

## 7-(4-Methoxyphenyl)-5-methyl-9-phenyl-7H-pyrrolo[2',3':4,5]pyrimido[1,6-d]-tetrazole

Mukesh M. Jotani,<sup>a</sup> Rina D. Shah<sup>b</sup> and Jerry P. Jasinski<sup>c\*</sup>

<sup>a</sup>Bhavan's Sheth R.A. College of Science, Ahmedabd, Gujarat 380 001, India, <sup>b</sup>M. G.

Science Institute, Navrangpura, Ahmedabad, Gujarat 380 009, India, and

<sup>c</sup>Department of Chemistry, Keene State College, 229 Main Street, Keene,

NH 03435-2001, USA

Correspondence e-mail: jjasinski@keene.edu

Received 25 October 2009; accepted 13 December 2009

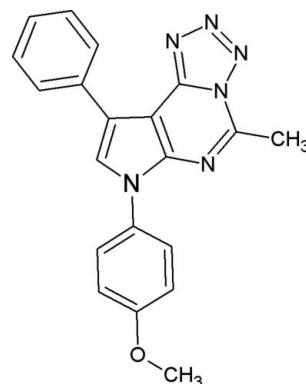
Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å; R factor = 0.045; wR factor = 0.129; data-to-parameter ratio = 20.8.

The title compound,  $\text{C}_{20}\text{H}_{16}\text{N}_6\text{O}$ , is composed of a tetrazolo ring and a 4-methoxyphenyl and a benzene-substituted pyrrole ring at the 7 and 9 positions fused to a pyrimidine ring in a nearly planar fashion [maximum deviation of 0.018 (1) Å for the fused ring system]. A methyl group at the 5 position is also in the plane of the hetero cyclic system. The dihedral angle between the mean planes of the benzene and 4-methoxyphenyl rings is 40.4 (2)°. The dihedral angles between the mean planes of the pyrimidine and the benzene and 4-methoxyphenyl rings are 15.6 (5)° and 52.6 (7)°, respectively. A weak intramolecular C—H···N hydrogen bond interaction, which forms an  $S(7)$  graph-set motif, helps to establish the relative conformations of the tetrazolo and benzene rings. In the crystal, weak intermolecular C—H···O, C—H··· $\pi$  and  $\pi$ — $\pi$  stacking interactions [centroid—centroid distances = 3.5270 (16), 3.5113 (16), 3.7275 (17) and 3.7866 (17) Å] link the molecules into a two-dimensional array obliquely parallel to (101) and propagating along the  $b$  axis.

### Related literature

For the biological activity of fused tetrazolopyrimidines, see: Wilkinson (1992); Omer *et al.* (1991); Schram *et al.* (1975). Fused pyrimidines with a halogen at the 2- or 4- position seem to be more labile towards a nucleophilic substitution reaction with reagents such as piperadine, piperazine, morpholine, hydrazine and azides, forming potent bi- and triheterocycles, see: Dave & Shah (2000, 2002); Peinador *et al.* (1992); Schneller & Clough (1992); Shishoo & Jain (1992). For the importance of the reduction of tetrazolopyrimidines *via* azidolysis in the development of synthetically important 4-aminopyrimidines, see: Shishoo & Jain (1992); Hand & Backer (1984). For nucleophilic substitution reactions in pyrrolo-

[2,3-*e*] pyrimidines, see: Dave & Shah (2002); Ali & Swealan (1992). For related structures, see: Jotani & Baldaniya (2007, 2008); Hou *et al.* (2009); Baldaniya & Jotani (2008); Malone *et al.* (1997). For the synthesis, see: Shah (2009). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For tetrazolo ring formation, see: Bourgurgnon *et al.* (1975); Robba *et al.* (1975).



### Experimental

#### Crystal data

$\text{C}_{20}\text{H}_{16}\text{N}_6\text{O}$	$V = 1761.9$ (8) Å <sup>3</sup>
$M_r = 356.39$	$Z = 4$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 13.738$ (2) Å	$\mu = 0.09$ mm <sup>-1</sup>
$b = 7.032$ (3) Å	$T = 293$ K
$c = 19.4350$ (3) Å	$0.47 \times 0.35 \times 0.2$ mm
$\beta = 110.217$ (2)°	

