–C10) ring makes dihedral angles of 39.89 (8)°, 35.33 (8)° and 46.45 (8)° with the two fluoro-substituted (C22–C27)/(C34–C39) phenyl rings and the benzene (C28–C33) ring, repectively. The corresponding angle between the two fluoro-substituted phenyl (C22–C27)/(C34–C39) rings is 75.21 (10)°.

In the crystal, (Fig. 2), the molecules are connected *via* C—H···O (Table 1) hydrogen bonds forming one-dimensional supramolecular chains along the *c*-axis. Furthermore, the crystal structure is stabilized by weak C—H···π interactions involving the centroids of the Cg5 (C1–C5/C10); Cg6 (C5–C10); Cg7 (C22–C27) and Cg9 (C34–C39) rings.

## Experimental

A mixture of 3,5-bis[(*E*)-(4-fluorophenyl)methylidene]tetrahydro-4 (1*H*)-pyridinone (1 mmol), acenaphthenequinone (1 mmol), and phenyl glycine (1 mmol) were dissolved in methanol (5 mL) and refluxed in a water bath for 1 hour. After completion of the reaction as evident from TLC, the mixture was poured into water (50 mL). The precipitated solid was filtered and washed with water to obtain the product which was further purified by recrystallisation from ethyl acetate to yield colourless blocks.

## Refinement

Atoms H1O2 and H1N2 were located from a difference Fourier maps and refined freely [N–H = 0.91 (2) Å and O–H = 0.90 (2) Å]. The remaining H atoms were positioned geometrically [C–H = 0.93–0.98 Å] and were refined using a riding model, with $U_{iso}$(H) = 1.2 $U_{eq}$(C).

**Figures**

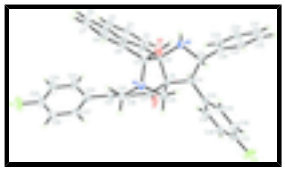

Fig. 1. The asymmetric unit of the title compound, showing 30% probability displacement ellipsoids.

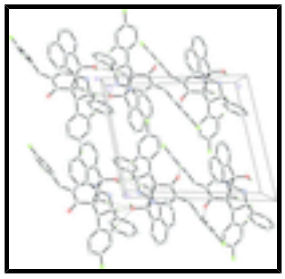

Fig. 2. The crystal packing of the title compound (I). H atoms not involved in hydrogen bonding are omitted.

## 5-[(*E*)-4-Fluorobenzylidene]-8-(4-fluorophenyl)-2-hydroxy-9- phenyl-3,10-diazahexacyclo[10.7.1.1$^{3,7}$.0$^{2,11}$.0$^{7,11}$.0$^{16,20}$]henicosa- 1(20),12,14,16,18-pentaen-6-one

*Crystal data*

| | |
|---|---|
| $C_{38}H_{28}F_2N_2O_2$ | $Z = 2$ |
| $M_r = 582.62$ | $F(000) = 608$ |
| Triclinic, $P\overline{1}$ | $D_x = 1.333$ Mg m$^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $a = 9.3269$ (3) Å | Cell parameters from 4467 reflections |
| $b = 11.8635$ (4) Å | $\theta = 2.6$–$27.1°$ |
| $c = 14.2095$ (4) Å | $\mu = 0.09$ mm$^{-1}$ |
| $\alpha = 75.904$ (2)° | $T = 296$ K |
| $\beta = 74.726$ (2)° | Block, colourless |
| $\gamma = 77.627$ (2)° | 0.39 × 0.25 × 0.15 mm |
| $V = 1452.01$ (8) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Bruker SMART APEXII CCD diffractometer | 5915 independent reflections |
| Radiation source: fine-focus sealed tube | 4221 reflections with $I > 2\sigma(I)$ |
| graphite | $R_{int} = 0.033$ |
| φ and ω scans | $\theta_{max} = 26.5°$, $\theta_{min} = 1.8°$ |
| Absorption correction: multi-scan (*SADABS*; Bruker, 2009) | $h = -11 \rightarrow 11$ |
| $T_{min} = 0.965$, $T_{max} = 0.987$ | $k = -14 \rightarrow 14$ |
| 16981 measured reflections | $l = -17 \rightarrow 17$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.044$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.115$ | H atoms treated by a mixture of independent and constrained refinement |
| $S = 1.03$ | $w = 1/[\sigma^2(F_o^2) + (0.0505P)^2 + 0.1929P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 5915 reflections | $(\Delta/\sigma)_{max} < 0.001$ |
| 405 parameters | $\Delta\rho_{max} = 0.17$ e Å$^{-3}$ |
| 0 restraints | $\Delta\rho_{min} = -0.20$ e Å$^{-3}$ |

