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4-(4-Fluorophenyl)-3-methyl-1-phenylindeno-[1,2-*b*]pyrazolo[4,3-e]pyridin-5(1*H*)-one: sheets built from C—H···N, C—H···O and C—H···π(arene) hydrogen bonds

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.040
wR factor = 0.110
Data-to-parameter ratio = 15.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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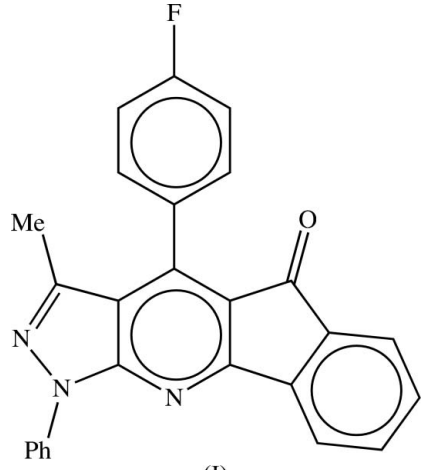
The molecules of the title compound, $C_{26}H_{16}FN_3O$, are linked into chains of edge-fused rings by a combination of C—H···N and C—H···O hydrogen bonds, and these chains are linked into sheets by a single C—H···π(arene) hydrogen bond.

Comment

We have recently reported several different methodologies for the synthesis of biologically active compounds containing pyrazolo[3,4-*b*]pyridine skeletons using 5-aminopyrazoles as starting materials (Quiroga *et al.*, 1998; Quiroga, Cruz *et al.*, 2001; Quiroga, Mejía *et al.*, 2001). We now report the molecular structure of (I) (Fig. 1) which was prepared by a three-component reaction between 5-amino-3-methyl-1-phenylpyrazole, 4-fluorobenzaldehyde and 1,3-indandione. To the best of our knowledge, this is the first reported structure of an indeno[1,2-*b*]pyrazolo[4,3-*e*]pyridine system.



(I)

The bond distances within the fused tetracyclic core of the molecule (Table 1) provide strong evidence for aromatic-type delocalization in both the aryl ring and the pyridine ring, with strong bond fixation in the pyrazole ring. The bonds C4*A*—C5, C5—C5*A* and C9*A*—C9*B* are all quite long for their types, while C5—O5 is quite short, indicating the lack of any π-delocalization or charge separation within the five-membered carbocyclic ring.

The molecules are linked into chains of edge-fused rings by two hydrogen bonds, one each of C—H···N and C—H···O types (Table 2). The aryl atom C46 in the molecule at (*x*, *y*, *z*) acts as hydrogen-bond donor to the pyridine atom N10 in the molecule at (1 − *x*, 1 − *y*, 1 − *z*), so generating by inversion an $R_2^2(14)$ (Bernstein *et al.*, 1995) motif centred at ($\frac{1}{2}$, $\frac{1}{2}$, $\frac{1}{2}$). This

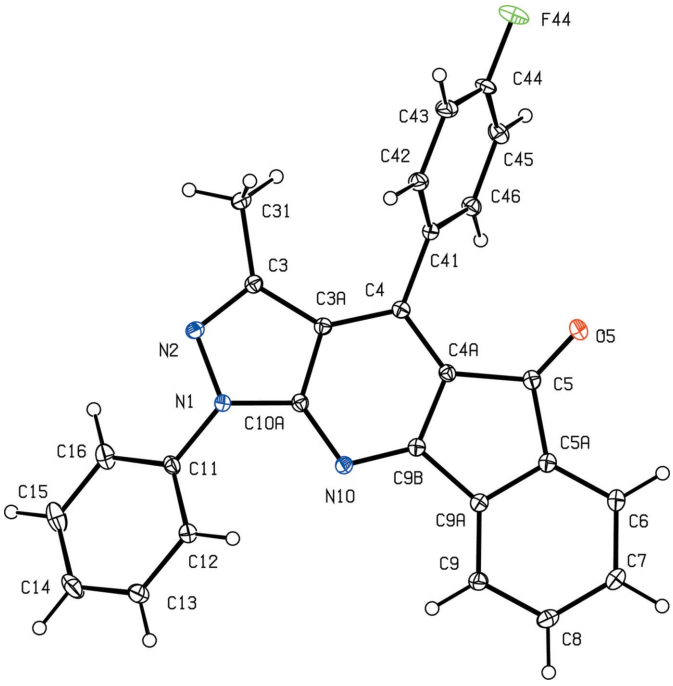


Figure 1
The molecular structure of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

