Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5-(4-Fluorophenyl)-1,3,7,9-tetramethylpyrido[2,3-*d*:6,5-*d*]dipyrimidine-2,4,6,8(1*H*,3*H*,7*H*,9*H*)-tetrone

Hosein Ghorbani and Ayoob Bazgir*

Department of Chemistry, Islamic Azad University, Dorood Branch, Dorood 688173551, Iran
Correspondence e-mail: a_bazgir@yahoo.com

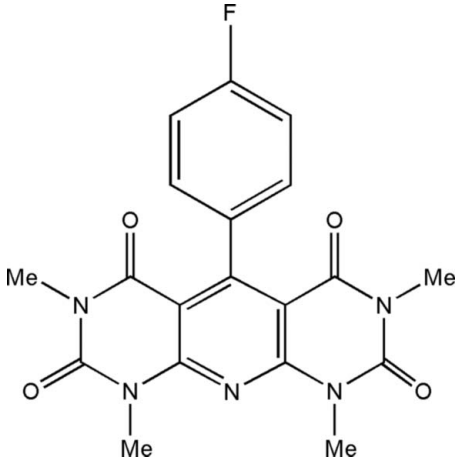
Received 13 April 2007; accepted 24 April 2007

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.005 Å; R factor = 0.098; wR factor = 0.184; data-to-parameter ratio = 15.1.

The asymmetric unit of the title compound, $C_{19}H_{16}FN_5O_4$, contains two independent molecules. The dihedral angles between the main planes and the fluorophenyl rings in the two molecules are 83.24 (5) and 71.76 (4)°. Weak π–π stacking interactions may be effective in the stabilization of the crystal structure.

Related literature

For general backgroud, see: Allen *et al.* (1987); Sanghhvi *et al.* (1989); Tenser *et al.* (2001); Nizamuddin-Mishra *et al.* (2001); Perreux & Loupy (2001). For related literature, see: Bazgir *et al.* (2006*a*,*b*).



Experimental

Crystal data

$C_{19}H_{16}FN_5O_4$
M_r = 397.37
Triclinic, $P\overline{1}$
a = 9.074 (2) Å
b = 10.930 (3) Å
c = 20.091 (5) Å
α = 75.04 (2)°
β = 76.82 (2)°
γ = 70.93 (2)°
V = 1796.8 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 294 (2) K
0.25 × 0.25 × 0.04 mm

Data collection

Stoe IPDSII diffractometer
Absorption correction: numerical shape of crystal determined optically (*X-RED32* and *X-SHAPE*; Stoe & Cie, 2005)
T_{min} = 0.950, T_{max} = 0.995
15435 measured reflections
8540 independent reflections
5359 reflections with $I > 2\sigma(I)$
R_{int} = 0.050

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.098
$wR(F^2)$ = 0.184
S = 1.23
8540 reflections
531 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Data collection: *X-AREA* (Stoe & Cie, 2005); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors acknowledge Islamic Azad University for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2233).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bazgir, A., Amani, V. & Khavasi, H. R. (2006*a*). *Acta Cryst.* E**62**, o3533–o3534.
Bazgir, A., Amani, V. & Khavasi, H. R. (2006*b*). *Acta Cryst.* E**62**, o3875–o3876.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Nizamuddin-Mishra, M., Srivastava, M. K. & Khan, M. H. (2001). *Indian J. Chem.* **40**, 49–53.
Perreux, L. & Loupy, A. (2001). *Tetrahedron*, **57**, 9199–9202.
Sanghhvi, Y. S., Larson, S. B., Matsumoto, S. S., Nord, L. D., Smee, D. F., Willis, R. C., Avery, T. H., Robins, R. K. & Revankar, G. R. (1989). *J. Med. Chem.* **32**, 629–632.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Stoe & Cie (2005). *X-AREA* (Version 1.31), *X-RED*32 (Version 1.28b) and *X-SHAPE* (Version 2.05). Stoe & Cie, Darmstadt, Germany.
Tenser, R. B., Gaydos, A. & Hay, K. A. (2001). *Antimicrob. Agents Chemother.* **45**, 3657–3660.

  doi:10.1107/S1600536807020454

supplementary materials

Acta Cryst. (2007). E**63**, o2790 [**doi:10.1107/S1600536807020454**]

5-(4-Fluorophenyl)-1,3,7,9-tetramethylpyrido[2,3-*d*:6,5-*d*]dipyrimidine-2,4,6,8(1*H*,3*H*,7*H*,9*H*)-tetrone

H. Ghorbani and A. Bazgir

Comment

Pyridopyrimidines are annelated uracils that have attracted considerable interest in recent years. Their derivatives have been known to display a wide range of pharmacological activities, such as antitumor (Sanghhvi *et al.*, 1989), antiviral (Tenser *et al.*, 2001) and antifungal (Nizamuddin-Mishra *et al.*, 2001). Therefore, for the preparation of these complex molecules large efforts have been directed towards the synthetic manipulation of uracils. As a result, a number of reports have appeared in the literature that usually describe forcing conditions, long reaction times and complex synthetic pathways. Thus, new routes for the synthesis of these molecules have attracted considerable attention allowing for a rapid entry to these heterocycles. Microwave-assisted organic synthesis is an increasingly popular field as indicated by numerous publications in the past few years owing to several advantages, such as enhanced reaction rates and increase in yields under milder conditions (Perreux & Loupy, 2001). In light of the above, we have synthesized the title compound, (I), under microwave-assisted conditions and characterized its structure.