*Special details*

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

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted R-factor wR and goodness of fit S are based on $F^2$, conventional R-factors R are based on F, with F set to zero for negative $F^2$. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on $F^2$ are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| F1 | 0.31463 (18) | 0.41565 (14) | 0.63374 (12) | 0.1021 (5) |
| F2 | 0.56249 (16) | −0.63672 (10) | 0.27077 (12) | 0.0887 (4) |
| O1 | 0.19391 (15) | −0.13595 (11) | 0.41318 (8) | 0.0499 (3) |
| O2 | 0.40049 (15) | 0.12921 (11) | 0.03939 (8) | 0.0451 (3) |
| N1 | 0.46807 (15) | 0.06737 (11) | 0.19282 (9) | 0.0358 (3) |
| N2 | 0.17795 (18) | 0.00560 (12) | 0.10268 (9) | 0.0376 (3) |
| C1 | 0.08709 (18) | 0.11620 (13) | 0.24038 (10) | 0.0338 (3) |
| C2 | −0.0512 (2) | 0.09298 (16) | 0.29527 (12) | 0.0451 (4) |
| H2A | −0.0774 | 0.0189 | 0.3052 | 0.054* |
| C3 | −0.1538 (2) | 0.18431 (18) | 0.33664 (14) | 0.0553 (5) |
| H3A | −0.2463 | 0.1678 | 0.3765 | 0.066* |
| C4 | −0.1225 (2) | 0.29506 (18) | 0.32049 (14) | 0.0556 (5) |
| H4A | −0.1936 | 0.3526 | 0.3486 | 0.067* |
| C5 | 0.0179 (2) | 0.32359 (15) | 0.26106 (12) | 0.0445 (4) |
| C6 | 0.0639 (3) | 0.43508 (16) | 0.22979 (15) | 0.0567 (5) |
| H6A | −0.0006 | 0.5002 | 0.2507 | 0.068* |
| C7 | 0.2025 (3) | 0.44862 (16) | 0.16899 (16) | 0.0583 (5) |

| | | | | |
|---|---|---|---|---|
| H7A | 0.2288 | 0.5236 | 0.1481 | 0.070* |
| C8 | 0.3068 (2) | 0.35270 (15) | 0.13694 (13) | 0.0477 (4) |
| H8A | 0.4013 | 0.3636 | 0.0970 | 0.057* |
| C9 | 0.26533 (19) | 0.24316 (14) | 0.16619 (11) | 0.0370 (4) |
| C10 | 0.12161 (19) | 0.23011 (13) | 0.22507 (11) | 0.0359 (4) |
| C11 | 0.34421 (18) | 0.12516 (13) | 0.14237 (10) | 0.0344 (4) |
| C13 | 0.23219 (19) | −0.12206 (13) | 0.10589 (11) | 0.0350 (4) |
| H13A | 0.3328 | −0.1310 | 0.0621 | 0.042* |
| C14 | 0.24873 (18) | −0.17285 (13) | 0.21430 (10) | 0.0333 (3) |
| H14A | 0.1473 | −0.1791 | 0.2554 | 0.040* |
| C15 | 0.30126 (17) | −0.07313 (13) | 0.24017 (10) | 0.0316 (3) |
| C16 | 0.21783 (18) | 0.04178 (13) | 0.18294 (10) | 0.0325 (3) |
| C17 | 0.26517 (18) | −0.06749 (13) | 0.34904 (11) | 0.0343 (4) |
| C18 | 0.31892 (18) | 0.03074 (13) | 0.37188 (11) | 0.0345 (4) |
| C19 | 0.44652 (19) | 0.08607 (15) | 0.29507 (11) | 0.0381 (4) |
| H19A | 0.4271 | 0.1701 | 0.2934 | 0.046* |
| H19B | 0.5397 | 0.0543 | 0.3171 | 0.046* |
| C20 | 0.46882 (18) | −0.05780 (14) | 0.19841 (12) | 0.0373 (4) |
| H20A | 0.5312 | −0.1069 | 0.2426 | 0.045* |
| H20B | 0.5058 | −0.0778 | 0.1331 | 0.045* |
| C21 | 0.2415 (2) | 0.06910 (14) | 0.45470 (11) | 0.0397 (4) |
| H21A | 0.1604 | 0.0311 | 0.4903 | 0.048* |
| C22 | 0.26546 (19) | 0.16222 (15) | 0.49779 (11) | 0.0397 (4) |
| C23 | 0.1410 (2) | 0.23249 (19) | 0.54396 (15) | 0.0630 (6) |
| H23A | 0.0448 | 0.2219 | 0.5445 | 0.076* |
| C24 | 0.1579 (3) | 0.3180 (2) | 0.58917 (18) | 0.0773 (7) |
| H24A | 0.0740 | 0.3656 | 0.6192 | 0.093* |
| C25 | 0.2994 (3) | 0.33166 (19) | 0.58914 (15) | 0.0619 (6) |
| C26 | 0.4247 (2) | 0.26458 (17) | 0.54571 (14) | 0.0540 (5) |
| H26A | 0.5201 | 0.2753 | 0.5468 | 0.065* |
| C27 | 0.4069 (2) | 0.17962 (16) | 0.49972 (12) | 0.0457 (4) |
| H27A | 0.4918 | 0.1332 | 0.4694 | 0.055* |
| C28 | 0.1336 (2) | −0.18155 (14) | 0.07132 (12) | 0.0394 (4) |
| C29 | 0.1885 (2) | −0.22550 (17) | −0.01543 (13) | 0.0551 (5) |
| H29A | 0.2832 | −0.2131 | −0.0543 | 0.066* |
| C30 | 0.1038 (3) | −0.28786 (19) | −0.04489 (18) | 0.0736 (7) |
| H30A | 0.1420 | −0.3172 | −0.1031 | 0.088* |
| C31 | −0.0349 (3) | −0.30622 (19) | 0.0110 (2) | 0.0766 (7) |
| H31A | −0.0904 | −0.3495 | −0.0084 | 0.092* |
| C32 | −0.0932 (3) | −0.2612 (2) | 0.09581 (17) | 0.0706 (6) |
| H32A | −0.1889 | −0.2725 | 0.1333 | 0.085* |
| C33 | −0.0093 (2) | −0.19859 (18) | 0.12561 (14) | 0.0551 (5) |
| H33A | −0.0497 | −0.1676 | 0.1829 | 0.066* |
| C34 | 0.34205 (19) | −0.29458 (13) | 0.23043 (11) | 0.0361 (4) |
| C35 | 0.4672 (2) | −0.33131 (15) | 0.16058 (13) | 0.0463 (4) |
| H35A | 0.5004 | −0.2780 | 0.1029 | 0.056* |
| C36 | 0.5438 (2) | −0.44564 (16) | 0.17465 (16) | 0.0561 (5) |
| H36A | 0.6284 | −0.4692 | 0.1277 | 0.067* |
| C37 | 0.4923 (2) | −0.52266 (15) | 0.25895 (17) | 0.0574 (5) |