#### Data collection

Bruker Kappa APEXII CCD diffractometer	42323 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)	5137 independent reflections
$T_{\min} = 0.96$ , $T_{\max} = 0.98$	3694 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.030$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$	247 parameters
$wR(F^2) = 0.129$	H-atom parameters constrained
$S = 1.04$	$\Delta\rho_{\text{max}} = 0.29$ e Å <sup>-3</sup>
5137 reflections	$\Delta\rho_{\text{min}} = -0.20$ e Å <sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C13—H13···N6	0.95	2.38	3.2159 (18)	146
C12—H12···O1 <sup>i</sup>	0.95	2.57	3.3902 (17)	144
C7—H7B···Cg4 <sup>ii</sup>	0.96	2.90	3.620 (2)	132
C7—H7C···Cg4 <sup>iii</sup>	0.96	2.69	3.554 (2)	150

Symmetry codes: (i)  $x - \frac{1}{2}, -y + \frac{3}{2}, z + \frac{1}{2}$ ; (ii)  $-x + 1, -y + 1, -z + 1$ ; (iii)  $-x + 1, -y + 2, -z + 1$ . Cg4 is the centroid of the C8—C13 ring.

Data collection: APEX2 (Bruker, 2004); cell refinement: APEX2 and SAINT (Bruker, 2004); data reduction: SAINT and XPREP (Bruker, 2004); program(s) used to solve structure: SIR2004 (Burla *et al.*, 2003); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-32 and PLATON (Spek, 2009); software used to prepare material for publication: SHELXL97.

The authors thank Department of Science and Technology (DST) and the SAIF, IIT Madras, Chennai, India, for the intensity data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GW2072).

## References

- Ali, A. S. & Swealan, S. A. (1992). *Egypt J. Pharm. Sci.* **33**, 473–477.
- Baldaniya, B. B. & Jotani, M. M. (2008). *Anal. Sci. X-Ray Struct. Online* **24**, x217–x218.
- Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N. L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
- Bourgurgnon, J., Gougeon, E., Queguiner, G. & Pastor, B. (1975). *Bull. Soc. Chim. Fr.* **3-4**, 815–819.
- Bruker (2004). *APEX2, SAINT, XPREP and SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Burla, M. C., Camalli, M., Carrozzini, B., Cascarano, G. L., Giacovazzo, C., Polidori, G. & Spagna, R. (2003). *J. Appl. Cryst.* **36**, 1103.
- Dave, C. G. & Shah, R. D. (2002). *Molecules*, **7**, 534–543.
- Hand, E. S. & Backer, D. C. (1984). *Can. J. Chem.* **62**, 2570–2577.
- Hou, Z.-H., Zhou, N.-B., He, B.-H. & Li, X.-F. (2009). *Acta Cryst.* **E65**, o375.
- Jotani, M. M. & Baldaniya, B. B. (2007). *Acta Cryst.* **E63**, o1937–o1939.
- Jotani, M. M. & Baldaniya, B. B. (2008). *Acta Cryst.* **E64**, o739.
- Malone, J. F., Murray, C. M., Charlton, M. H., Docherty, R. & Lavery, A. J. (1997). *J. Chem. Soc. Faraday Trans.* pp. 3429–3436.
- Omer, A., Mohson, M., Shams, A. & Labouta, I. A. (1991). *J. Pharm. Sci.* **5**, 213–218.
- Peinador, C., Ojea, V. & Quintela, J. M. (1992). *J. Heterocycl. Chem.* **29**, 1698–1702.
- Robba, M., Lecomte, J. M. & Cugnon de, M. (1975). *J. Heterocycl. Chem.* **12**, 525–527.
- Schneller, S. W. & Clough, F. W. (1992). *J. Heterocycl. Chem.* **11**, 975–977.
- Schram, K. L., Manning, S. J. & Townsend, L. B. (1975). *J. Heterocycl. Chem.* **12**, 1021–1023.
- Shah, R. D. (2009). Unpublished results.
- Sheldrick, G. M. (2004). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Shishoo, C. J. & Jain, S. K. (1992). *J. Heterocycl. Chem.* **29**, 883–893.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
- Wilkinson, J. A. (1992). *Chem. Rev.* **92**, 505–519.

**supplementary materials**

*Acta Cryst.* (2010). E66, o212-o213 [ doi:10.1107/S1600536809053653 ]