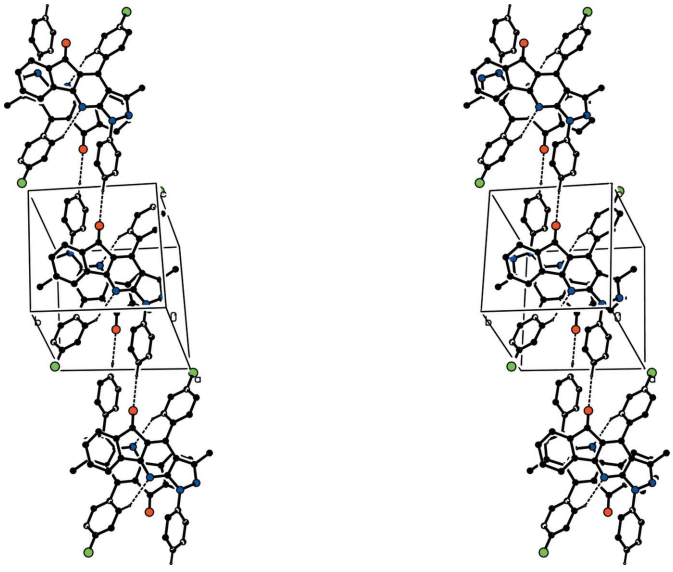


Figure 2
A stereoview of part of the crystal structure of compound (I), showing the formation of a chain of edge-fused rings along [10$\bar{1}$]. Hydrogen bonds are shown as dashed lines and, for the sake of clarity, the H atoms not involved in the motifs shown have been omitted.

dimeric motif in reinforced by a π–π stacking interaction involving the two pyridyl rings: these are strictly parallel with an interplanar spacing of 3.463 (2) Å, and a ring-centroid separation of 3.647 (2) Å, corresponding to a ring-centroid offset of 1.144 (2) Å. In addition, aryl atom C14 at (x, y, z) acts as hydrogen-bond donor to atom O5 in the molecule at ($-1+x$, y, $1+z$), so generating by translation a $C(12)$ chain running parallel to the [10$\bar{1}$] direction. The propagation by translation and inversion of these two hydrogen bonds then generates a chain of edge-fused centrosymmetric rings parallel to [10$\bar{1}$] with $R_2^2(14)$ rings centred at ($\frac{1}{2}+n$, $\frac{1}{2}$, $\frac{1}{2}-n$) (n = zero or an integer) and $R_4^4(30)$ rings centred at (n, $\frac{1}{2}$, $1-n$) (n = zero or an integer) (Fig. 2). These chains are in turn linked by a C—H$\cdots\pi$(arene) hydrogen bond (Table 2) to form sheets parallel to (101).

Experimental

A solution of 5-amino-3-methyl-1-phenylpyrazole (1 mmol), 4-fluorobenzaldehyde (1 mmol) and 1,3-indandione (1 mmol) in dimethylformamide (10 ml) containing a catalytic amount of triethylamine was heated under reflux for 7 h. The resulting solid product (I) was collected by filtration, washed with ethanol, dried and finally recrystallized from dimethylformamide to afford yellow crystals which were suitable for single-crystal X-ray diffraction [yield 54%, m.p. 528–530 K]. MS m/z (%): 406 (36), 405 (100, M^+), 404 (41), 390 [15, $(M-CH_3)^+$].

Crystal data

$C_{26}H_{16}FN_3O$
M_r = 405.42
Triclinic, $P\bar{1}$
a = 8.7566 (3) Å
b = 9.9924 (2) Å
c = 11.9100 (4) Å
α = 73.359 (2)°
β = 78.009 (2)°
γ = 81.238 (2)°
V = 971.88 (5) Å^3
Z = 2
D_x = 1.385 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 120 (2) K
Block, yellow
0.66 × 0.26 × 0.24 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.922, T_{max} = 0.978
19619 measured reflections
4446 independent reflections
3446 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 27.6°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.110
S = 1.06
4446 reflections
281 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.059P)^2 + 0.2111P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Table 1
Selected bond lengths (Å).