| | | | | |
|---|---|---|---|---|
| C38 | 0.3723 (3) | −0.49040 (17) | 0.33078 (16) | 0.0604 (5) |
| H38A | 0.3414 | −0.5444 | 0.3885 | 0.073* |
| C39 | 0.2972 (2) | −0.37606 (15) | 0.31647 (13) | 0.0485 (4) |
| H39A | 0.2150 | −0.3531 | 0.3653 | 0.058* |
| H1O2 | 0.334 (2) | 0.0994 (18) | 0.0207 (15) | 0.068 (7)* |
| H1N2 | 0.078 (2) | 0.0265 (17) | 0.1045 (15) | 0.063 (6)* |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| F1 | 0.1084 (12) | 0.1056 (11) | 0.1283 (12) | −0.0232 (9) | −0.0316 (10) | −0.0792 (10) |
| F2 | 0.0824 (10) | 0.0379 (6) | 0.1443 (12) | 0.0081 (6) | −0.0506 (9) | −0.0034 (7) |
| O1 | 0.0685 (9) | 0.0515 (7) | 0.0319 (6) | −0.0280 (6) | −0.0041 (6) | −0.0042 (5) |
| O2 | 0.0559 (8) | 0.0511 (7) | 0.0276 (6) | −0.0205 (6) | 0.0018 (5) | −0.0083 (5) |
| N1 | 0.0367 (8) | 0.0365 (7) | 0.0340 (7) | −0.0090 (6) | −0.0025 (6) | −0.0097 (5) |
| N2 | 0.0495 (9) | 0.0322 (7) | 0.0346 (7) | −0.0078 (7) | −0.0143 (6) | −0.0067 (5) |
| C1 | 0.0363 (9) | 0.0351 (8) | 0.0293 (7) | −0.0041 (7) | −0.0074 (7) | −0.0062 (6) |
| C2 | 0.0418 (10) | 0.0445 (10) | 0.0446 (9) | −0.0072 (8) | −0.0053 (8) | −0.0054 (8) |
| C3 | 0.0398 (11) | 0.0667 (13) | 0.0510 (11) | −0.0010 (9) | 0.0002 (9) | −0.0138 (9) |
| C4 | 0.0485 (12) | 0.0602 (13) | 0.0569 (11) | 0.0106 (10) | −0.0112 (9) | −0.0270 (9) |
| C5 | 0.0515 (11) | 0.0416 (10) | 0.0458 (9) | 0.0031 (8) | −0.0189 (8) | −0.0194 (8) |
| C6 | 0.0693 (14) | 0.0393 (10) | 0.0703 (13) | 0.0041 (10) | −0.0281 (11) | −0.0247 (9) |
| C7 | 0.0788 (16) | 0.0332 (10) | 0.0742 (13) | −0.0109 (10) | −0.0338 (12) | −0.0117 (9) |
| C8 | 0.0576 (12) | 0.0374 (10) | 0.0532 (10) | −0.0153 (9) | −0.0162 (9) | −0.0075 (8) |
| C9 | 0.0470 (10) | 0.0334 (8) | 0.0338 (8) | −0.0086 (7) | −0.0122 (7) | −0.0073 (6) |
| C10 | 0.0439 (10) | 0.0344 (8) | 0.0322 (8) | −0.0044 (7) | −0.0127 (7) | −0.0087 (6) |
| C11 | 0.0393 (9) | 0.0351 (8) | 0.0281 (7) | −0.0104 (7) | −0.0012 (7) | −0.0076 (6) |
| C13 | 0.0413 (9) | 0.0325 (8) | 0.0317 (8) | −0.0078 (7) | −0.0060 (7) | −0.0080 (6) |
| C14 | 0.0366 (9) | 0.0332 (8) | 0.0307 (7) | −0.0083 (7) | −0.0056 (6) | −0.0074 (6) |
| C15 | 0.0354 (9) | 0.0300 (8) | 0.0291 (7) | −0.0066 (7) | −0.0052 (6) | −0.0063 (6) |
| C16 | 0.0390 (9) | 0.0296 (8) | 0.0290 (7) | −0.0085 (7) | −0.0059 (6) | −0.0056 (6) |
| C17 | 0.0358 (9) | 0.0348 (8) | 0.0311 (8) | −0.0061 (7) | −0.0070 (7) | −0.0047 (6) |
| C18 | 0.0372 (9) | 0.0362 (8) | 0.0307 (8) | −0.0071 (7) | −0.0087 (7) | −0.0056 (6) |
| C19 | 0.0381 (9) | 0.0423 (9) | 0.0369 (8) | −0.0115 (7) | −0.0075 (7) | −0.0102 (7) |
| C20 | 0.0374 (9) | 0.0366 (9) | 0.0364 (8) | −0.0048 (7) | −0.0037 (7) | −0.0101 (7) |
| C21 | 0.0435 (10) | 0.0435 (9) | 0.0339 (8) | −0.0133 (8) | −0.0071 (7) | −0.0069 (7) |
| C22 | 0.0447 (10) | 0.0454 (10) | 0.0301 (8) | −0.0108 (8) | −0.0043 (7) | −0.0107 (7) |
| C23 | 0.0445 (11) | 0.0833 (15) | 0.0718 (13) | −0.0175 (11) | 0.0027 (10) | −0.0456 (12) |
| C24 | 0.0604 (14) | 0.0917 (17) | 0.0944 (17) | −0.0149 (13) | 0.0037 (12) | −0.0661 (14) |
| C25 | 0.0756 (15) | 0.0627 (13) | 0.0626 (12) | −0.0189 (11) | −0.0156 (11) | −0.0335 (10) |
| C26 | 0.0565 (12) | 0.0600 (12) | 0.0558 (11) | −0.0145 (10) | −0.0243 (10) | −0.0136 (9) |
| C27 | 0.0448 (10) | 0.0504 (11) | 0.0451 (9) | −0.0045 (8) | −0.0149 (8) | −0.0130 (8) |
| C28 | 0.0511 (11) | 0.0321 (8) | 0.0381 (8) | −0.0064 (7) | −0.0169 (8) | −0.0055 (7) |
| C29 | 0.0700 (14) | 0.0553 (12) | 0.0475 (10) | −0.0040 (10) | −0.0224 (10) | −0.0192 (9) |
| C30 | 0.109 (2) | 0.0606 (14) | 0.0721 (14) | −0.0011 (14) | −0.0513 (15) | −0.0295 (11) |
| C31 | 0.113 (2) | 0.0535 (13) | 0.0912 (17) | −0.0260 (14) | −0.0699 (17) | −0.0037 (12) |
| C32 | 0.0741 (15) | 0.0735 (15) | 0.0764 (15) | −0.0347 (12) | −0.0385 (13) | 0.0060 (12) |
| C33 | 0.0587 (13) | 0.0617 (12) | 0.0526 (11) | −0.0207 (10) | −0.0174 (10) | −0.0107 (9) |