## 7-(4-Methoxyphenyl)-5-methyl-9-phenyl-7*H*-pyrrolo[2',3':4,5]pyrimido[1,6-*d*]tetrazole

M. M. Jotani, R. D. Shah and J. P. Jasinski

### Comment

Fused tetrazolopyrimidines are important to the activities of a variety of biological substances (Willkinson, 1992; Omer *et al.*, 1991; Schram *et al.*, 1975). Moreover, fused pyrimidines having a halogen at the 2- or 4- position seem to be more labile towards a nucleophilic substitution reaction with reagents such as piperadine, piperazine, morpholine, hydrazine and azides to form potent bi and triheterocycles (Dave & Shah, 2002; Peinador *et al.*, 1992; Schneller & Clough, 1992; Shishoo & Jain, 1992). The reduction of tetrazolopyrimidines *via* azidolysis studies have been shown to be attractive to development of synthetically important 4-aminopyrimidines (Shishoo & Jain, 1992; Hand & Backer, 1984). The treatment of sodium azide with 4-chloropyrrolo[2,3-*e*] pyrimidine can result the formation of either an azido group or tetrazole ring upon a fused pyrimidine ring. Such nucleophilic substitution reactions have rarely been attempted in pyrrolo[2,3-*e*] pyrimidines (Dave & Shah, 2002; Ali & Swealan, 1992). In view of the importance of these molecules, a crystal structure of the title compound, C<sub>20</sub>H<sub>16</sub>N<sub>6</sub>O, (I) has been determined.

The title compound, C<sub>20</sub>H<sub>16</sub>N<sub>6</sub>O, (I), is composed of a tetrazolo ring and a 4-methoxyphenyl and benzene substituted pyrrole ring at the 7 and 9 position fused to a pyrimidine ring in a nearly planar fashion (Fig. 1). The r.m.s. deviation of atoms of the fused ring from the mean plane through the heterocyclic system is 0.0085 Å, with a maximum deviation of -0.018 (1) and 0.013 (1) Å for atoms C2 and C3 respectively. Bond lengths and angles for the fused pyrrole and tetrazole rings in (I) are normal and similar to that observed for a related structure. The dihedral angles between the mean planes of fused pyrimidine and tetrazole rings with that of the pyrrole ring are 1.26 (6) ° and 1.13 (7)°, respectively. A methyl group at the 5 position is also in the plane of the pyrimidine ring. The dihedral angle between the mean planes of the benzene and 4-methoxyphenyl rings is 40.4 (2)°. The angles between the mean planes of the pyrimidine and the benzene and 4-methoxyphenyl rings are 15.6 (5)° and 52.6 (7)°, respectively. A weak intramolecular C13–H13···N6 hydrogen bond interaction, which forms an S(7) graph set, helps stabilize the separation angle between the tetrazolo and benzene rings. Weak intermolecular C12–H12···O1 (Fig. 2), C–H···π-ring (C7–H7B(H7C)···Cg4 [= 3.620 (2) (3.554 (2) Å; 1 - x, 1 - y, 1 - z (1 - x, 2 - y, 1 - z); where Cg4 = C8–C13 ring centroid; Table 1] and π–π [Cg1···Cg2; = 3.5270 (16)Å & 3.5113 (16) Å, 1 - x, 1 - y, 1 - x & 1 - x, 2 - y, 1 - z; Cg2···Cg3; = 3.7275 (17) Å, 1 - x, 2 - y, 1 - z; Cg3···Cg1; = 3.7866 (17) Å, 1 - x, 1 - y, 1 - z; where Cg1 = N1/C1–C4, Cg2 = N3–N6/C6, Cg3 = N2/C1–C5] stacking interactions (Fig. 3) help to link the molecules into a 2-D array obliquely parallel to (101) and propagating along the b axis.

### Experimental

The title compound was synthesized according to method of Shah (2009). A mixture of sodium azide (0.011 mole), ammonium chloride (0.011 mole) and 2-methyl-5-phenyl-7-(4-methoxyphenyl)-4-chloro-7*H*-pyrrolo[2,3-*d*]pyrimidine (0.01 mole) in DMSO (20 ml) was stirred for for 2 h at 363 K to obtain the title compound (I). Colorless platlike single crystals, suitable for X-ray diffraction were grown from a solution of 1,4-dioxane.

## Refinement

All of the H atoms were placed in their calculated positions and then refined using the riding model with C—H = 0.95–0.98 Å, and with  $U_{\text{iso}}(\text{H}) = 1.18\text{--}1.50U_{\text{eq}}(\text{C})$ .

## Figures

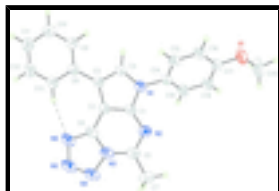


Fig. 1. The molecular structure of (I), showing the atom labelling scheme and 50% probability displacement ellipsoids. The dashed line represents a weak intramolecular C—H $\cdots$ N hydrogen bond interaction.

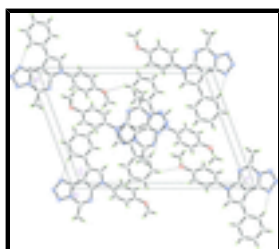


Fig. 2. Crystal packing of (I), showing weak intramolecular C—H $\cdots$ N and intermolecular C—H $\cdots$ O hydrogen bond interactions as dashed lines.