N1—N2	1.3800 (15)	C8—C9	1.3968 (18)
N2—C3	1.3213 (16)	C9—C9*A*	1.3856 (18)
C3—C3*A*	1.4342 (17)	C9*A*—C9*B*	1.4812 (17)
C3*A*—C4	1.4147 (16)	C9*B*—N10	1.3266 (15)
C4—C4*A*	1.3910 (17)	N10—C10*A*	1.3506 (16)
C4*A*—C5	1.4998 (17)	C10*A*—N1	1.3686 (15)
C5—C5*A*	1.4914 (18)	C3*A*—C10*A*	1.4127 (18)
C5*A*—C6	1.3868 (17)	C4*A*—C9*B*	1.4209 (17)
C6—C7	1.3917 (19)	C5*A*—C9*A*	1.3989 (17)
C7—C8	1.3905 (19)	C5—O5	1.2162 (15)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C14—H14···O5^{i}	0.95	2.48	3.4065 (19)	164
C46—H46···N10^{ii}	0.95	2.60	3.4657 (17)	152
C43—H43···Cg^{iii}	0.95	2.65	3.5060 (16)	150

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

All H atoms were located in difference maps and then treated as riding atoms, with C—H = 0.95 (aromatic) or 0.98 Å (methyl) and with $U_{iso}(H) = kU_{eq}(C)$, where $k = 1.5$ for methyl H atoms and 1.2 for all other H atoms.

Data collection: *COLLECT* (Hooft, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

X-ray data were collected at the EPSRC National X-ray Crystallography Service, University of Southampton, England. JC thanks the Consejería de Innovación, Ciencia *y* Empresa (Junta de Andalucía, Spain) and the Universidad de Jaén for financial support. DC and JQ thank COLCIENCIAS, UNIVALLE (Universidad del Valle, Colombia) for financial support.

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Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.7566$ (3) Å
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$c = 11.9100$ (4) Å
$\alpha = 73.359$ (2)°
$\beta = 78.009$ (2)°
$\gamma = 81.238$ (2)°
$V = 971.88$ (5) Å^3
$Z = 2$
$F(000) = 420$
$D_x = 1.385$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4446 reflections
$\theta = 3.2$–27.6°
$\mu = 0.09$ mm^{-1}
$T = 120$ K
Block, yellow
0.66 × 0.26 × 0.24 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Radiation source: Bruker-Nonius FR591 rotating anode
Graphite monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
$T_{min} = 0.922$, $T_{max} = 0.978$
19619 measured reflections
4446 independent reflections
3446 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 27.6°$, $\theta_{min} = 3.2°$
$h = -11 \rightarrow 11$
$k = -12 \rightarrow 12$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.110$
$S = 1.06$
4446 reflections
281 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.059P)^2 + 0.2111P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.32$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.36566 (12)	0.75883 (11)	0.65474 (9)	0.0169 (2)
C11	0.29292 (15)	0.71861 (13)	0.77628 (11)	0.0180 (3)
C12	0.38176 (16)	0.65357 (14)	0.86418 (12)	0.0236 (3)
C13	0.30792 (19)	0.61248 (15)	0.98194 (13)	0.0300 (3)
C14	0.14663 (19)	0.63630 (15)	1.01238 (14)	0.0320 (4)
C15	0.05945 (18)	0.70400 (17)	0.92378 (14)	0.0338 (4)
C16	0.13120 (16)	0.74590 (15)	0.80567 (13)	0.0270 (3)
N2	0.30324 (12)	0.87608 (11)	0.57809 (9)	0.0179 (2)
C3	0.38539 (14)	0.88295 (13)	0.47050 (11)	0.0167 (3)
C31	0.34501 (16)	1.00099 (13)	0.36834 (12)	0.0212 (3)
C3A	0.50539 (14)	0.76762 (12)	0.47334 (11)	0.0154 (3)
C4	0.61854 (14)	0.71496 (12)	0.38884 (11)	0.0153 (3)
C41	0.64260 (14)	0.78615 (13)	0.25961 (11)	0.0167 (3)
C42	0.68933 (15)	0.92146 (13)	0.21801 (12)	0.0207 (3)
C43	0.71361 (16)	0.98765 (14)	0.09729 (12)	0.0235 (3)
C44	0.68743 (16)	0.91716 (14)	0.02086 (12)	0.0227 (3)
F44	0.71087 (11)	0.98124 (9)	−0.09786 (7)	0.0363 (2)
C45	0.63971 (16)	0.78401 (14)	0.05820 (12)	0.0235 (3)
C46	0.61907 (15)	0.71766 (13)	0.17850 (11)	0.0199 (3)
C4A	0.70336 (14)	0.58952 (12)	0.43718 (11)	0.0154 (3)
C5	0.83913 (14)	0.50600 (13)	0.38070 (11)	0.0173 (3)
O5	0.90225 (11)	0.53006 (10)	0.27685 (8)	0.0248 (2)
C5A	0.88441 (14)	0.38623 (13)	0.47984 (11)	0.0171 (3)
C6	1.00240 (15)	0.27703 (13)	0.47665 (12)	0.0206 (3)
C7	1.02158 (15)	0.17763 (14)	0.58304 (13)	0.0226 (3)
C8	0.92587 (15)	0.18869 (13)	0.68953 (13)	0.0224 (3)
C9	0.80669 (15)	0.29830 (13)	0.69327 (12)	0.0196 (3)
C9A	0.78745 (14)	0.39640 (12)	0.58696 (11)	0.0164 (3)
C9B	0.67604 (14)	0.52408 (12)	0.56136 (11)	0.0152 (3)
N10	0.57050 (12)	0.56973 (10)	0.64300 (9)	0.0165 (2)
C10A	0.48785 (14)	0.69040 (12)	0.59408 (11)	0.0155 (3)
H12	0.4925	0.6372	0.8440	0.028*
H13	0.3687	0.5675	1.0423	0.036*
H14	0.0964	0.6067	1.0929	0.038*
H15	−0.0511	0.7220	0.9443	0.041*
H16	0.0706	0.7927	0.7456	0.032*
H31A	0.2392	1.0449	0.3903	0.032*
H31B	0.3487	0.9648	0.2994	0.032*
H31C	0.4206	1.0707	0.3482	0.032*
H42	0.7047	0.9688	0.2729	0.025*
H43	0.7474	1.0792	0.0683	0.028*
H45	0.6214	0.7389	0.0027	0.028*
H46	0.5887	0.6249	0.2062	0.024*
H6	1.0683	0.2703	0.4039	0.025*
H7	1.1010	0.1014	0.5829	0.027*