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|---|---|---|---|---|---|---|
| C34 | 0.0414 (9) | 0.0313 (8) | 0.0395 (8) | −0.0102 (7) | −0.0120 (7) | −0.0074 (7) |
| C35 | 0.0485 (11) | 0.0342 (9) | 0.0546 (10) | −0.0083 (8) | −0.0073 (9) | −0.0087 (8) |
| C36 | 0.0456 (11) | 0.0436 (11) | 0.0806 (14) | −0.0014 (9) | −0.0146 (10) | −0.0195 (10) |
| C37 | 0.0573 (13) | 0.0317 (10) | 0.0896 (15) | −0.0016 (9) | −0.0389 (12) | −0.0045 (10) |
| C38 | 0.0750 (15) | 0.0423 (11) | 0.0630 (12) | −0.0126 (10) | −0.0296 (11) | 0.0096 (9) |
| C39 | 0.0589 (12) | 0.0414 (10) | 0.0442 (9) | −0.0119 (9) | −0.0133 (9) | −0.0013 (8) |

*Geometric parameters (Å, °)*

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| F1—C25 | 1.353 (2) | C17—C18 | 1.495 (2) |
| F2—C37 | 1.362 (2) | C18—C21 | 1.336 (2) |
| O1—C17 | 1.2181 (18) | C18—C19 | 1.530 (2) |
| O2—C11 | 1.4110 (17) | C19—H19A | 0.9700 |
| O2—H1O2 | 0.90 (2) | C19—H19B | 0.9700 |
| N1—C20 | 1.466 (2) | C20—H20A | 0.9700 |
| N1—C11 | 1.475 (2) | C20—H20B | 0.9700 |
| N1—C19 | 1.4781 (19) | C21—C22 | 1.467 (2) |
| N2—C16 | 1.4675 (19) | C21—H21A | 0.9300 |
| N2—C13 | 1.484 (2) | C22—C27 | 1.386 (2) |
| N2—H1N2 | 0.91 (2) | C22—C23 | 1.387 (2) |
| C1—C2 | 1.366 (2) | C23—C24 | 1.380 (3) |
| C1—C10 | 1.408 (2) | C23—H23A | 0.9300 |
| C1—C16 | 1.516 (2) | C24—C25 | 1.363 (3) |
| C2—C3 | 1.416 (2) | C24—H24A | 0.9300 |
| C2—H2A | 0.9300 | C25—C26 | 1.357 (3) |
| C3—C4 | 1.359 (3) | C26—C27 | 1.386 (2) |
| C3—H3A | 0.9300 | C26—H26A | 0.9300 |
| C4—C5 | 1.418 (3) | C27—H27A | 0.9300 |
| C4—H4A | 0.9300 | C28—C33 | 1.382 (3) |
| C5—C10 | 1.408 (2) | C28—C29 | 1.386 (2) |
| C5—C6 | 1.410 (3) | C29—C30 | 1.387 (3) |
| C6—C7 | 1.370 (3) | C29—H29A | 0.9300 |
| C6—H6A | 0.9300 | C30—C31 | 1.359 (3) |
| C7—C8 | 1.412 (3) | C30—H30A | 0.9300 |
| C7—H7A | 0.9300 | C31—C32 | 1.371 (3) |
| C8—C9 | 1.371 (2) | C31—H31A | 0.9300 |
| C8—H8A | 0.9300 | C32—C33 | 1.386 (3) |
| C9—C10 | 1.398 (2) | C32—H32A | 0.9300 |
| C9—C11 | 1.508 (2) | C33—H33A | 0.9300 |
| C11—C16 | 1.601 (2) | C34—C35 | 1.385 (2) |
| C13—C28 | 1.506 (2) | C34—C39 | 1.390 (2) |
| C13—C14 | 1.547 (2) | C35—C36 | 1.385 (2) |
| C13—H13A | 0.9800 | C35—H35A | 0.9300 |
| C14—C34 | 1.517 (2) | C36—C37 | 1.361 (3) |
| C14—C15 | 1.526 (2) | C36—H36A | 0.9300 |
| C14—H14A | 0.9800 | C37—C38 | 1.359 (3) |
| C15—C17 | 1.509 (2) | C38—C39 | 1.381 (3) |
| C15—C20 | 1.552 (2) | C38—H38A | 0.9300 |
| C15—C16 | 1.570 (2) | C39—H39A | 0.9300 |