In the molecule of (I), (Fig. 1), the bond lengths and angles are generally within normal ranges (Allen *et al.*, 1987) and may be compared with the corresponding ones in similar structures (Bazgir *et al.*, 2006*a*,b). The asymmetric unit contains two independent molecules.

Rings A (N1/N2/C87C9/C11), B (N3/C7/C8/C13/C14/C19), C (N4/N5/C14/C16/C187C19) D (C1—C6) and A' (N6/N7/C27/C28/C30/C32), B' (N8/C26/C27/C32/C33/C38), C' (N9/N10/C33/C35/C37/C38), D' (C20—C25) are, of course, planar and the dihedral angles between them are A/B = 2.62 (2), A/C = 5.08 (3), B/C = 2.48 (2) and A'/B' = 7.58 (2), A'/C' = 12.28 (3), B'/C' = 4.92 (3)°.

The weak π-π stacking interactions, involving the adjacent rings with centroid-centroid distance of 3.507 (4) %A [symmetry code: x, y + 1/2, z] may be effective in the stabilization of the crystal structure.

Experimental

6-Amino-1,3-dimethyluracil (310 mg, 2 mmol), 4-fluorobenzaldehyde (124 mg, 1 mmol) and montmorillonite K10 (400 mg) were mixed together. The reaction mixture was placed in a screw-capped vial and irradiated for 5 min with microwave irradiation (700 W). After cooling, the reaction mixture was washed with ethanol and then recrystallized from ethyl acetate to afford the pure product (yield; 210 mg, 53%, m.p. 543-545 K).

Refinement

H atoms were positioned geometrically with C—H = 0.93 and 0.96 Å, for aromatic and methyl H atoms, and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

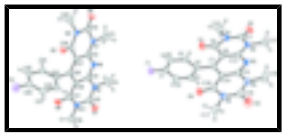


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

5-Fluorophenyl-1,3,7,9-tetramethylpyrido[2,3-d:6,5-d]dipyrimidine- 2,4,6,8(1*H*,3H,7*H*,9H)-tetrone

Crystal data

$C_{19}H_{16}FN_5O_4$	$Z = 4$
$M_r = 397.37$	$F_{000} = 824$
Triclinic, $P\overline{1}$	$D_x = 1.469$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 9.074$ (2) Å	Cell parameters from 2000 reflections
$b = 10.930$ (3) Å	$\theta = 2.0$–28.0º
$c = 20.091$ (5) Å	$\mu = 0.11$ mm^{-1}
$\alpha = 75.04$ (2)º	$T = 294$ (2) K
$\beta = 76.82$ (2)º	Plate, colorless
$\gamma = 70.93$ (2)º	0.25 × 0.25 × 0.04 mm
$V = 1796.8$ (8) Å^3	

Data collection

Stoe IPDSII diffractometer	$R_{int} = 0.050$
rotation method scans	$\theta_{max} = 28.0$º
Absorption correction: numerical shape of crystal determined optically (X-RED32 and X-SHAPE; Stoe & Cie, 2005)	$\theta_{min} = 2.0$º
$T_{min} = 0.950$, $T_{max} = 0.995$	$h = -11 \rightarrow 11$
15435 measured reflections	$k = -12 \rightarrow 14$
8540 independent reflections	$l = -26 \rightarrow 26$
5359 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0373P)^2 + 1.6536P]$ where $P = (F_o^2 + 2F_c^2)/3$
Least-squares matrix: full	$(\Delta/\sigma)_{max} = 0.096$
$R[F^2 > 2\sigma(F^2)] = 0.098$	$\Delta\rho_{max} = 0.19$ e Å^{-3}
$wR(F^2) = 0.184$	$\Delta\rho_{min} = -0.23$ e Å^{-3}
$S = 1.23$	Extinction correction: none
8540 reflections	
531 parameters	

H-atom parameters constrained

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 ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.4778 (5)	0.2016 (4)	0.2535 (2)	0.0503 (9)
C2	0.3908 (5)	0.2376 (4)	0.2011 (2)	0.0587 (11)
H2	0.3021	0.3107	0.1995	0.07*
C3	0.4381 (5)	0.1625 (4)	0.1504 (2)	0.0546 (10)
H3	0.3818	0.1858	0.1135	0.066*
C4	0.5682 (4)	0.0530 (3)	0.15386 (17)	0.0393 (8)
C5	0.6521 (4)	0.0174 (4)	0.20864 (18)	0.0442 (8)
H5	0.7394	−0.0568	0.2113	0.053*
C6	0.6056 (5)	0.0925 (4)	0.25931 (19)	0.0499 (9)
H6	0.6603	0.0695	0.2967	0.06*
C7	0.6216 (4)	−0.0241 (3)	0.09701 (17)	0.0410 (8)
C8	0.7181 (4)	0.0176 (3)	0.03454 (17)	0.0417 (8)
C9	0.7701 (5)	0.1376 (4)	0.0207 (2)	0.0496 (9)
C10	0.9087 (6)	0.2890 (4)	−0.0633 (2)	0.0691 (12)
H10A	0.9199	0.3093	−0.0213	0.083*
H10B	0.8312	0.361	−0.086	0.083*
H10C	1.0081	0.2754	−0.0939	0.083*
C11	0.9018 (5)	0.0935 (4)	−0.0969 (2)	0.0552 (10)
C12	0.9192 (6)	−0.1058 (5)	−0.1321 (2)	0.0713 (13)
H12A	0.8463	−0.0781	−0.1646	0.086*
H12B	0.9293	−0.1965	−0.1099	0.086*
H12C	1.0204	−0.0966	−0.1563	0.086*
C13	0.7668 (4)	−0.0605 (4)	−0.01636 (18)	0.0441 (8)
C14	0.6372 (4)	−0.2105 (4)	0.05081 (19)	0.0443 (8)
C15	0.6760 (7)	−0.4117 (4)	0.0055 (2)	0.0777 (15)
H15A	0.7724	−0.3925	−0.0191	0.093*
H15B	0.606	−0.3957	−0.0272	0.093*
H15C	0.6988	−0.5026	0.029	0.093*
C16	0.4980 (6)	−0.3747 (4)	0.1134 (2)	0.0607 (11)
C17	0.3346 (6)	−0.3582 (5)	0.2271 (2)	0.0679 (12)
H17A	0.2261	−0.3153	0.2217	0.081*
H17B	0.3538	−0.3429	0.2689	0.081*
H17C	0.3568	−0.4514	0.2302	0.081*
C18	0.4697 (5)	−0.1872 (4)	0.1662 (2)	0.0490 (9)
C19	0.5773 (4)	−0.1397 (3)	0.10484 (18)	0.0428 (8)
C20	0.1273 (5)	0.5408 (5)	0.7954 (2)	0.0605 (11)
C21	0.0886 (6)	0.4813 (5)	0.7527 (2)	0.0639 (11)