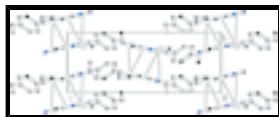


Fig. 3. The molecular packing of (I), showing  $\pi$ – $\pi$  stacking interactions as dashed lines forming chains of molecules along [1 0 1] plane of the unit cell.

## 7-(4-Methoxyphenyl)-5-methyl-9-phenyl-7H-pyrrolo[2',3':4,5]pyrimido[1,6-d]tetrazole

### Crystal data

$\text{C}_{20}\text{H}_{16}\text{N}_6\text{O}$

$M_r = 356.39$

Monoclinic,  $P2_1/n$

Hall symbol:  $-P\ 2_1/n$

$a = 13.738\ (2)\ \text{\AA}$

$b = 7.032\ (3)\ \text{\AA}$

$c = 19.4350\ (3)\ \text{\AA}$

$\beta = 110.217\ (2)^\circ$

$V = 1761.9\ (8)\ \text{\AA}^3$

$Z = 4$

$F(000) = 744$

$D_x = 1.344\ \text{Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 5176 reflections

$\theta = 2.2\text{--}31.3^\circ$

$\mu = 0.09\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Plate, yellow

$0.47 \times 0.35 \times 0.2\ \text{mm}$

### Data collection

Bruker Kappa APEXII CCD  
diffractometer

Radiation source: fine-focus sealed tube  
graphite

5137 independent reflections

3694 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.030$

$\omega$ and $\varphi$ scan	$\theta_{\max} = 30.0^\circ$ , $\theta_{\min} = 1.6^\circ$
Absorption correction: multi-scan ( <i>SADABS</i> ; Sheldrick, 2004)	$h = -19 \rightarrow 19$
$T_{\min} = 0.96$ , $T_{\max} = 0.98$	$k = -4 \rightarrow 9$
42323 measured reflections	$l = -27 \rightarrow 27$

### Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.045$	H-atom parameters constrained
$wR(F^2) = 0.129$	$w = 1/[\sigma^2(F_o^2) + (0.0656P)^2 + 0.2469P]$
$S = 1.03$	where $P = (F_o^2 + 2F_c^2)/3$
5137 reflections	$(\Delta/\sigma)_{\max} = 0.001$
247 parameters	$\Delta\rho_{\max} = 0.29 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\min} = -0.20 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: <i>SHELXL97</i> (Sheldrick, 2008), $F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
	Extinction coefficient: 0.0104 (12)

### Special details

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'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  $F^2 > \sigma(F^2)$  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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.49323 (7)	0.66669 (14)	0.35550 (5)	0.0364 (2)
N2	0.64444 (8)	0.71967 (13)	0.46114 (6)	0.0390 (2)
N3	0.60830 (8)	0.78499 (13)	0.56626 (5)	0.0387 (2)
N4	0.63253 (10)	0.82763 (16)	0.63905 (6)	0.0503 (3)
N5	0.54477 (10)	0.84057 (18)	0.64892 (6)	0.0553 (3)
N6	0.46241 (9)	0.80796 (15)	0.58676 (5)	0.0461 (3)
O1	0.67511 (8)	0.63011 (14)	0.14173 (5)	0.0557 (3)
C1	0.54006 (9)	0.70521 (14)	0.42843 (6)	0.0342 (2)
C2	0.46308 (9)	0.72839 (14)	0.45931 (6)	0.0322 (2)
C3	0.36448 (9)	0.70460 (14)	0.40206 (6)	0.0331 (2)
C4	0.38831 (9)	0.66682 (16)	0.34039 (6)	0.0372 (2)