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| C11—O2—H1O2 | 104.9 (13) | C21—C18—C19 | 125.49 (15) |
| C20—N1—C11 | 102.48 (12) | C17—C18—C19 | 118.04 (12) |
| C20—N1—C19 | 108.12 (12) | N1—C19—C18 | 115.25 (13) |
| C11—N1—C19 | 115.60 (12) | N1—C19—H19A | 108.5 |
| C16—N2—C13 | 109.96 (12) | C18—C19—H19A | 108.5 |
| C16—N2—H1N2 | 111.2 (13) | N1—C19—H19B | 108.5 |
| C13—N2—H1N2 | 113.2 (13) | C18—C19—H19B | 108.5 |
| C2—C1—C10 | 119.20 (15) | H19A—C19—H19B | 107.5 |
| C2—C1—C16 | 131.77 (15) | N1—C20—C15 | 104.08 (12) |
| C10—C1—C16 | 108.91 (13) | N1—C20—H20A | 110.9 |
| C1—C2—C3 | 118.44 (17) | C15—C20—H20A | 110.9 |
| C1—C2—H2A | 120.8 | N1—C20—H20B | 110.9 |
| C3—C2—H2A | 120.8 | C15—C20—H20B | 110.9 |
| C4—C3—C2 | 122.67 (18) | H20A—C20—H20B | 109.0 |
| C4—C3—H3A | 118.7 | C18—C21—C22 | 129.62 (16) |
| C2—C3—H3A | 118.7 | C18—C21—H21A | 115.2 |
| C3—C4—C5 | 120.48 (16) | C22—C21—H21A | 115.2 |
| C3—C4—H4A | 119.8 | C27—C22—C23 | 117.87 (16) |
| C5—C4—H4A | 119.8 | C27—C22—C21 | 123.22 (15) |
| C10—C5—C6 | 115.95 (17) | C23—C22—C21 | 118.81 (16) |
| C10—C5—C4 | 116.03 (16) | C24—C23—C22 | 120.89 (19) |
| C6—C5—C4 | 127.95 (17) | C24—C23—H23A | 119.6 |
| C7—C6—C5 | 120.74 (17) | C22—C23—H23A | 119.6 |
| C7—C6—H6A | 119.6 | C25—C24—C23 | 119.03 (19) |
| C5—C6—H6A | 119.6 | C25—C24—H24A | 120.5 |
| C6—C7—C8 | 122.24 (18) | C23—C24—H24A | 120.5 |
| C6—C7—H7A | 118.9 | F1—C25—C26 | 119.1 (2) |
| C8—C7—H7A | 118.9 | F1—C25—C24 | 118.54 (19) |
| C9—C8—C7 | 118.34 (18) | C26—C25—C24 | 122.37 (18) |
| C9—C8—H8A | 120.8 | C25—C26—C27 | 118.26 (18) |
| C7—C8—H8A | 120.8 | C25—C26—H26A | 120.9 |
| C8—C9—C10 | 119.39 (16) | C27—C26—H26A | 120.9 |
| C8—C9—C11 | 132.10 (16) | C26—C27—C22 | 121.57 (17) |
| C10—C9—C11 | 108.41 (13) | C26—C27—H27A | 119.2 |
| C9—C10—C5 | 123.22 (16) | C22—C27—H27A | 119.2 |
| C9—C10—C1 | 113.65 (14) | C33—C28—C29 | 118.09 (17) |
| C5—C10—C1 | 123.03 (16) | C33—C28—C13 | 122.14 (15) |
| O2—C11—N1 | 107.74 (12) | C29—C28—C13 | 119.71 (17) |
| O2—C11—C9 | 112.93 (12) | C28—C29—C30 | 120.6 (2) |
| N1—C11—C9 | 115.59 (12) | C28—C29—H29A | 119.7 |
| O2—C11—C16 | 109.11 (12) | C30—C29—H29A | 119.7 |
| N1—C11—C16 | 106.18 (11) | C31—C30—C29 | 120.3 (2) |
| C9—C11—C16 | 104.93 (12) | C31—C30—H30A | 119.9 |
| N2—C13—C28 | 113.74 (14) | C29—C30—H30A | 119.9 |
| N2—C13—C14 | 104.58 (11) | C30—C31—C32 | 120.2 (2) |
| C28—C13—C14 | 114.42 (13) | C30—C31—H31A | 119.9 |
| N2—C13—H13A | 107.9 | C32—C31—H31A | 119.9 |
| C28—C13—H13A | 107.9 | C31—C32—C33 | 119.9 (2) |
| C14—C13—H13A | 107.9 | C31—C32—H32A | 120.1 |

