IUCrData

ISSN 2414-3146

1-(4-Fluorophenyl)-5-methyl-*N*′-{1-[5-methyl-1-(4-methylphenyl)-1*H*-1,2,3-triazol-4-yl]ethylidene}-1*H*-1,2,3-triazole-4-carbohydrazide

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Received 6 February 2019
Accepted 20 February 2019

Edited by O. Blacque, University of Zürich, Switzerland

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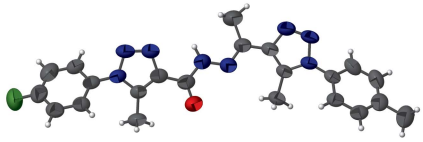
Keywords: crystal structure; carbohydrazide; 1,2,3-triazole.

CCDC reference: 1898411

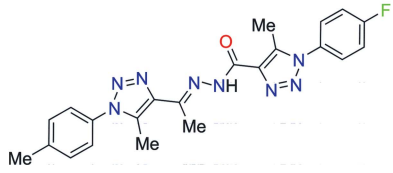
Structural data: full structural data are available from iucrdata.iucr.org

The title molecule, $C_{22}H_{21}FN_8O$, comprises fluorophenyl (*A*), methyltriazolyl (*B*), methyltriazolyl (*C*) and tolyl (*D*) rings. The twist angles between the planes through neighbouring ring pairs *A*/*B*, *B*/*C* and *C*/*D* are 45.0 (1), 9.4 (1) and 43.2 (1)°, respectively. Intermolecular π–π interactions between rings *A* and *C* and between *B* and *D* propagate the structure in the [010] direction and weak C—H···O interactions also occur.

3D view



Chemical scheme



Structure description

Acyl hydrazides are common precursors for the synthesis of various heterocycles and have a wide range of applications such as use in pharmaceuticals, as chemical preservatives for plants, and in the manufacture of polymers (Abdel-Wahab *et al.*, 2017; Hassan & Shawky, 2010; Sarma *et al.*, 2011; Shamsabadi & Chudasama, 2017).

The asymmetric unit comprises one molecule of the title compound. The molecule has fluorophenyl (*A*; C1–C6), methyltriazolyl (*B*; N1–N3/C7/C8), methyltriazolyl (*C*; N6–N8/C13/C14) and tolyl (*D*; C16–C21) rings (Fig. 1). The twist angles between the planes through neighbouring ring pairs *A*/*B*, *B*/*C* and *C*/*D* are 45.0 (1), 9.4 (1) and 43.2 (1)°, respectively. Intermolecular π–π interactions between rings *A* and *C* and between *B* and *D* with centroid-to-centroid distances in the range 3.774 (3) to 4.075 (3) Å propagate the structure in the [010] direction (Fig. 2). Weak C—H···O interactions (Table 1) also occur.

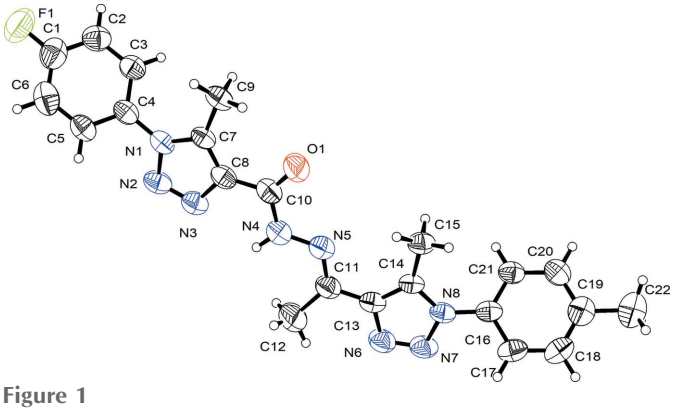


Figure 1
An *ORTEP* representation of the asymmetric unit showing 50% probability ellipsoids.