H21	0.0456	0.4108	0.7709	0.077*
C22	0.1154 (4)	0.5295 (4)	0.6808 (2)	0.0479 (9)
H22	0.0903	0.4904	0.6505	0.057*
C23	0.1790 (4)	0.6346 (3)	0.65424 (17)	0.0362 (7)
C24	0.2156 (4)	0.6923 (4)	0.69921 (19)	0.0467 (9)
H24	0.2581	0.7632	0.6816	0.056*
C25	0.1894 (5)	0.6452 (4)	0.7709 (2)	0.0572 (10)
H25	0.2136	0.684	0.8015	0.069*
C26	0.2175 (4)	0.6864 (3)	0.57664 (17)	0.0356 (7)
C27	0.3742 (4)	0.6615 (3)	0.54272 (17)	0.0364 (7)
C28	0.5081 (4)	0.5747 (3)	0.57779 (18)	0.0389 (8)
C29	0.7927 (4)	0.5130 (4)	0.5778 (2)	0.0572 (10)
H29A	0.7922	0.4226	0.5952	0.069*
H29B	0.7857	0.5533	0.616	0.069*
H29C	0.8888	0.5162	0.5461	0.069*
C30	0.6873 (4)	0.6504 (4)	0.4721 (2)	0.0465 (9)
C31	0.5894 (5)	0.7748 (4)	0.36256 (19)	0.0588 (11)
H31C	0.6929	0.7869	0.3519	0.071*
H31B	0.5122	0.8589	0.3532	0.071*
H31A	0.5833	0.7186	0.3342	0.071*
C32	0.4047 (4)	0.7221 (3)	0.47162 (18)	0.0386 (8)
C33	0.1436 (4)	0.8122 (3)	0.46496 (18)	0.0411 (8)
C34	0.0756 (6)	0.9241 (5)	0.3460 (2)	0.0720 (14)
H34A	0.1705	0.8604	0.3304	0.086*
H34B	0.0935	1.0094	0.3354	0.086*
H34C	−0.0077	0.9279	0.3227	0.086*
C35	−0.1298 (5)	0.9211 (4)	0.4493 (2)	0.0515 (10)
C36	−0.3408 (5)	0.9229 (4)	0.5498 (2)	0.0649 (12)
H36A	−0.3893	0.8598	0.5456	0.078*
H36B	−0.3906	1.0086	0.5246	0.078*
H36C	−0.3525	0.9259	0.5981	0.078*
C37	−0.0688 (4)	0.8092 (4)	0.5684 (2)	0.0455 (9)
C38	0.0974 (4)	0.7670 (3)	0.53688 (17)	0.0376 (7)
N1	0.8576 (4)	0.1681 (3)	−0.04580 (16)	0.0519 (8)
N2	0.8604 (4)	−0.0233 (3)	−0.07896 (16)	0.0522 (8)
N3	0.7300 (4)	−0.1727 (3)	−0.00823 (15)	0.0470 (7)
N4	0.6006 (4)	−0.3263 (3)	0.05688 (17)	0.0545 (8)
N5	0.4385 (4)	−0.3039 (3)	0.16596 (16)	0.0518 (8)
N6	0.6570 (3)	0.5847 (3)	0.54102 (16)	0.0441 (7)
N7	0.5578 (4)	0.7124 (3)	0.43777 (15)	0.0451 (7)
N8	0.2920 (4)	0.7919 (3)	0.43248 (15)	0.0428 (7)
N9	0.0297 (4)	0.8846 (3)	0.42268 (16)	0.0483 (8)
N10	−0.1716 (3)	0.8834 (3)	0.52074 (17)	0.0471 (7)
O1	0.7445 (4)	0.2088 (3)	0.06135 (16)	0.0782 (10)
O2	0.9736 (4)	0.1306 (3)	−0.15385 (15)	0.0748 (9)
O3	0.4617 (5)	−0.4732 (3)	0.11551 (19)	0.0890 (11)
O4	0.4062 (4)	−0.1330 (3)	0.21484 (15)	0.0713 (9)
O5	0.4976 (3)	0.4989 (3)	0.63411 (14)	0.0533 (7)
O6	0.8207 (3)	0.6520 (3)	0.44300 (15)	0.0611 (8)