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| C34—C14—C15 | 117.75 (13) | C33—C32—H32A | 120.1 |
| C34—C14—C13 | 114.81 (12) | C28—C33—C32 | 120.91 (19) |
| C15—C14—C13 | 102.44 (11) | C28—C33—H33A | 119.5 |
| C34—C14—H14A | 107.1 | C32—C33—H33A | 119.5 |
| C15—C14—H14A | 107.1 | C35—C34—C39 | 117.66 (16) |
| C13—C14—H14A | 107.1 | C35—C34—C14 | 122.94 (14) |
| C17—C15—C14 | 116.38 (12) | C39—C34—C14 | 119.31 (15) |
| C17—C15—C20 | 106.73 (12) | C36—C35—C34 | 121.48 (17) |
| C14—C15—C20 | 117.62 (12) | C36—C35—H35A | 119.3 |
| C17—C15—C16 | 108.69 (12) | C34—C35—H35A | 119.3 |
| C14—C15—C16 | 104.15 (12) | C37—C36—C35 | 118.36 (19) |
| C20—C15—C16 | 101.98 (12) | C37—C36—H36A | 120.8 |
| N2—C16—C1 | 112.71 (13) | C35—C36—H36A | 120.8 |
| N2—C16—C15 | 105.47 (12) | C38—C37—C36 | 122.46 (17) |
| C1—C16—C15 | 119.60 (12) | C38—C37—F2 | 118.92 (19) |
| N2—C16—C11 | 112.65 (12) | C36—C37—F2 | 118.6 (2) |
| C1—C16—C11 | 103.30 (12) | C37—C38—C39 | 118.78 (18) |
| C15—C16—C11 | 102.82 (12) | C37—C38—H38A | 120.6 |
| O1—C17—C18 | 122.52 (13) | C39—C38—H38A | 120.6 |
| O1—C17—C15 | 122.42 (14) | C38—C39—C34 | 121.21 (18) |
| C18—C17—C15 | 115.02 (13) | C38—C39—H39A | 119.4 |
| C21—C18—C17 | 116.22 (14) | C34—C39—H39A | 119.4 |
| C10—C1—C2—C3 | −1.3 (2) | N1—C11—C16—N2 | −124.04 (13) |
| C16—C1—C2—C3 | −176.77 (16) | C9—C11—C16—N2 | 113.09 (14) |
| C1—C2—C3—C4 | 2.8 (3) | O2—C11—C16—C1 | −130.08 (13) |
| C2—C3—C4—C5 | −0.7 (3) | N1—C11—C16—C1 | 114.06 (12) |
| C3—C4—C5—C10 | −2.7 (3) | C9—C11—C16—C1 | −8.81 (14) |
| C3—C4—C5—C6 | 174.06 (18) | O2—C11—C16—C15 | 104.87 (13) |
| C10—C5—C6—C7 | −0.7 (3) | N1—C11—C16—C15 | −11.00 (14) |
| C4—C5—C6—C7 | −177.53 (18) | C9—C11—C16—C15 | −133.86 (12) |
| C5—C6—C7—C8 | −1.7 (3) | C14—C15—C17—O1 | −2.3 (2) |
| C6—C7—C8—C9 | 1.6 (3) | C20—C15—C17—O1 | −135.86 (16) |
| C7—C8—C9—C10 | 0.9 (2) | C16—C15—C17—O1 | 114.85 (17) |
| C7—C8—C9—C11 | 176.86 (17) | C14—C15—C17—C18 | 179.67 (13) |
| C8—C9—C10—C5 | −3.5 (2) | C20—C15—C17—C18 | 46.08 (17) |
| C11—C9—C10—C5 | 179.68 (14) | C16—C15—C17—C18 | −63.21 (17) |
| C8—C9—C10—C1 | 173.13 (14) | O1—C17—C18—C21 | −24.6 (2) |
| C11—C9—C10—C1 | −3.69 (18) | C15—C17—C18—C21 | 153.42 (15) |
| C6—C5—C10—C9 | 3.3 (2) | O1—C17—C18—C19 | 160.88 (15) |
| C4—C5—C10—C9 | −179.46 (15) | C15—C17—C18—C19 | −21.1 (2) |
| C6—C5—C10—C1 | −172.97 (15) | C20—N1—C19—C18 | −47.68 (17) |
| C4—C5—C10—C1 | 4.2 (2) | C11—N1—C19—C18 | 66.45 (17) |
| C2—C1—C10—C9 | −178.85 (14) | C21—C18—C19—N1 | −153.25 (16) |
| C16—C1—C10—C9 | −2.45 (18) | C17—C18—C19—N1 | 20.7 (2) |
| C2—C1—C10—C5 | −2.2 (2) | C11—N1—C20—C15 | −48.50 (13) |
| C16—C1—C10—C5 | 174.19 (14) | C19—N1—C20—C15 | 74.05 (15) |
| C20—N1—C11—O2 | −80.31 (13) | C17—C15—C20—N1 | −73.35 (14) |
| C19—N1—C11—O2 | 162.36 (12) | C14—C15—C20—N1 | 153.74 (12) |
| C20—N1—C11—C9 | 152.34 (13) | C16—C15—C20—N1 | 40.60 (14) |