Synthesis and crystallization

The title compound was synthesized by condensation of 1-(4-fluorophenyl)-5-methyl-1*H*-1,2,3-triazole-4-carbohydrazide with 1-(5-methyl-1-*p*-tolyl-1*H*-1,2,3-triazol-4-yl)ethanone in ethanol in the presence of a catalytic amount of glacial acetic acid under reflux for 2 h. The solid obtained was collected by filtration, washed with ethanol, dried and recrystallized from dimethylformamide solution to give colourless crystals (78%), m.p. 241–242°C.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The geometries of the fluorophenyl and tolyl rings were restrained to be regular hexagons.

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

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12A···O1^{i}	0.96	2.48	3.229 (12)	135

Symmetry code: (i) $x+\frac{1}{2}, -y+\frac{1}{2}, z$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{22}H_{21}FN_8O$
M_r	432.47
Crystal system, space group	Orthorhombic, $Pna2_1$
Temperature (K)	293
a, b, c (Å)	12.2574 (10), 7.6912 (8), 22.821 (3)
V (Å^3)	2151.5 (4)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.25 × 0.16 × 0.09
Data collection	
Diffractometer	Rigaku Oxford Diffraction SuperNova, Dual, Cu at home/near, Atlas
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2018)
T_{min}, T_{max}	0.329, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	13165, 3917, 2393
R_{int}	0.059
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.602
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.062, 0.178, 1.08
No. of reflections	3917
No. of parameters	272
No. of restraints	2
H-atom treatment	H-atom parameters not refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.17, −0.23

Computer programs: *CrysAlis PRO* (Rigaku OD, 2018), *SHELXS* (Sheldrick, 2008), *SHELXL2018* (Sheldrick, 2015), *ORTEP-3 for Windows* and *WinGX* (Farrugia, 2012) and *CHEMDRAW Ultra* (Cambridge Soft, 2001).

Funding information

MHA thanks King Abdulaziz City for Science and Technology (KACST), Saudi Arabia, for financial support (award No. 020–0180).

References

Abdel-Wahab, B. F., Alotaibi, M. H. & El-Hiti, G. A. (2017). *Lett. Org. Chem.* **14**, 591–596.

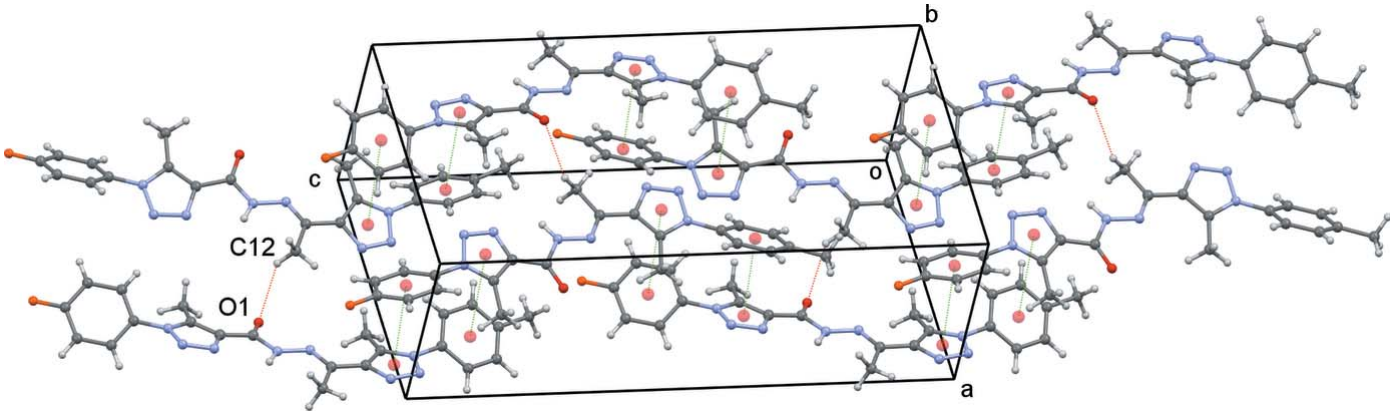


Figure 2
A segment of the crystal structure showing intermolecular π–π contacts as green dotted lines and C—H···O contacts in red.