O7	−0.2280 (3)	0.9851 (3)	0.41175 (16)	0.0687 (9)
O8	−0.1205 (3)	0.7855 (3)	0.63026 (15)	0.0720 (9)
F1	0.4364 (3)	0.2782 (3)	0.30224 (13)	0.0746 (8)
F2	0.1014 (4)	0.4928 (4)	0.86628 (13)	0.1061 (11)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.059 (2)	0.051 (2)	0.050 (2)	−0.0282 (19)	0.0049 (19)	−0.0228 (18)
C2	0.056 (3)	0.053 (2)	0.066 (3)	−0.0056 (19)	−0.012 (2)	−0.022 (2)
C3	0.061 (3)	0.055 (2)	0.049 (2)	−0.0068 (19)	−0.025 (2)	−0.0112 (18)
C4	0.046 (2)	0.0394 (18)	0.0365 (17)	−0.0160 (15)	−0.0070 (15)	−0.0087 (14)
C5	0.048 (2)	0.0452 (19)	0.0410 (19)	−0.0128 (16)	−0.0117 (16)	−0.0084 (16)
C6	0.063 (3)	0.057 (2)	0.0408 (19)	−0.031 (2)	−0.0129 (18)	−0.0079 (17)
C7	0.044 (2)	0.047 (2)	0.0350 (17)	−0.0122 (15)	−0.0124 (15)	−0.0086 (15)
C8	0.043 (2)	0.048 (2)	0.0358 (18)	−0.0124 (16)	−0.0086 (15)	−0.0096 (15)
C9	0.052 (2)	0.053 (2)	0.044 (2)	−0.0180 (18)	−0.0062 (18)	−0.0072 (18)
C10	0.070 (3)	0.062 (3)	0.068 (3)	−0.030 (2)	0.000 (2)	0.004 (2)
C11	0.042 (2)	0.066 (3)	0.047 (2)	−0.0083 (19)	−0.0033 (18)	−0.006 (2)
C12	0.067 (3)	0.092 (4)	0.056 (3)	−0.020 (3)	0.007 (2)	−0.033 (3)
C13	0.041 (2)	0.053 (2)	0.0381 (18)	−0.0092 (16)	−0.0122 (16)	−0.0084 (16)
C14	0.050 (2)	0.0427 (19)	0.043 (2)	−0.0110 (16)	−0.0190 (17)	−0.0065 (16)
C15	0.112 (4)	0.058 (3)	0.072 (3)	−0.022 (3)	−0.015 (3)	−0.031 (2)
C16	0.082 (3)	0.048 (2)	0.059 (3)	−0.023 (2)	−0.025 (2)	−0.005 (2)
C17	0.086 (3)	0.063 (3)	0.062 (3)	−0.039 (2)	−0.017 (2)	0.003 (2)
C18	0.063 (3)	0.047 (2)	0.044 (2)	−0.0243 (18)	−0.0137 (18)	−0.0045 (17)
C19	0.050 (2)	0.0444 (19)	0.0366 (18)	−0.0132 (16)	−0.0136 (16)	−0.0078 (15)
C20	0.074 (3)	0.075 (3)	0.037 (2)	−0.036 (2)	−0.006 (2)	−0.001 (2)
C21	0.076 (3)	0.069 (3)	0.056 (2)	−0.045 (2)	−0.005 (2)	0.000 (2)
C22	0.050 (2)	0.053 (2)	0.048 (2)	−0.0232 (17)	−0.0095 (17)	−0.0095 (18)
C23	0.0302 (16)	0.0394 (17)	0.0369 (17)	−0.0058 (13)	−0.0087 (14)	−0.0062 (14)
C24	0.050 (2)	0.049 (2)	0.048 (2)	−0.0201 (17)	−0.0113 (17)	−0.0097 (17)
C25	0.064 (3)	0.069 (3)	0.046 (2)	−0.025 (2)	−0.011 (2)	−0.017 (2)
C26	0.0379 (18)	0.0311 (16)	0.0408 (18)	−0.0106 (13)	−0.0107 (15)	−0.0072 (14)
C27	0.0343 (18)	0.0362 (17)	0.0389 (17)	−0.0080 (13)	−0.0095 (14)	−0.0070 (14)
C28	0.0352 (18)	0.0355 (17)	0.0450 (19)	−0.0046 (14)	−0.0108 (15)	−0.0092 (15)
C29	0.034 (2)	0.066 (3)	0.067 (3)	−0.0052 (18)	−0.0152 (19)	−0.011 (2)
C30	0.039 (2)	0.047 (2)	0.056 (2)	−0.0112 (16)	−0.0043 (18)	−0.0194 (18)
C31	0.058 (3)	0.062 (3)	0.046 (2)	−0.019 (2)	0.0027 (19)	−0.0015 (19)
C32	0.0396 (19)	0.0370 (17)	0.0424 (18)	−0.0098 (14)	−0.0080 (15)	−0.0133 (15)
C33	0.045 (2)	0.0364 (18)	0.0447 (19)	−0.0074 (15)	−0.0187 (16)	−0.0076 (15)
C34	0.081 (3)	0.078 (3)	0.047 (2)	−0.007 (3)	−0.027 (2)	−0.001 (2)
C35	0.053 (2)	0.047 (2)	0.058 (2)	−0.0073 (18)	−0.028 (2)	−0.0092 (18)
C36	0.039 (2)	0.071 (3)	0.082 (3)	−0.004 (2)	−0.022 (2)	−0.015 (2)
C37	0.0374 (19)	0.048 (2)	0.050 (2)	−0.0067 (16)	−0.0126 (17)	−0.0100 (17)
C38	0.0356 (18)	0.0374 (17)	0.0412 (18)	−0.0079 (14)	−0.0131 (15)	−0.0072 (15)
N1	0.0486 (19)	0.0530 (19)	0.0480 (18)	−0.0177 (15)	−0.0026 (15)	0.0000 (15)
N2	0.0476 (19)	0.064 (2)	0.0433 (17)	−0.0147 (16)	−0.0018 (15)	−0.0147 (16)