H8	0.9419	0.1203	0.7612	0.027*
H9	0.7412	0.3053	0.7661	0.024*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0184 (5)	0.0156 (5)	0.0149 (6)	0.0015 (4)	−0.0015 (4)	−0.0039 (4)
C11	0.0223 (6)	0.0154 (6)	0.0153 (7)	−0.0032 (5)	0.0021 (5)	−0.0059 (5)
C12	0.0264 (7)	0.0212 (7)	0.0190 (7)	0.0031 (5)	0.0002 (6)	−0.0045 (5)
C13	0.0422 (9)	0.0226 (7)	0.0183 (8)	0.0030 (6)	0.0015 (6)	−0.0024 (6)
C14	0.0424 (9)	0.0283 (8)	0.0209 (8)	−0.0105 (6)	0.0109 (6)	−0.0076 (6)
C15	0.0240 (7)	0.0453 (9)	0.0342 (9)	−0.0110 (7)	0.0087 (6)	−0.0195 (7)
C16	0.0213 (7)	0.0356 (8)	0.0264 (8)	−0.0032 (6)	−0.0004 (6)	−0.0144 (6)
N2	0.0202 (5)	0.0157 (5)	0.0171 (6)	0.0007 (4)	−0.0048 (4)	−0.0034 (4)
C3	0.0181 (6)	0.0155 (6)	0.0176 (7)	−0.0012 (5)	−0.0044 (5)	−0.0055 (5)
C31	0.0246 (7)	0.0184 (6)	0.0193 (7)	0.0031 (5)	−0.0066 (5)	−0.0033 (5)
C3A	0.0168 (6)	0.0139 (6)	0.0158 (6)	−0.0023 (5)	−0.0031 (5)	−0.0039 (5)
C4	0.0171 (6)	0.0138 (6)	0.0156 (6)	−0.0039 (5)	−0.0027 (5)	−0.0039 (5)
C41	0.0155 (6)	0.0166 (6)	0.0164 (7)	0.0003 (4)	−0.0017 (5)	−0.0038 (5)
C42	0.0256 (7)	0.0174 (6)	0.0185 (7)	−0.0013 (5)	−0.0041 (5)	−0.0042 (5)
C43	0.0291 (7)	0.0180 (6)	0.0198 (7)	−0.0044 (5)	−0.0020 (6)	0.0002 (5)
C44	0.0244 (7)	0.0282 (7)	0.0102 (7)	−0.0016 (5)	−0.0011 (5)	0.0015 (5)
F44	0.0490 (6)	0.0413 (5)	0.0142 (4)	−0.0116 (4)	−0.0043 (4)	0.0028 (4)
C45	0.0256 (7)	0.0295 (7)	0.0175 (7)	−0.0041 (6)	−0.0029 (5)	−0.0090 (6)
C46	0.0216 (7)	0.0194 (6)	0.0182 (7)	−0.0034 (5)	−0.0016 (5)	−0.0047 (5)
C4A	0.0164 (6)	0.0146 (6)	0.0149 (6)	−0.0030 (5)	−0.0008 (5)	−0.0041 (5)
C5	0.0174 (6)	0.0164 (6)	0.0188 (7)	−0.0033 (5)	−0.0014 (5)	−0.0060 (5)
O5	0.0260 (5)	0.0261 (5)	0.0182 (5)	0.0007 (4)	0.0026 (4)	−0.0057 (4)
C5A	0.0163 (6)	0.0159 (6)	0.0197 (7)	−0.0026 (5)	−0.0024 (5)	−0.0057 (5)
C6	0.0177 (6)	0.0209 (6)	0.0236 (7)	−0.0009 (5)	−0.0014 (5)	−0.0086 (5)
C7	0.0197 (6)	0.0186 (6)	0.0292 (8)	0.0025 (5)	−0.0067 (6)	−0.0064 (6)
C8	0.0220 (7)	0.0183 (6)	0.0248 (8)	−0.0005 (5)	−0.0073 (6)	−0.0006 (5)
C9	0.0194 (6)	0.0192 (6)	0.0186 (7)	−0.0018 (5)	−0.0028 (5)	−0.0028 (5)
C9A	0.0153 (6)	0.0152 (6)	0.0188 (7)	−0.0029 (5)	−0.0028 (5)	−0.0043 (5)
C9B	0.0155 (6)	0.0142 (6)	0.0168 (7)	−0.0028 (5)	−0.0027 (5)	−0.0047 (5)
N10	0.0175 (5)	0.0144 (5)	0.0169 (6)	−0.0012 (4)	−0.0021 (4)	−0.0039 (4)
C10A	0.0162 (6)	0.0151 (6)	0.0159 (6)	−0.0022 (5)	−0.0019 (5)	−0.0053 (5)