Cambridge Soft (2001). *CHEMDRAW Ultra*. Cambridge Soft Corporation, Cambridge, Massachusetts, USA.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Hassan, A. A. & Shawky, A. M. (2010). *J. Heterocycl. Chem.* **47**, 745–763.
Rigaku OD (2018). *CrysAlis PRO*. Rigaku Oxford Diffraction, Yarnton, England.
Sarma, B. K., Yousufuddin, M. & Kodadek, T. (2011). *Chem. Commun.* **47**, 10590–10592.
Shamsabadi, A. & Chudasama, V. (2017). *Org. Biomol. Chem.* **15**, 17–33.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.

full crystallographic data

IUCrData (2019). **4**, x190261 [https://doi.org/10.1107/S241431461900261X]

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Crystal data

$C_{22}H_{21}FN_8O$
M_r = 432.47
Orthorhombic, $Pna2_1$
a = 12.2574 (10) Å
b = 7.6912 (8) Å
c = 22.821 (3) Å
V = 2151.5 (4) Å^3
Z = 4
$F(000)$ = 904
D_x = 1.335 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1237 reflections
θ = 3.7–22.8°
μ = 0.09 mm^{-1}
T = 293 K
Plate, colourless
0.25 × 0.16 × 0.09 mm

Data collection

Rigaku Oxford Diffraction SuperNova, Dual, Cu at home/near, Atlas diffractometer
ω scans
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2018)
T_{min} = 0.329, T_{max} = 1.000
13165 measured reflections
3917 independent reflections
2393 reflections with $I > 2\sigma(I)$
R_{int} = 0.059
θ_{max} = 25.3°, θ_{min} = 3.3°
h = −14→14
k = −9→9
l = −27→27

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.178
S = 1.08
3917 reflections
272 parameters
2 restraints
Hydrogen site location: mixed
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0857P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Special details

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

Refinement. All hydrogen atoms except N–H were placed in calculated positions and refined using a riding model. Bond distances for sp^2 C–H H atoms were set to 0.93 Å with their U_{iso} set to 1.2 times U_{eq}(C). Bond distances for methyl C–H H atoms were set to 0.96 Å and their U_{iso} set to 1.5 times U_{eq}(C) with the group free to spin about the C–C bond. The bond distance for the N–H hydrogen was restrained to 0.86 (1) Å and the U_{iso}(H) set to 1.2 times U_{eq}(N).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.1001 (4)	0.3862 (6)	0.57816 (15)	0.099 (2)
C2	0.0357 (3)	0.3166 (6)	0.5338 (2)	0.0845 (18)
H2	−0.033152	0.272817	0.542505	0.101*
C3	0.0742 (3)	0.3125 (5)	0.47657 (17)	0.0728 (15)
H3	0.031140	0.265995	0.446919	0.087*
C4	0.1771 (3)	0.3780 (5)	0.46362 (15)	0.0656 (16)
C5	0.2415 (3)	0.4476 (5)	0.5079 (2)	0.0768 (17)
H5	0.310362	0.491423	0.499269	0.092*
C6	0.2030 (4)	0.4517 (6)	0.56520 (18)	0.100 (2)
H6	0.246072	0.498247	0.594855	0.120*
C7	0.1671 (4)	0.4180 (6)	0.3554 (3)	0.0631 (14)
C8	0.2445 (5)	0.3941 (6)	0.3136 (3)	0.0690 (16)
C9	0.0536 (4)	0.4855 (8)	0.3510 (3)	0.0775 (16)
H9A	0.035619	0.548120	0.386103	0.116*
H9B	0.003943	0.389995	0.346236	0.116*
H9C	0.047980	0.561882	0.317889	0.116*
C10	0.2357 (5)	0.4209 (8)	0.2504 (3)	0.0759 (17)
C11	0.4198 (5)	0.3741 (8)	0.1336 (3)	0.0767 (17)
C12	0.5277 (6)	0.3532 (16)	0.1639 (4)	0.140 (4)
H12A	0.523474	0.258780	0.191350	0.209*
H12B	0.545407	0.458534	0.184426	0.209*
H12C	0.583298	0.329026	0.135441	0.209*
C13	0.4183 (5)	0.3728 (7)	0.0706 (3)	0.0673 (16)
C14	0.3354 (4)	0.4048 (6)	0.0315 (3)	0.0600 (14)
C15	0.2226 (5)	0.4680 (8)	0.0398 (3)	0.0730 (15)
H15A	0.174259	0.370597	0.044115	0.110*
H15B	0.200989	0.535370	0.006314	0.110*
H15C	0.219163	0.539128	0.074298	0.110*
C16	0.3374 (3)	0.3845 (5)	−0.07862 (13)	0.0619 (15)
C17	0.3995 (3)	0.4562 (5)	−0.12351 (18)	0.0797 (17)
H17	0.468108	0.502043	−0.115472	0.096*
C18	0.3591 (4)	0.4593 (5)	−0.18040 (15)	0.0878 (19)
H18	0.400619	0.507228	−0.210432	0.105*
C19	0.2566 (4)	0.3907 (5)	−0.19241 (14)	0.0851 (19)
C20	0.1945 (3)	0.3191 (5)	−0.14752 (19)	0.0771 (16)