## supplementary materials

---

H4	0.3383	0.6437	0.2933	0.045*
C5	0.67850 (10)	0.75945 (15)	0.53047 (7)	0.0400 (3)
C6	0.50261 (9)	0.77276 (14)	0.53513 (6)	0.0350 (2)
C7	0.79065 (11)	0.7757 (2)	0.57366 (8)	0.0538 (3)
H7A	0.8307	0.7651	0.5408	0.081*
H7B	0.8109	0.6735	0.6102	0.081*
H7C	0.8044	0.8991	0.5986	0.081*
C8	0.25890 (9)	0.71760 (15)	0.40362 (6)	0.0348 (2)
C9	0.17475 (10)	0.73306 (18)	0.33836 (7)	0.0437 (3)
H9	0.1867	0.7361	0.2931	0.052*
C10	0.07452 (11)	0.7441 (2)	0.33822 (8)	0.0531 (3)
H10	0.0183	0.7540	0.2931	0.064*
C11	0.05569 (11)	0.7407 (2)	0.40303 (9)	0.0572 (4)
H11	-0.0134	0.7493	0.4030	0.069*
C12	0.13717 (12)	0.7249 (2)	0.46801 (9)	0.0559 (4)
H12	0.1242	0.7217	0.5129	0.067*
C13	0.23768 (11)	0.71372 (18)	0.46865 (7)	0.0444 (3)
H13	0.2932	0.7032	0.5141	0.053*
C14	0.54240 (9)	0.65119 (16)	0.30183 (6)	0.0352 (2)
C15	0.50818 (9)	0.76648 (17)	0.24049 (6)	0.0409 (3)
H15	0.4533	0.8542	0.2348	0.049*
C16	0.55355 (10)	0.75407 (18)	0.18782 (7)	0.0437 (3)
H16	0.5294	0.8320	0.1454	0.052*
C17	0.63448 (9)	0.62818 (17)	0.19641 (6)	0.0397 (3)
C18	0.66942 (9)	0.51457 (18)	0.25825 (7)	0.0430 (3)
H18	0.7254	0.4291	0.2646	0.052*
C19	0.62275 (9)	0.52564 (17)	0.31077 (6)	0.0413 (3)
H19	0.6461	0.4467	0.3530	0.050*
C20	0.75810 (14)	0.5043 (3)	0.14708 (10)	0.0782 (6)
H20A	0.8168	0.5324	0.1918	0.117*
H20B	0.7795	0.5207	0.1043	0.117*
H20C	0.7354	0.3728	0.1488	0.117*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
N1	0.0378 (5)	0.0443 (5)	0.0276 (5)	0.0018 (4)	0.0121 (4)	-0.0010 (4)
N2	0.0382 (5)	0.0396 (5)	0.0362 (5)	0.0018 (4)	0.0092 (4)	0.0011 (4)
N3	0.0492 (6)	0.0345 (4)	0.0271 (5)	-0.0023 (4)	0.0064 (4)	0.0001 (3)
N4	0.0663 (8)	0.0509 (6)	0.0272 (5)	-0.0076 (5)	0.0080 (5)	-0.0035 (4)
N5	0.0717 (8)	0.0613 (7)	0.0300 (6)	-0.0110 (6)	0.0139 (5)	-0.0068 (5)
N6	0.0620 (7)	0.0495 (6)	0.0284 (5)	-0.0074 (5)	0.0175 (5)	-0.0042 (4)
O1	0.0641 (6)	0.0690 (6)	0.0459 (5)	0.0180 (5)	0.0340 (5)	0.0111 (4)
C1	0.0401 (6)	0.0329 (5)	0.0285 (5)	0.0015 (4)	0.0105 (4)	0.0016 (4)
C2	0.0406 (6)	0.0288 (4)	0.0272 (5)	0.0002 (4)	0.0118 (4)	0.0018 (4)
C3	0.0388 (6)	0.0327 (5)	0.0278 (5)	0.0006 (4)	0.0115 (4)	0.0016 (4)
C4	0.0365 (6)	0.0436 (6)	0.0298 (5)	0.0000 (4)	0.0095 (5)	-0.0017 (4)
C5	0.0437 (7)	0.0334 (5)	0.0373 (6)	0.0010 (4)	0.0069 (5)	0.0014 (4)

C6	0.0458 (6)	0.0289 (5)	0.0293 (5)	-0.0020 (4)	0.0115 (5)	0.0017 (4)
C7	0.0451 (7)	0.0510 (7)	0.0520 (8)	-0.0023 (5)	-0.0001 (6)	-0.0040 (6)
C8	0.0407 (6)	0.0320 (5)	0.0326 (6)	-0.0005 (4)	0.0137 (5)	-0.0007 (4)
C9	0.0426 (7)	0.0532 (7)	0.0346 (6)	0.0003 (5)	0.0124 (5)	-0.0012 (5)
C10	0.0404 (7)	0.0647 (8)	0.0496 (8)	0.0015 (6)	0.0096 (6)	-0.0037 (6)
C11	0.0430 (8)	0.0684 (9)	0.0646 (10)	-0.0028 (6)	0.0244 (7)	-0.0089 (7)
C12	0.0543 (8)	0.0711 (9)	0.0512 (8)	-0.0043 (7)	0.0297 (7)	-0.0068 (6)
C13	0.0464 (7)	0.0536 (7)	0.0349 (6)	-0.0008 (5)	0.0161 (5)	-0.0016 (5)
C14	0.0369 (6)	0.0420 (5)	0.0277 (5)	-0.0006 (4)	0.0124 (4)	-0.0023 (4)
C15	0.0381 (6)	0.0512 (6)	0.0327 (6)	0.0103 (5)	0.0113 (5)	0.0035 (5)
C16	0.0448 (7)	0.0550 (7)	0.0312 (6)	0.0087 (5)	0.0129 (5)	0.0090 (5)
C17	0.0416 (6)	0.0474 (6)	0.0337 (6)	0.0020 (5)	0.0174 (5)	0.0000 (4)
C18	0.0451 (7)	0.0448 (6)	0.0426 (7)	0.0110 (5)	0.0195 (5)	0.0049 (5)
C19	0.0472 (7)	0.0433 (6)	0.0348 (6)	0.0075 (5)	0.0158 (5)	0.0069 (4)
C20	0.0943 (13)	0.0825 (11)	0.0864 (12)	0.0367 (10)	0.0674 (11)	0.0245 (9)