Geometric parameters (Å, º)

N1—N2	1.3800 (15)	C11—C16	1.3904 (19)
N2—C3	1.3213 (16)	C12—C13	1.3885 (19)
C3—C3A	1.4342 (17)	C12—H12	0.95
C3A—C4	1.4147 (16)	C13—C14	1.385 (2)
C4—C4A	1.3910 (17)	C13—H13	0.95
C4A—C5	1.4998 (17)	C14—C15	1.387 (2)
C5—C5A	1.4914 (18)	C14—H14	0.95
C5A—C6	1.3868 (17)	C15—C16	1.387 (2)

C6—C7	1.3917 (19)	C15—H15	0.95
C7—C8	1.3905 (19)	C16—H16	0.95
C8—C9	1.3968 (18)	C3—C31	1.4921 (18)
C9—C9A	1.3856 (18)	C31—H31A	0.98
C9A—C9B	1.4812 (17)	C31—H31B	0.98
C9B—N10	1.3266 (15)	C31—H31C	0.98
N10—C10A	1.3506 (16)	C4—C41	1.4842 (17)
C10A—N1	1.3686 (15)	C41—C42	1.3934 (17)
C3A—C10A	1.4127 (18)	C41—C46	1.3959 (17)
C4A—C9B	1.4209 (17)	C42—C43	1.3860 (19)
C5A—C9A	1.3989 (17)	C42—H42	0.95
C5—O5	1.2162 (15)	C43—C44	1.3713 (19)
C6—H6	0.95	C43—H43	0.95
C7—H7	0.95	C44—F44	1.3610 (15)
C8—H8	0.95	C44—C45	1.3769 (19)
C9—H9	0.95	C45—C46	1.3820 (18)
N1—C11	1.4228 (16)	C45—H45	0.95
C11—C12	1.3850 (19)	C46—H46	0.95