H20	0.125893	0.273217	−0.155560	0.092*
C21	0.2349 (3)	0.3160 (5)	−0.09063 (16)	0.0689 (15)
H21	0.193380	0.268032	−0.060599	0.083*
C22	0.2099 (9)	0.3958 (12)	−0.2541 (4)	0.121 (3)
H22A	0.236919	0.298342	−0.275996	0.181*
H22B	0.231591	0.501791	−0.272951	0.181*
H22C	0.131744	0.390311	−0.252184	0.181*
N1	0.2179 (4)	0.3754 (6)	0.4059 (2)	0.0660 (12)
N2	0.3231 (4)	0.3254 (7)	0.3958 (3)	0.0812 (15)
N3	0.3393 (4)	0.3382 (7)	0.3394 (3)	0.0783 (14)
N4	0.3292 (4)	0.3919 (7)	0.2199 (3)	0.0825 (15)
H4	0.386 (4)	0.375 (9)	0.242 (3)	0.099*
N5	0.3285 (4)	0.3929 (6)	0.1598 (3)	0.0761 (14)
N6	0.5106 (4)	0.3288 (7)	0.0395 (3)	0.0846 (15)
N7	0.4885 (4)	0.3301 (7)	−0.0159 (3)	0.0821 (14)
N8	0.3819 (4)	0.3770 (5)	−0.0218 (2)	0.0641 (12)
O1	0.1518 (4)	0.4662 (7)	0.2271 (2)	0.1086 (16)
F1	0.0628 (5)	0.3904 (9)	0.6317 (2)	0.149 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.110 (6)	0.104 (5)	0.083 (6)	0.032 (4)	−0.008 (5)	0.005 (4)
C2	0.073 (4)	0.082 (4)	0.099 (5)	0.014 (3)	0.001 (4)	0.009 (4)
C3	0.066 (4)	0.065 (3)	0.087 (4)	0.003 (3)	−0.005 (3)	−0.001 (3)
C4	0.058 (3)	0.055 (3)	0.084 (5)	0.007 (2)	−0.012 (3)	0.004 (3)
C5	0.070 (4)	0.072 (3)	0.088 (5)	−0.002 (3)	−0.018 (4)	0.007 (3)
C6	0.115 (7)	0.093 (5)	0.094 (6)	0.012 (4)	−0.029 (5)	−0.007 (4)
C7	0.052 (3)	0.057 (3)	0.080 (4)	−0.009 (2)	−0.007 (3)	−0.001 (3)
C8	0.061 (4)	0.055 (3)	0.091 (5)	−0.007 (2)	−0.009 (3)	−0.005 (3)
C9	0.054 (3)	0.078 (3)	0.101 (5)	0.003 (3)	−0.013 (3)	0.004 (3)
C10	0.065 (4)	0.073 (3)	0.090 (5)	−0.002 (3)	−0.011 (4)	−0.010 (3)
C11	0.058 (4)	0.084 (4)	0.088 (5)	0.001 (3)	−0.005 (3)	0.004 (3)
C12	0.074 (5)	0.232 (11)	0.112 (7)	0.014 (5)	−0.015 (5)	−0.005 (6)
C13	0.051 (3)	0.063 (3)	0.088 (5)	0.000 (2)	0.006 (3)	0.003 (3)
C14	0.047 (3)	0.055 (3)	0.079 (4)	−0.003 (2)	0.008 (3)	0.003 (3)
C15	0.057 (3)	0.074 (3)	0.088 (4)	0.008 (3)	0.011 (3)	0.003 (3)
C16	0.054 (3)	0.048 (3)	0.083 (4)	0.009 (2)	0.013 (3)	0.000 (2)
C17	0.077 (4)	0.070 (4)	0.092 (5)	−0.005 (3)	0.027 (4)	−0.009 (3)
C18	0.103 (5)	0.081 (4)	0.080 (5)	0.001 (4)	0.028 (4)	0.003 (3)
C19	0.098 (5)	0.069 (3)	0.088 (5)	0.020 (3)	0.001 (4)	0.001 (3)
C20	0.070 (4)	0.066 (3)	0.095 (5)	0.015 (3)	−0.007 (4)	−0.001 (3)
C21	0.058 (3)	0.060 (3)	0.088 (4)	0.005 (3)	0.004 (3)	0.008 (3)
C22	0.151 (8)	0.119 (6)	0.091 (6)	0.013 (5)	−0.004 (5)	0.002 (5)
N1	0.055 (3)	0.062 (3)	0.081 (4)	−0.001 (2)	−0.012 (3)	−0.001 (2)
N2	0.054 (3)	0.085 (3)	0.105 (5)	0.011 (2)	−0.005 (3)	0.005 (3)
N3	0.060 (3)	0.081 (3)	0.094 (4)	0.003 (2)	−0.004 (3)	0.005 (3)
N4	0.067 (4)	0.097 (4)	0.084 (4)	−0.002 (3)	−0.006 (3)	0.000 (3)