*Geometric parameters (Å, °)*

N1—C1	1.3661 (14)	C8—C9	1.3942 (17)
N1—C4	1.3680 (15)	C9—C10	1.3783 (18)
N1—C14	1.4294 (14)	C9—H9	0.9500
N2—C5	1.2951 (16)	C10—C11	1.371 (2)
N2—C1	1.3567 (15)	C10—H10	0.9500
N3—C6	1.3691 (16)	C11—C12	1.372 (2)
N3—N4	1.3699 (14)	C11—H11	0.9500
N3—C5	1.3819 (17)	C12—C13	1.379 (2)
N4—N5	1.2879 (17)	C12—H12	0.9500
N5—N6	1.3591 (16)	C13—H13	0.9500
N6—C6	1.3254 (15)	C14—C19	1.3768 (16)
O1—C17	1.3606 (13)	C14—C15	1.3828 (16)
O1—C20	1.4183 (17)	C15—C16	1.3730 (17)
C1—C2	1.3942 (16)	C15—H15	0.9500
C2—C6	1.4178 (15)	C16—C17	1.3854 (17)
C2—C3	1.4351 (15)	C16—H16	0.9500
C3—C4	1.3730 (15)	C17—C18	1.3835 (16)
C3—C8	1.4640 (16)	C18—C19	1.3825 (16)
C4—H4	0.9500	C18—H18	0.9500
C5—C7	1.4824 (18)	C19—H19	0.9500
C7—H7A	0.9800	C20—H20A	0.9800
C7—H7B	0.9800	C20—H20B	0.9800
C7—H7C	0.9800	C20—H20C	0.9800
C8—C13	1.3920 (17)		
C1—N1—C4	107.82 (9)	C10—C9—H9	119.3
C1—N1—C14	126.90 (10)	C8—C9—H9	119.3
C4—N1—C14	124.89 (9)	C11—C10—C9	120.20 (14)
C5—N2—C1	116.55 (11)	C11—C10—H10	119.9
C6—N3—N4	108.19 (10)	C9—C10—H10	119.9
C6—N3—C5	125.97 (10)	C10—C11—C12	119.60 (13)
N4—N3—C5	125.83 (11)	C10—C11—H11	120.2