N3	0.0503 (19)	0.0531 (18)	0.0411 (16)	−0.0118 (15)	−0.0114 (14)	−0.0155 (14)
N4	0.071 (2)	0.0483 (18)	0.0509 (19)	−0.0167 (16)	−0.0167 (17)	−0.0157 (15)
N5	0.065 (2)	0.0492 (18)	0.0453 (18)	−0.0252 (16)	−0.0122 (16)	−0.0011 (15)
N6	0.0321 (15)	0.0491 (17)	0.0493 (17)	−0.0042 (13)	−0.0113 (13)	−0.0118 (14)
N7	0.0413 (17)	0.0483 (17)	0.0429 (16)	−0.0133 (13)	−0.0017 (14)	−0.0078 (14)
N8	0.0450 (18)	0.0425 (16)	0.0392 (15)	−0.0093 (13)	−0.0102 (14)	−0.0062 (13)
N9	0.0501 (19)	0.0480 (18)	0.0450 (17)	−0.0068 (14)	−0.0207 (14)	−0.0042 (14)
N10	0.0335 (16)	0.0469 (17)	0.0596 (19)	−0.0033 (13)	−0.0192 (14)	−0.0088 (15)
O1	0.114 (3)	0.077 (2)	0.0609 (19)	−0.061 (2)	0.0169 (18)	−0.0278 (17)
O2	0.065 (2)	0.092 (2)	0.0536 (18)	−0.0229 (17)	0.0123 (15)	−0.0082 (17)
O3	0.135 (3)	0.064 (2)	0.087 (2)	−0.055 (2)	−0.015 (2)	−0.0165 (18)
O4	0.090 (2)	0.078 (2)	0.0577 (18)	−0.0478 (18)	0.0144 (16)	−0.0253 (16)
O5	0.0413 (15)	0.0524 (15)	0.0547 (16)	−0.0080 (12)	−0.0133 (12)	0.0070 (13)
O6	0.0390 (15)	0.0728 (19)	0.0664 (18)	−0.0159 (13)	0.0013 (13)	−0.0137 (15)
O7	0.0583 (18)	0.0715 (19)	0.0726 (19)	−0.0025 (15)	−0.0401 (16)	−0.0027 (16)
O8	0.0363 (15)	0.107 (3)	0.0541 (18)	−0.0014 (15)	−0.0083 (13)	−0.0079 (17)
F1	0.0872 (19)	0.0824 (18)	0.0699 (16)	−0.0328 (15)	0.0044 (14)	−0.0452 (14)
F2	0.150 (3)	0.144 (3)	0.0419 (14)	−0.089 (2)	−0.0125 (16)	0.0096 (16)

Geometric parameters (Å, °)

C1—F1	1.360 (4)	C20—C21	1.362 (6)
C1—C2	1.361 (5)	C20—C25	1.365 (6)
C1—C6	1.366 (6)	C20—F2	1.379 (4)
C2—C3	1.377 (5)	C21—C22	1.398 (5)
C2—H2	0.93	C21—H21	0.93
C3—C4	1.379 (5)	C22—C23	1.383 (5)
C3—H3	0.93	C22—H22	0.93
C4—C5	1.382 (4)	C23—C24	1.376 (5)
C4—C7	1.493 (5)	C23—C26	1.516 (5)
C5—C6	1.378 (5)	C24—C25	1.392 (5)
C5—H5	0.93	C24—H24	0.93
C6—H6	0.93	C25—H25	0.93
C7—C19	1.408 (5)	C26—C27	1.401 (5)
C7—C8	1.419 (5)	C26—C38	1.417 (4)
C8—C13	1.403 (5)	C27—C32	1.417 (5)
C8—C9	1.474 (5)	C27—C28	1.471 (4)
C9—O1	1.205 (4)	C28—O5	1.222 (4)
C9—N1	1.409 (5)	C28—N6	1.408 (5)
C10—N1	1.473 (5)	C29—N6	1.470 (4)
C10—H10A	0.96	C29—H29A	0.96
C10—H10B	0.96	C29—H29B	0.96
C10—H10C	0.96	C29—H29C	0.96
C11—O2	1.221 (5)	C30—O6	1.222 (4)
C11—N1	1.382 (5)	C30—N7	1.388 (5)
C11—N2	1.384 (5)	C30—N6	1.396 (5)
C12—N2	1.469 (5)	C31—N7	1.493 (5)
C12—H12A	0.96	C31—H31C	0.96
C12—H12B	0.96	C31—H31B	0.96