N5	0.064 (3)	0.079 (3)	0.085 (4)	−0.001 (2)	−0.002 (3)	0.002 (3)
N6	0.058 (3)	0.097 (4)	0.099 (5)	0.008 (3)	−0.002 (3)	0.002 (3)
N7	0.051 (3)	0.093 (3)	0.102 (4)	0.018 (2)	0.010 (3)	0.003 (3)
N8	0.048 (3)	0.060 (3)	0.085 (4)	0.0050 (19)	0.005 (3)	0.002 (2)
O1	0.078 (3)	0.159 (5)	0.089 (3)	0.020 (3)	−0.013 (3)	−0.011 (3)
F1	0.150 (5)	0.216 (6)	0.082 (3)	0.031 (4)	0.018 (3)	−0.001 (3)

Geometric parameters (Å, º)

C1—F1	1.305 (6)	C13—C14	1.375 (8)
C1—C2	1.3900	C13—N6	1.378 (8)
C1—C6	1.3900	C14—N8	1.360 (8)
C2—C3	1.3900	C14—C15	1.478 (8)
C2—H2	0.9300	C15—H15A	0.9600
C3—C4	1.3900	C15—H15B	0.9600
C3—H3	0.9300	C15—H15C	0.9600
C4—C5	1.3900	C16—C17	1.3900
C4—N1	1.410 (6)	C16—C21	1.3900
C5—C6	1.3900	C16—N8	1.408 (6)
C5—H5	0.9300	C17—C18	1.3900
C6—H6	0.9300	C17—H17	0.9300
C7—N1	1.349 (8)	C18—C19	1.3900
C7—C8	1.358 (9)	C18—H18	0.9300
C7—C9	1.489 (8)	C19—C20	1.3900
C8—N3	1.371 (8)	C19—C22	1.519 (9)
C8—C10	1.462 (10)	C20—C21	1.3900
C9—H9A	0.9600	C20—H20	0.9300
C9—H9B	0.9600	C21—H21	0.9300
C9—H9C	0.9600	C22—H22A	0.9600
C10—O1	1.209 (7)	C22—H22B	0.9600
C10—N4	1.358 (8)	C22—H22C	0.9600
C11—N5	1.278 (8)	N1—N2	1.365 (6)
C11—C13	1.438 (9)	N2—N3	1.304 (7)
C11—C12	1.501 (10)	N4—N5	1.373 (8)
C12—H12A	0.9600	N4—H4	0.867 (15)
C12—H12B	0.9600	N6—N7	1.294 (7)
C12—H12C	0.9600	N7—N8	1.362 (7)
F1—C1—C2	119.5 (5)	N8—C14—C15	124.0 (5)
F1—C1—C6	120.5 (5)	C13—C14—C15	131.8 (6)
C2—C1—C6	120.0	C14—C15—H15A	109.5
C3—C2—C1	120.0	C14—C15—H15B	109.5
C3—C2—H2	120.0	H15A—C15—H15B	109.5
C1—C2—H2	120.0	C14—C15—H15C	109.5
C2—C3—C4	120.0	H15A—C15—H15C	109.5
C2—C3—H3	120.0	H15B—C15—H15C	109.5
C4—C3—H3	120.0	C17—C16—C21	120.0
C5—C4—C3	120.0	C17—C16—N8	118.9 (3)