## supplementary materials

---

N5—N4—N3	105.27 (10)	C12—C11—H11	120.2
N4—N5—N6	112.98 (11)	C11—C12—C13	120.58 (13)
C6—N6—N5	105.53 (11)	C11—C12—H12	119.7
C17—O1—C20	118.22 (10)	C13—C12—H12	119.7
N2—C1—N1	122.92 (10)	C12—C13—C8	120.93 (13)
N2—C1—C2	128.74 (10)	C12—C13—H13	119.5
N1—C1—C2	108.33 (10)	C8—C13—H13	119.5
C1—C2—C6	113.45 (10)	C19—C14—C15	120.24 (10)
C1—C2—C3	107.80 (9)	C19—C14—N1	121.15 (10)
C6—C2—C3	138.71 (11)	C15—C14—N1	118.61 (10)
C4—C3—C2	104.68 (10)	C16—C15—C14	119.92 (11)
C4—C3—C8	124.54 (10)	C16—C15—H15	120.0
C2—C3—C8	130.77 (10)	C14—C15—H15	120.0
N1—C4—C3	111.35 (10)	C15—C16—C17	120.19 (11)
N1—C4—H4	124.3	C15—C16—H16	119.9
C3—C4—H4	124.3	C17—C16—H16	119.9
N2—C5—N3	119.19 (11)	O1—C17—C18	124.85 (11)
N2—C5—C7	122.47 (12)	O1—C17—C16	115.35 (10)
N3—C5—C7	118.33 (11)	C18—C17—C16	119.78 (11)
N6—C6—N3	108.03 (10)	C19—C18—C17	119.91 (11)
N6—C6—C2	135.87 (11)	C19—C18—H18	120.0
N3—C6—C2	116.10 (10)	C17—C18—H18	120.0
C5—C7—H7A	109.5	C14—C19—C18	119.94 (11)
C5—C7—H7B	109.5	C14—C19—H19	120.0
H7A—C7—H7B	109.5	C18—C19—H19	120.0
C5—C7—H7C	109.5	O1—C20—H20A	109.5
H7A—C7—H7C	109.5	O1—C20—H20B	109.5
H7B—C7—H7C	109.5	H20A—C20—H20B	109.5
C13—C8—C9	117.35 (11)	O1—C20—H20C	109.5
C13—C8—C3	122.51 (11)	H20A—C20—H20C	109.5
C9—C8—C3	120.13 (10)	H20B—C20—H20C	109.5
C10—C9—C8	121.33 (12)		
C6—N3—N4—N5	-0.45 (12)	C5—N3—C6—C2	0.47 (15)
C5—N3—N4—N5	179.34 (10)	C1—C2—C6—N6	179.44 (12)
N3—N4—N5—N6	0.33 (14)	C3—C2—C6—N6	1.9 (2)
N4—N5—N6—C6	-0.08 (14)	C1—C2—C6—N3	-0.34 (13)
C5—N2—C1—N1	179.05 (10)	C3—C2—C6—N3	-177.88 (11)
C5—N2—C1—C2	0.35 (17)	C4—C3—C8—C13	165.29 (11)
C4—N1—C1—N2	-178.52 (10)	C2—C3—C8—C13	-15.64 (17)
C14—N1—C1—N2	-5.42 (17)	C4—C3—C8—C9	-14.18 (16)
C4—N1—C1—C2	0.41 (12)	C2—C3—C8—C9	164.89 (11)
C14—N1—C1—C2	173.51 (10)	C13—C8—C9—C10	0.00 (17)
N2—C1—C2—C6	-0.04 (16)	C3—C8—C9—C10	179.50 (11)
N1—C1—C2—C6	-178.89 (9)	C8—C9—C10—C11	0.3 (2)
N2—C1—C2—C3	178.25 (10)	C9—C10—C11—C12	-0.5 (2)
N1—C1—C2—C3	-0.60 (11)	C10—C11—C12—C13	0.4 (2)
C1—C2—C3—C4	0.54 (11)	C11—C12—C13—C8	-0.2 (2)
C6—C2—C3—C4	178.17 (12)	C9—C8—C13—C12	-0.05 (17)
C1—C2—C3—C8	-178.67 (10)	C3—C8—C13—C12	-179.54 (11)

C6—C2—C3—C8	-1.0 (2)	C1—N1—C14—C19	55.88 (16)
C1—N1—C4—C3	-0.06 (13)	C4—N1—C14—C19	-132.14 (12)
C14—N1—C4—C3	-173.33 (10)	C1—N1—C14—C15	-123.61 (12)
C2—C3—C4—N1	-0.30 (12)	C4—N1—C14—C15	48.36 (16)
C8—C3—C4—N1	178.97 (9)	C19—C14—C15—C16	0.85 (18)
C1—N2—C5—N3	-0.24 (15)	N1—C14—C15—C16	-179.66 (11)
C1—N2—C5—C7	178.70 (10)	C14—C15—C16—C17	-0.88 (19)
C6—N3—C5—N2	-0.17 (16)	C20—O1—C17—C18	1.4 (2)
N4—N3—C5—N2	-179.92 (10)	C20—O1—C17—C16	-179.88 (14)
C6—N3—C5—C7	-179.15 (10)	C15—C16—C17—O1	-178.74 (12)
N4—N3—C5—C7	1.10 (16)	C15—C16—C17—C18	0.1 (2)
N5—N6—C6—N3	-0.22 (12)	O1—C17—C18—C19	179.42 (12)
N5—N6—C6—C2	179.99 (12)	C16—C17—C18—C19	0.70 (19)
N4—N3—C6—N6	0.42 (12)	C15—C14—C19—C18	-0.04 (18)
C5—N3—C6—N6	-179.37 (10)	N1—C14—C19—C18	-179.52 (11)
N4—N3—C6—C2	-179.74 (9)	C17—C18—C19—C14	-0.73 (19)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C13—H13...N6	0.95	2.38	3.2159 (18)	146
C12—H12...O1 <sup>i</sup>	0.95	2.57	3.3902 (17)	144
C7—H7B...Cg4 <sup>ii</sup>	0.96	2.90	3.620 (2)	132
C7—H7C...Cg4 <sup>iii</sup>	0.96	2.69	3.554 (2)	150