C10A—N1—N2	110.56 (10)	C44—C43—C42	118.06 (12)
C10A—N1—C11	129.34 (11)	C44—C43—H43	121.0
N2—N1—C11	119.85 (10)	C42—C43—H43	121.0
C12—C11—C16	120.41 (12)	F44—C44—C43	118.53 (12)
C12—C11—N1	120.56 (11)	F44—C44—C45	118.25 (12)
C16—C11—N1	119.03 (12)	C43—C44—C45	123.22 (12)
C11—C12—C13	119.58 (13)	C44—C45—C46	118.30 (12)
C11—C12—H12	120.2	C44—C45—H45	120.8
C13—C12—H12	120.2	C46—C45—H45	120.8
C14—C13—C12	120.70 (15)	C45—C46—C41	120.39 (12)
C14—C13—H13	119.6	C45—C46—H46	119.8
C12—C13—H13	119.6	C41—C46—H46	119.8
C13—C14—C15	119.10 (14)	C4—C4A—C9B	121.28 (11)
C13—C14—H14	120.4	C4—C4A—C5	130.78 (12)
C15—C14—H14	120.4	C9B—C4A—C5	107.77 (11)
C14—C15—C16	120.99 (14)	O5—C5—C5A	126.40 (12)
C14—C15—H15	119.5	O5—C5—C4A	128.21 (12)
C16—C15—H15	119.5	C5A—C5—C4A	105.38 (10)
C15—C16—C11	119.19 (14)	C6—C5A—C9A	120.98 (12)
C15—C16—H16	120.4	C6—C5A—C5	129.33 (12)
C11—C16—H16	120.4	C9A—C5A—C5	109.69 (11)
C3—N2—N1	107.25 (10)	C5A—C6—C7	118.15 (12)
N2—C3—C3A	110.61 (11)	C5A—C6—H6	120.9
N2—C3—C31	119.36 (11)	C7—C6—H6	120.9
C3A—C3—C31	130.02 (11)	C8—C7—C6	120.73 (12)
C3—C31—H31A	109.5	C8—C7—H7	119.6
C3—C31—H31B	109.5	C6—C7—H7	119.6
H31A—C31—H31B	109.5	C7—C8—C9	121.38 (13)
C3—C31—H31C	109.5	C7—C8—H8	119.3

H31A—C31—H31C	109.5	C9—C8—H8	119.3
H31B—C31—H31C	109.5	C9A—C9—C8	117.63 (12)
C10A—C3A—C4	118.83 (11)	C9A—C9—H9	121.2
C10A—C3A—C3	104.60 (10)	C8—C9—H9	121.2
C4—C3A—C3	136.41 (12)	C9—C9A—C5A	121.13 (12)
C4A—C4—C3A	114.14 (11)	C9—C9A—C9B	130.83 (12)
C4A—C4—C41	122.97 (11)	C5A—C9A—C9B	108.03 (11)
C3A—C4—C41	122.88 (11)	N10—C9B—C4A	126.27 (11)
C42—C41—C46	119.37 (12)	N10—C9B—C9A	124.63 (11)
C42—C41—C4	120.48 (11)	C4A—C9B—C9A	109.09 (10)
C46—C41—C4	120.15 (11)	C9B—N10—C10A	111.53 (11)
C43—C42—C41	120.63 (12)	N10—C10A—N1	125.08 (12)
C43—C42—H42	119.7	N10—C10A—C3A	127.92 (11)
C41—C42—H42	119.7	N1—C10A—C3A	106.98 (11)