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| C19—N1—C11—C9 | 35.01 (18) | C17—C18—C21—C22 | 179.54 (16) |
| C20—N1—C11—C16 | 36.48 (13) | C19—C18—C21—C22 | −6.4 (3) |
| C19—N1—C11—C16 | −80.86 (14) | C18—C21—C22—C27 | −40.3 (3) |
| C8—C9—C11—O2 | −49.7 (2) | C18—C21—C22—C23 | 143.4 (2) |
| C10—C9—C11—O2 | 126.53 (14) | C27—C22—C23—C24 | 0.8 (3) |
| C8—C9—C11—N1 | 75.0 (2) | C21—C22—C23—C24 | 177.3 (2) |
| C10—C9—C11—N1 | −108.77 (15) | C22—C23—C24—C25 | −0.9 (4) |
| C8—C9—C11—C16 | −168.46 (17) | C23—C24—C25—F1 | −179.9 (2) |
| C10—C9—C11—C16 | 7.80 (16) | C23—C24—C25—C26 | 0.4 (4) |
| C16—N2—C13—C28 | 148.04 (13) | F1—C25—C26—C27 | −179.51 (18) |
| C16—N2—C13—C14 | 22.51 (17) | C24—C25—C26—C27 | 0.2 (3) |
| N2—C13—C14—C34 | −164.10 (13) | C25—C26—C27—C22 | −0.2 (3) |
| C28—C13—C14—C34 | 70.80 (18) | C23—C22—C27—C26 | −0.3 (3) |
| N2—C13—C14—C15 | −35.25 (16) | C21—C22—C27—C26 | −176.57 (16) |
| C28—C13—C14—C15 | −160.35 (13) | N2—C13—C28—C33 | −69.3 (2) |
| C34—C14—C15—C17 | −78.91 (17) | C14—C13—C28—C33 | 50.8 (2) |
| C13—C14—C15—C17 | 154.11 (13) | N2—C13—C28—C29 | 113.44 (17) |
| C34—C14—C15—C20 | 49.57 (18) | C14—C13—C28—C29 | −126.44 (16) |
| C13—C14—C15—C20 | −77.41 (16) | C33—C28—C29—C30 | −1.9 (3) |
| C34—C14—C15—C16 | 161.50 (12) | C13—C28—C29—C30 | 175.42 (17) |
| C13—C14—C15—C16 | 34.52 (14) | C28—C29—C30—C31 | 0.3 (3) |
| C13—N2—C16—C1 | −132.84 (14) | C29—C30—C31—C32 | 1.3 (3) |
| C13—N2—C16—C15 | −0.66 (16) | C30—C31—C32—C33 | −1.2 (3) |
| C13—N2—C16—C11 | 110.74 (14) | C29—C28—C33—C32 | 2.1 (3) |
| C2—C1—C16—N2 | 60.9 (2) | C13—C28—C33—C32 | −175.23 (17) |
| C10—C1—C16—N2 | −114.87 (14) | C31—C32—C33—C28 | −0.5 (3) |
| C2—C1—C16—C15 | −63.9 (2) | C15—C14—C34—C35 | −85.00 (19) |
| C10—C1—C16—C15 | 120.36 (14) | C13—C14—C34—C35 | 35.7 (2) |
| C2—C1—C16—C11 | −177.22 (16) | C15—C14—C34—C39 | 98.43 (18) |
| C10—C1—C16—C11 | 7.00 (15) | C13—C14—C34—C39 | −140.82 (16) |
| C17—C15—C16—N2 | −146.38 (13) | C39—C34—C35—C36 | 1.1 (3) |
| C14—C15—C16—N2 | −21.70 (15) | C14—C34—C35—C36 | −175.49 (16) |
| C20—C15—C16—N2 | 101.13 (13) | C34—C35—C36—C37 | 0.7 (3) |
| C17—C15—C16—C1 | −18.22 (19) | C35—C36—C37—C38 | −2.3 (3) |
| C14—C15—C16—C1 | 106.46 (15) | C35—C36—C37—F2 | 176.64 (16) |
| C20—C15—C16—C1 | −130.71 (14) | C36—C37—C38—C39 | 1.9 (3) |
| C17—C15—C16—C11 | 95.40 (13) | F2—C37—C38—C39 | −177.05 (17) |
| C14—C15—C16—C11 | −139.92 (11) | C37—C38—C39—C34 | 0.1 (3) |
| C20—C15—C16—C11 | −17.09 (13) | C35—C34—C39—C38 | −1.6 (3) |
| O2—C11—C16—N2 | −8.18 (18) | C14—C34—C39—C38 | 175.19 (16) |