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

Fig. 1

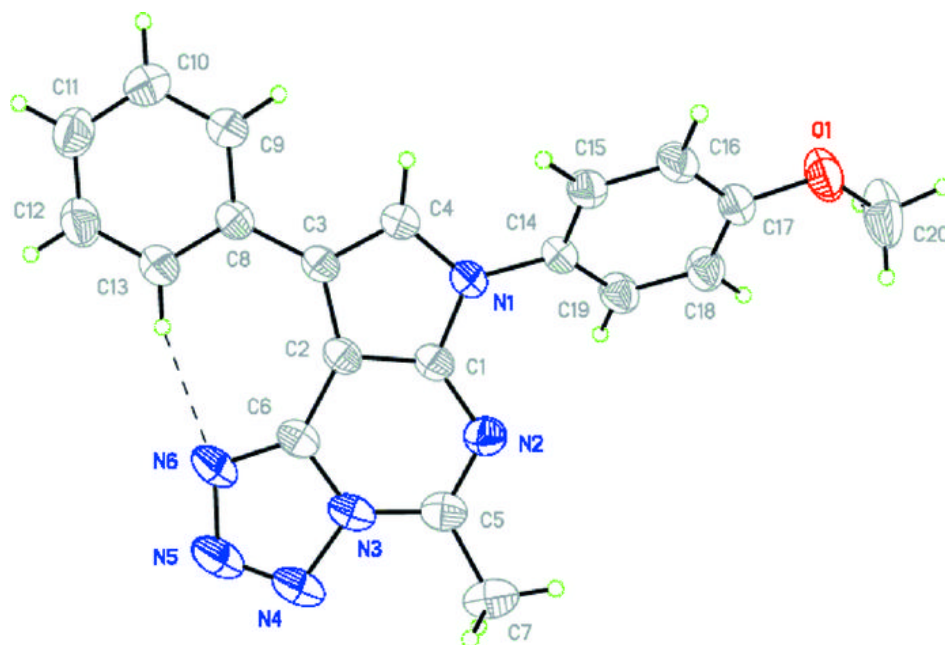


Fig. 2

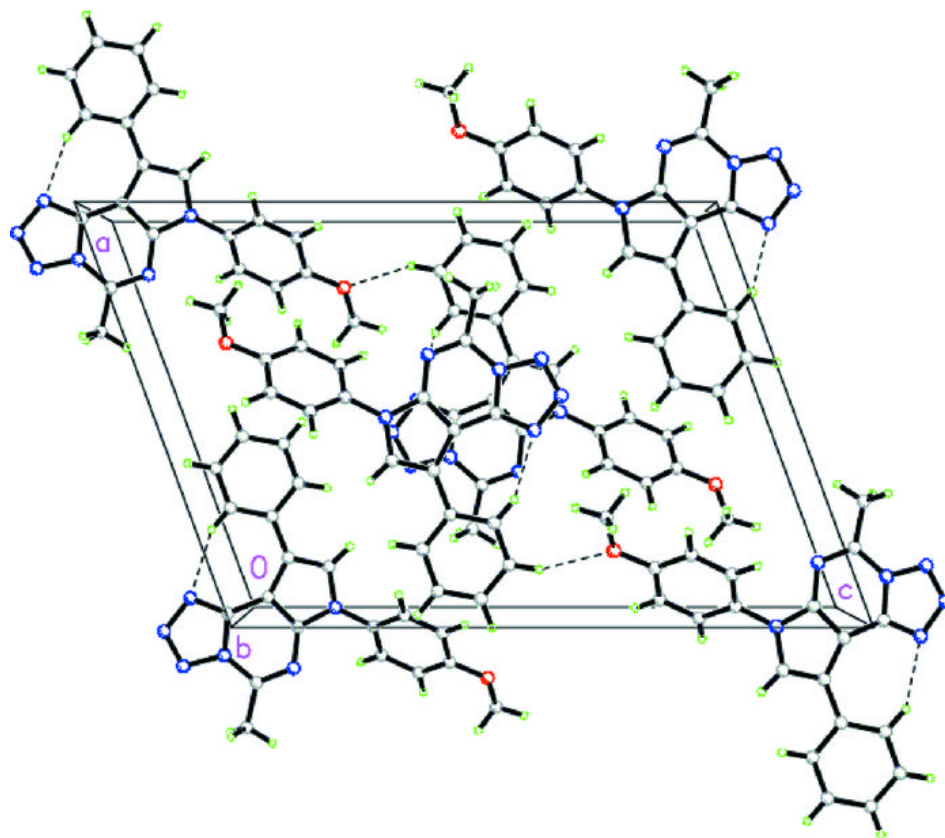


Fig. 3