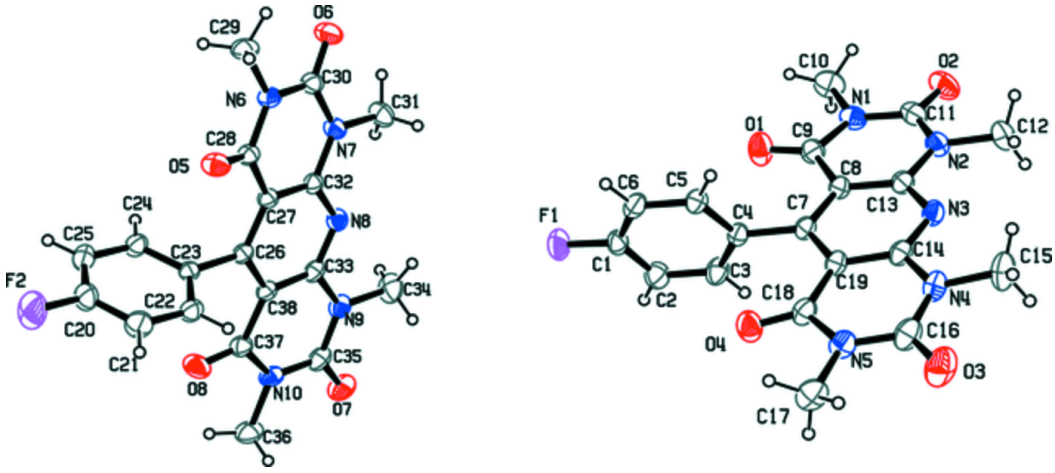
C12—H12C	0.96	C31—H31A	0.96
C13—N3	1.334 (5)	C32—N8	1.339 (4)
C13—N2	1.394 (5)	C32—N7	1.383 (4)
C14—N3	1.344 (5)	C33—N8	1.329 (5)
C14—N4	1.379 (5)	C33—N9	1.391 (4)
C14—C19	1.401 (5)	C33—C38	1.414 (5)
C15—N4	1.472 (5)	C34—N9	1.488 (5)
C15—H15A	0.96	C34—H34A	0.96
C15—H15B	0.96	C34—H34B	0.96
C15—H15C	0.96	C34—H34C	0.96
C16—O3	1.213 (5)	C35—O7	1.216 (4)
C16—N5	1.375 (5)	C35—N9	1.386 (5)
C16—N4	1.403 (6)	C35—N10	1.386 (5)
C17—N5	1.487 (5)	C36—N10	1.474 (5)
C17—H17A	0.96	C36—H36A	0.96
C17—H17B	0.96	C36—H36B	0.96
C17—H17C	0.96	C36—H36C	0.96
C18—O4	1.208 (4)	C37—O8	1.216 (4)
C18—N5	1.395 (5)	C37—N10	1.399 (4)
C18—C19	1.481 (5)	C37—C38	1.466 (5)
F1—C1—C2	118.6 (4)	C23—C24—H24	119.8
F1—C1—C6	118.4 (4)	C25—C24—H24	119.8
C2—C1—C6	123.0 (3)	C20—C25—C24	118.7 (4)
C1—C2—C3	117.9 (4)	C20—C25—H25	120.7
C1—C2—H2	121	C24—C25—H25	120.7
C3—C2—H2	121	C27—C26—C38	117.9 (3)
C2—C3—C4	120.6 (3)	C27—C26—C23	120.8 (3)
C2—C3—H3	119.7	C38—C26—C23	121.3 (3)
C4—C3—H3	119.7	C26—C27—C32	118.5 (3)
C3—C4—C5	120.0 (3)	C26—C27—C28	122.7 (3)
C3—C4—C7	119.9 (3)	C32—C27—C28	118.7 (3)
C5—C4—C7	120.0 (3)	O5—C28—N6	120.3 (3)
C6—C5—C4	119.6 (3)	O5—C28—C27	125.3 (3)
C6—C5—H5	120.2	N6—C28—C27	114.4 (3)
C4—C5—H5	120.2	N6—C29—H29A	109.5
C1—C6—C5	118.7 (3)	N6—C29—H29B	109.5
C1—C6—H6	120.6	H29A—C29—H29B	109.5
C5—C6—H6	120.6	N6—C29—H29C	109.5
C19—C7—C8	119.1 (3)	H29A—C29—H29C	109.5
C19—C7—C4	120.4 (3)	H29B—C29—H29C	109.5
C8—C7—C4	120.5 (3)	O6—C30—N7	121.5 (4)
C13—C8—C7	117.8 (3)	O6—C30—N6	122.1 (3)
C13—C8—C9	118.6 (3)	N7—C30—N6	116.4 (3)
C7—C8—C9	123.6 (3)	N7—C31—H31C	109.5
O1—C9—N1	118.8 (4)	N7—C31—H31B	109.5
O1—C9—C8	125.3 (4)	H31C—C31—H31B	109.5
N1—C9—C8	115.9 (3)	N7—C31—H31A	109.5
N1—C10—H10A	109.5	H31C—C31—H31A	109.5
N1—C10—H10B	109.5	H31B—C31—H31A	109.5