C5—C4—N1	119.0 (3)	C21—C16—N8	121.1 (3)
C3—C4—N1	121.0 (3)	C16—C17—C18	120.0
C6—C5—C4	120.0	C16—C17—H17	120.0
C6—C5—H5	120.0	C18—C17—H17	120.0
C4—C5—H5	120.0	C19—C18—C17	120.0
C5—C6—C1	120.0	C19—C18—H18	120.0
C5—C6—H6	120.0	C17—C18—H18	120.0
C1—C6—H6	120.0	C20—C19—C18	120.0
N1—C7—C8	104.1 (5)	C20—C19—C22	119.1 (5)
N1—C7—C9	125.0 (6)	C18—C19—C22	120.9 (5)
C8—C7—C9	130.8 (6)	C19—C20—C21	120.0
C7—C8—N3	109.4 (6)	C19—C20—H20	120.0
C7—C8—C10	128.5 (6)	C21—C20—H20	120.0
N3—C8—C10	122.1 (6)	C20—C21—C16	120.0
C7—C9—H9A	109.5	C20—C21—H21	120.0
C7—C9—H9B	109.5	C16—C21—H21	120.0
H9A—C9—H9B	109.5	C19—C22—H22A	109.5
C7—C9—H9C	109.5	C19—C22—H22B	109.5
H9A—C9—H9C	109.5	H22A—C22—H22B	109.5
H9B—C9—H9C	109.5	C19—C22—H22C	109.5
O1—C10—N4	122.7 (7)	H22A—C22—H22C	109.5
O1—C10—C8	122.5 (6)	H22B—C22—H22C	109.5
N4—C10—C8	114.8 (6)	C7—N1—N2	111.1 (5)
N5—C11—C13	117.1 (6)	C7—N1—C4	129.1 (4)
N5—C11—C12	124.7 (7)	N2—N1—C4	119.8 (5)
C13—C11—C12	118.2 (6)	N3—N2—N1	106.8 (5)
C11—C12—H12A	109.5	N2—N3—C8	108.6 (5)
C11—C12—H12B	109.5	C10—N4—N5	120.4 (5)
H12A—C12—H12B	109.5	C10—N4—H4	115 (5)
C11—C12—H12C	109.5	N5—N4—H4	125 (5)
H12A—C12—H12C	109.5	C11—N5—N4	117.5 (5)
H12B—C12—H12C	109.5	N7—N6—C13	109.3 (5)
C14—C13—N6	108.4 (6)	N6—N7—N8	107.4 (5)
C14—C13—C11	131.2 (6)	C14—N8—N7	110.9 (5)
N6—C13—C11	120.4 (6)	C14—N8—C16	130.9 (4)
N8—C14—C13	104.0 (5)	N7—N8—C16	118.2 (5)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12*A*···O1^{i}	0.96	2.48	3.229 (12)	135

Symmetry code: (i) *x*+1/2, −*y*+1/2, *z*.