*Hydrogen-bond geometry (Å, °)*

Cg5, Cg6, Cg7 and Cg9 are the centroids of the C1–C5/C10, C5–C10, C22–C27 and C34–C39 rings, respectively.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O2—H1O2···N2 | 0.90 (2) | 1.97 (2) | 2.636 (2) | 129.6 (17) |
| C13—H13A···O2$^{i}$ | 0.98 | 2.53 | 3.499 (2) | 171 |
| C20—H20B···O2$^{i}$ | 0.97 | 2.55 | 3.522 (2) | 179 |

| | | | | |
|---|---|---|---|---|
| C23—H23A···O1$^{ii}$ | 0.93 | 2.52 | 3.410 (2) | 162 |
| C35—H35A···O2$^{i}$ | 0.93 | 2.46 | 3.383 (2) | 174 |
| C30—H30A···Cg5$^{iii}$ | 0.93 | 2.94 | 3.836 (2) | 168 |
| C31—H31A···Cg6$^{iii}$ | 0.93 | 2.91 | 3.609 (3) | 133 |
| C38—H38A···Cg7$^{iv}$ | 0.93 | 2.92 | 3.836 (2) | 168 |
| C7—H7A···Cg9$^{v}$ | 0.93 | 2.85 | 3.400 (3) | 119 |

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

**Fig. 1**

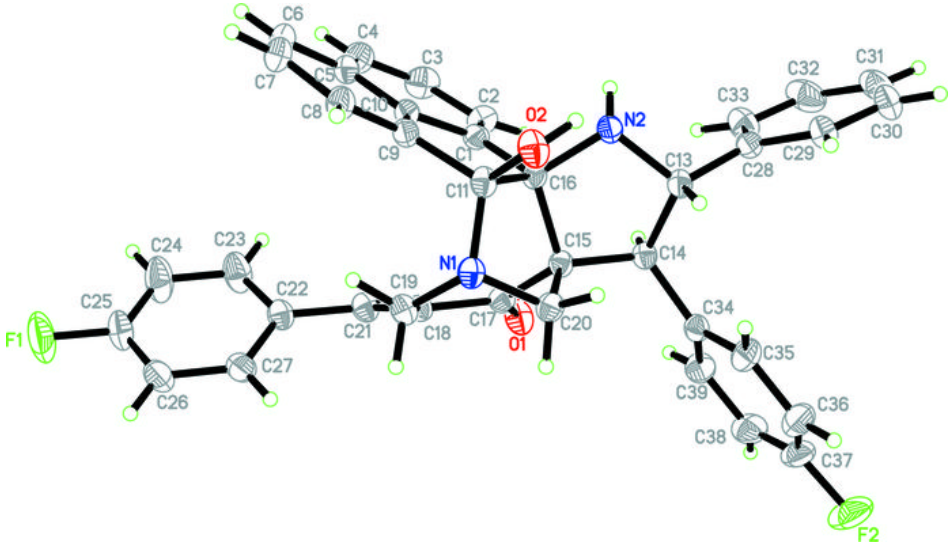
C6
C4
C3
C7
C5
C2
O2
N2
C8
C10
C9
C1
C11
C16
C13
C33
C32
C31
C28
C29
C30
C24
C23
C15
C14
N1
C22
C19
C25
C34
F1
C21
C18
C17
C20
O1
C35
C27
C39
C36
C26
C38
C37
F2

**Fig. 2**