H10A—C10—H10B	109.5	N8—C32—N7	115.2 (3)
N1—C10—H10C	109.5	N8—C32—C27	124.0 (3)
H10A—C10—H10C	109.5	N7—C32—C27	120.8 (3)
H10B—C10—H10C	109.5	N8—C33—N9	115.0 (3)
O2—C11—N1	120.6 (4)	N8—C33—C38	125.1 (3)
O2—C11—N2	123.4 (4)	N9—C33—C38	119.9 (3)
N1—C11—N2	115.9 (4)	N9—C34—H34A	109.5
N2—C12—H12A	109.5	N9—C34—H34B	109.5
N2—C12—H12B	109.5	H34A—C34—H34B	109.5
H12A—C12—H12B	109.5	N9—C34—H34C	109.5
N2—C12—H12C	109.5	H34A—C34—H34C	109.5
H12A—C12—H12C	109.5	H34B—C34—H34C	109.5
H12B—C12—H12C	109.5	O7—C35—N9	121.3 (4)
N3—C13—N2	116.5 (3)	O7—C35—N10	121.8 (4)
N3—C13—C8	123.4 (3)	N9—C35—N10	116.9 (3)
N2—C13—C8	120.1 (4)	N10—C36—H36A	109.5
N3—C14—N4	116.5 (3)	N10—C36—H36B	109.5
N3—C14—C19	124.0 (3)	H36A—C36—H36B	109.5
N4—C14—C19	119.6 (4)	N10—C36—H36C	109.5
N4—C15—H15A	109.5	H36A—C36—H36C	109.5
N4—C15—H15B	109.5	H36B—C36—H36C	109.5
H15A—C15—H15B	109.5	O8—C37—N10	119.8 (3)
N4—C15—H15C	109.5	O8—C37—C38	125.8 (3)
H15A—C15—H15C	109.5	N10—C37—C38	114.4 (3)
H15B—C15—H15C	109.5	C33—C38—C26	117.6 (3)
O3—C16—N5	121.6 (5)	C33—C38—C37	120.0 (3)
O3—C16—N4	121.6 (4)	C26—C38—C37	122.3 (3)
N5—C16—N4	116.8 (4)	C11—N1—C9	125.6 (4)
N5—C17—H17A	109.5	C11—N1—C10	116.2 (4)
N5—C17—H17B	109.5	C9—N1—C10	118.2 (3)
H17A—C17—H17B	109.5	C11—N2—C13	123.5 (3)
N5—C17—H17C	109.5	C11—N2—C12	114.9 (4)
H17A—C17—H17C	109.5	C13—N2—C12	121.5 (4)
H17B—C17—H17C	109.5	C13—N3—C14	118.3 (3)
O4—C18—N5	118.2 (4)	C14—N4—C16	123.2 (3)
O4—C18—C19	125.8 (4)	C14—N4—C15	121.2 (4)
N5—C18—C19	116.0 (3)	C16—N4—C15	115.5 (4)
C14—C19—C7	117.4 (3)	C16—N5—C18	125.0 (4)
C14—C19—C18	119.4 (3)	C16—N5—C17	116.7 (4)
C7—C19—C18	123.2 (3)	C18—N5—C17	118.3 (3)
C21—C20—C25	122.9 (4)	C30—N6—C28	125.8 (3)
C21—C20—F2	117.9 (4)	C30—N6—C29	117.7 (3)
C25—C20—F2	119.2 (4)	C28—N6—C29	116.4 (3)
C20—C21—C22	117.9 (4)	C32—N7—C30	122.2 (3)
C20—C21—H21	121	C32—N7—C31	120.6 (3)
C22—C21—H21	121	C30—N7—C31	117.1 (3)
C23—C22—C21	120.7 (3)	C33—N8—C32	116.6 (3)
C23—C22—H22	119.7	C35—N9—C33	122.1 (3)
C21—C22—H22	119.7	C35—N9—C34	117.3 (3)

C24—C23—C22	119.5 (3)	C33—N9—C34	120.6 (3)
C24—C23—C26	117.7 (3)	C35—N10—C37	126.4 (3)
C22—C23—C26	122.7 (3)	C35—N10—C36	117.4 (3)
C23—C24—C25	120.3 (4)	C37—N10—C36	116.2 (3)
F1—C1—C2—C3	177.2 (4)	O8—C37—C38—C26	2.9 (6)
C6—C1—C2—C3	−2.3 (7)	N10—C37—C38—C26	−177.3 (3)
C1—C2—C3—C4	1.1 (7)	O2—C11—N1—C9	177.7 (4)
C2—C3—C4—C5	0.2 (6)	N2—C11—N1—C9	−2.7 (5)
C2—C3—C4—C7	−177.6 (4)	O2—C11—N1—C10	−2.8 (6)
C3—C4—C5—C6	−0.5 (6)	N2—C11—N1—C10	176.9 (3)
C7—C4—C5—C6	177.3 (3)	O1—C9—N1—C11	177.3 (4)
F1—C1—C6—C5	−177.5 (3)	C8—C9—N1—C11	−2.2 (5)
C2—C1—C6—C5	2.0 (6)	O1—C9—N1—C10	−2.3 (6)
C4—C5—C6—C1	−0.6 (6)	C8—C9—N1—C10	178.2 (3)
C3—C4—C7—C19	−97.7 (4)	O2—C11—N2—C13	−174.3 (4)
C5—C4—C7—C19	84.5 (4)	N1—C11—N2—C13	6.1 (5)
C3—C4—C7—C8	82.6 (5)	O2—C11—N2—C12	4.2 (6)
C5—C4—C7—C8	−95.3 (4)	N1—C11—N2—C12	−175.5 (3)
C19—C7—C8—C13	−0.9 (5)	N3—C13—N2—C11	176.5 (3)
C4—C7—C8—C13	178.8 (3)	C8—C13—N2—C11	−4.4 (5)
C19—C7—C8—C9	179.4 (3)	N3—C13—N2—C12	−1.9 (5)
C4—C7—C8—C9	−0.8 (5)	C8—C13—N2—C12	177.3 (3)
C13—C8—C9—O1	−175.5 (4)	N2—C13—N3—C14	−178.6 (3)
C7—C8—C9—O1	4.2 (6)	C8—C13—N3—C14	2.2 (5)
C13—C8—C9—N1	4.0 (5)	N4—C14—N3—C13	179.4 (3)
C7—C8—C9—N1	−176.4 (3)	C19—C14—N3—C13	−0.5 (5)
C7—C8—C13—N3	−1.5 (5)	N3—C14—N4—C16	−176.3 (3)
C9—C8—C13—N3	178.1 (3)	C19—C14—N4—C16	3.6 (5)
C7—C8—C13—N2	179.4 (3)	N3—C14—N4—C15	7.6 (5)
C9—C8—C13—N2	−1.0 (5)	C19—C14—N4—C15	−172.6 (4)
N3—C14—C19—C7	−1.8 (5)	O3—C16—N4—C14	176.1 (4)
N4—C14—C19—C7	178.3 (3)	N5—C16—N4—C14	−2.9 (6)
N3—C14—C19—C18	177.0 (3)	O3—C16—N4—C15	−7.5 (6)
N4—C14—C19—C18	−2.8 (5)	N5—C16—N4—C15	173.5 (4)
C8—C7—C19—C14	2.5 (5)	O3—C16—N5—C18	−177.4 (4)
C4—C7—C19—C14	−177.3 (3)	N4—C16—N5—C18	1.6 (6)
C8—C7—C19—C18	−176.4 (3)	O3—C16—N5—C17	3.2 (6)
C4—C7—C19—C18	3.9 (5)	N4—C16—N5—C17	−177.8 (3)
O4—C18—C19—C14	−177.4 (4)	O4—C18—N5—C16	178.0 (4)
N5—C18—C19—C14	1.5 (5)	C19—C18—N5—C16	−0.9 (5)
O4—C18—C19—C7	1.5 (6)	O4—C18—N5—C17	−2.7 (5)
N5—C18—C19—C7	−179.7 (3)	C19—C18—N5—C17	178.4 (3)
C25—C20—C21—C22	0.6 (7)	O6—C30—N6—C28	−178.1 (3)
F2—C20—C21—C22	−179.7 (4)	N7—C30—N6—C28	0.9 (5)
C20—C21—C22—C23	−0.2 (6)	O6—C30—N6—C29	−1.7 (5)
C21—C22—C23—C24	−0.2 (6)	N7—C30—N6—C29	177.3 (3)
C21—C22—C23—C26	177.1 (4)	O5—C28—N6—C30	169.1 (3)
C22—C23—C24—C25	0.2 (5)	C27—C28—N6—C30	−11.1 (5)
C26—C23—C24—C25	−177.2 (3)	O5—C28—N6—C29	−7.4 (5)