C10A—N1—C11—C12	38.33 (18)	C41—C4—C4A—C5	5.9 (2)
N2—N1—C11—C12	−148.03 (12)	C4—C4A—C5—O5	−2.1 (2)
C10A—N1—C11—C16	−141.84 (13)	C9B—C4A—C5—O5	−177.21 (12)
N2—N1—C11—C16	31.80 (16)	C4—C4A—C5—C5A	176.82 (12)
C16—C11—C12—C13	1.57 (19)	C9B—C4A—C5—C5A	1.72 (12)
N1—C11—C12—C13	−178.61 (11)	O5—C5—C5A—C6	−1.4 (2)
C11—C12—C13—C14	−0.2 (2)	C4A—C5—C5A—C6	179.67 (12)
C12—C13—C14—C15	−1.0 (2)	O5—C5—C5A—C9A	178.16 (12)
C13—C14—C15—C16	1.0 (2)	C4A—C5—C5A—C9A	−0.80 (13)
C14—C15—C16—C11	0.3 (2)	C9A—C5A—C6—C7	0.08 (18)
C12—C11—C16—C15	−1.62 (19)	C5—C5A—C6—C7	179.57 (12)
N1—C11—C16—C15	178.56 (11)	C5A—C6—C7—C8	−0.63 (19)
C10A—N1—N2—C3	−0.59 (13)	C6—C7—C8—C9	0.7 (2)
C11—N1—N2—C3	−175.34 (10)	C7—C8—C9—C9A	−0.29 (19)
N1—N2—C3—C3A	0.51 (13)	C8—C9—C9A—C5A	−0.26 (18)
N1—N2—C3—C31	−179.51 (10)	C8—C9—C9A—C9B	−178.77 (12)
N2—C3—C3A—C10A	−0.26 (13)	C6—C5A—C9A—C9	0.37 (18)
C31—C3—C3A—C10A	179.77 (12)	C5—C5A—C9A—C9	−179.21 (11)
N2—C3—C3A—C4	174.82 (13)	C6—C5A—C9A—C9B	179.18 (11)
C31—C3—C3A—C4	−5.1 (2)	C5—C5A—C9A—C9B	−0.40 (13)
C10A—C3A—C4—C4A	−0.80 (16)	C4—C4A—C9B—N10	1.11 (18)
C3—C3A—C4—C4A	−175.37 (13)	C5—C4A—C9B—N10	176.77 (11)
C10A—C3A—C4—C41	178.18 (10)	C4—C4A—C9B—C9A	−177.66 (10)
C3—C3A—C4—C41	3.6 (2)	C5—C4A—C9B—C9A	−2.01 (13)
C4A—C4—C41—C42	−119.63 (13)	C9—C9A—C9B—N10	1.4 (2)
C3A—C4—C41—C42	61.48 (16)	C5A—C9A—C9B—N10	−177.27 (11)
C4A—C4—C41—C46	60.13 (16)	C9—C9A—C9B—C4A	−179.81 (12)
C3A—C4—C41—C46	−118.76 (13)	C5A—C9A—C9B—C4A	1.53 (13)
C46—C41—C42—C43	−0.49 (19)	C4A—C9B—N10—C10A	−0.10 (17)
C4—C41—C42—C43	179.28 (12)	C9A—C9B—N10—C10A	178.50 (10)
C41—C42—C43—C44	1.2 (2)	C9B—N10—C10A—N1	176.48 (11)
C42—C43—C44—F44	179.98 (12)	C9B—N10—C10A—C3A	−1.49 (17)
C42—C43—C44—C45	−0.6 (2)	N2—N1—C10A—N10	−177.90 (11)

F44—C44—C45—C46	178.65 (11)	C11—N1—C10A—N10	−3.8 (2)
C43—C44—C45—C46	−0.8 (2)	N2—N1—C10A—C3A	0.42 (13)
C44—C45—C46—C41	1.54 (19)	C11—N1—C10A—C3A	174.53 (11)
C42—C41—C46—C45	−0.91 (19)	C4—C3A—C10A—N10	2.02 (18)
C4—C41—C46—C45	179.32 (11)	C3—C3A—C10A—N10	178.15 (11)
C3A—C4—C4A—C9B	−0.56 (16)	C4—C3A—C10A—N1	−176.24 (10)
C41—C4—C4A—C9B	−179.54 (10)	C3—C3A—C10A—N1	−0.11 (12)
C3A—C4—C4A—C5	−175.09 (11)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14···O5^{i}	0.95	2.48	3.4065 (19)	164
C46—H46···N10^{ii}	0.95	2.60	3.4657 (17)	152
C43—H43···*Cg*^{iii}	0.95	2.65	3.5060 (16)	150

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