C21—C20—C25—C24	−0.6 (7)	C27—C28—N6—C29	172.4 (3)
F2—C20—C25—C24	179.7 (4)	N8—C32—N7—C30	176.0 (3)
C23—C24—C25—C20	0.2 (6)	C27—C32—N7—C30	−3.8 (5)
C24—C23—C26—C27	71.1 (4)	N8—C32—N7—C31	−1.1 (5)
C22—C23—C26—C27	−106.2 (4)	C27—C32—N7—C31	179.1 (3)
C24—C23—C26—C38	−105.6 (4)	O6—C30—N7—C32	−173.9 (3)
C22—C23—C26—C38	77.1 (4)	N6—C30—N7—C32	7.1 (5)
C38—C26—C27—C32	2.5 (4)	O6—C30—N7—C31	3.4 (5)
C23—C26—C27—C32	−174.3 (3)	N6—C30—N7—C31	−175.7 (3)
C38—C26—C27—C28	−177.3 (3)	N9—C33—N8—C32	−179.8 (3)
C23—C26—C27—C28	5.9 (5)	C38—C33—N8—C32	0.6 (5)
C26—C27—C28—O5	13.4 (5)	N7—C32—N8—C33	−174.7 (3)
C32—C27—C28—O5	−166.4 (3)	C27—C32—N8—C33	5.1 (5)
C26—C27—C28—N6	−166.4 (3)	O7—C35—N9—C33	179.6 (4)
C32—C27—C28—N6	13.8 (4)	N10—C35—N9—C33	0.8 (5)
C26—C27—C32—N8	−6.8 (5)	O7—C35—N9—C34	−1.5 (6)
C28—C27—C32—N8	173.0 (3)	N10—C35—N9—C34	179.7 (3)
C26—C27—C32—N7	173.0 (3)	N8—C33—N9—C35	−176.0 (3)
C28—C27—C32—N7	−7.2 (5)	C38—C33—N9—C35	3.6 (5)
N8—C33—C38—C26	−4.5 (5)	N8—C33—N9—C34	5.1 (5)
N9—C33—C38—C26	176.0 (3)	C38—C33—N9—C34	−175.3 (4)
N8—C33—C38—C37	172.5 (3)	O7—C35—N10—C37	179.5 (4)
N9—C33—C38—C37	−7.1 (5)	N9—C35—N10—C37	−1.7 (6)
C27—C26—C38—C33	2.6 (4)	O7—C35—N10—C36	0.6 (6)
C23—C26—C38—C33	179.4 (3)	N9—C35—N10—C36	179.4 (3)
C27—C26—C38—C37	−174.3 (3)	O8—C37—N10—C35	178.2 (4)
C23—C26—C38—C37	2.5 (5)	C38—C37—N10—C35	−1.6 (5)
O8—C37—C38—C33	−173.9 (4)	O8—C37—N10—C36	−2.9 (5)
N10—C37—C38—C33	5.9 (5)	C38—C37—N10—C36	177.3 (3)

Fig. 1


O6
C29
C30
N6
C31
N7
C28
O5
C32
C27
N8
C24
C25
C23
C26
C33
F2
C22
C34
C38
N9
C20
C37
C35
C21
O8
N10
O7
C36
C10
O2
N1
C11
O1
C9
C12
N2
C8
C6
C5
C7
C13
N3
F1
C4
C14
C15
C1
C3
C19
N4
C2
C18
C16
O4
N5
O3
C17