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A second monoclinic polymorph of 1-benzyl-N-methyl-1H-pyrrole-2-carboxamide

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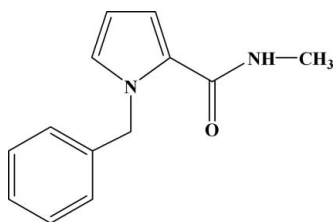
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.063; wR factor = 0.169; data-to-parameter ratio = 16.7.

In the title compound, $\text{C}_{13}\text{H}_{14}\text{N}_2\text{O}$, the $\text{N}_{\text{pyrrole}}-\text{C}(\text{H}_2)-\text{C}-\text{C}$ torsion angle is $-7.7(3)^\circ$ and the dihedral angle between the pyrrole and benzene rings is $83.6(2)^\circ$. In the crystal, intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules into chains extending along the c axis. We have previously reported another polymorphic form of this title compound, which has the same space group with different cell parameters: $a = 9.8285(18)$ Å, $b = 23.588(4)$ Å, $c = 9.9230(17)$ Å, $\beta = 90.107(3)^\circ$, $Z = 8$ and $V = 2300.5(7)$ Å³ [Zeng *et al.* (2010). *Acta Cryst. E* **66**, o2051].

Related literature

For details of the synthesis, see: Zeng *et al.* (2010); For the previously reported polymorph, see: Zeng *et al.* (2010) and for a related structure, see: Zeng *et al.* (2007).



Experimental

Crystal data

$\text{C}_{13}\text{H}_{14}\text{N}_2\text{O}$
 $M_r = 214.26$
 Monoclinic, $P2_1/c$
 $a = 5.4326(4)$ Å
 $b = 22.5218(16)$ Å
 $c = 9.7358(5)$ Å
 $\beta = 101.676(6)^\circ$

$V = 1166.55(13)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.08$ mm⁻¹
 $T = 293$ K
 $0.50 \times 0.28 \times 0.19$ mm

Data collection

Oxford Gemini S Ultra area-detector diffractometer
 Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
 $T_{\text{min}} = 0.962$, $T_{\text{max}} = 0.985$

5327 measured reflections
 2504 independent reflections
 1597 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.063$
 $wR(F^2) = 0.169$
 $S = 1.02$
 2504 reflections
 150 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.17$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.16$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N2}-\text{H2A}\cdots\text{O1}^i$	0.86 (3)	2.08 (3)	2.852 (2)	149 (2)

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZK2014).

References

- Oxford Diffraction (2010). *CrysAlis PRO*. Oxford Diffraction Ltd, Yarnton, England.
 Sheldrick, G. M. (2008). *Acta Cryst. A* **64**, 112–122.
 Zeng, X. C., Li, K. P., Hu, F. & Zheng, L. (2010). *Acta Cryst. E* **66**, o2051.
 Zeng, X.-C., Zeng, J., Li, X. & Ling, X. (2007). *Acta Cryst. E* **63**, o3424.

supporting information

Acta Cryst. (2011). E67, o2235 [doi:10.1107/S1600536811030364]

A second monoclinic polymorph of 1-benzyl-*N*-methyl-1*H*-pyrrole-2-carboxamide

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S1. Comment

This study is related to our previous structural investigations of 1-Benzyl-*N*-methyl-1*H*-pyrrole-2-carboxamide, I, (Zeng *et al.*, 2010) and methyl 2-(4,5-dibromo-1*H*-pyrrole-2-carboxamido)propionate (Zeng *et al.*, 2007)

In I (Fig. 1), the bond lengths and angles are almost same as those observed in the previously reported polymorphic form (Zeng *et al.*, 2010). In the crystal structure, the molecules are linked through N—H···O hydrogen bonds, forming chains extending to the *c* axis (shown in Fig. 2).

It is interesting to note here that the previously reported form was crystallized from an ethanol solution (Zeng *et al.*, 2010), while the present form was crystallized from the solution of ethanol/water (2:1 *v/v*). Although these two polymorphs have the same space group ($P2_1/c$), their unit-cell parameters and melting point are different. For the previous one, the unit-cell parameters are $a = 9.8285$ (18), $b = 23.588$ (4), $c = 9.9230$ (17) Å, $\beta = 90.107$ (3)°, with $Z = 8$, $V = 2300.5$ (7) and m.p. = 365 K. For the present one, they are 5.4326 (4), 22.5218 (16), 9.7358 (5) Å, 101.676 (6)°, $Z = 4$, $V = 1166.55$ (13) and m.p. = 360 K.

As reported, the previous polymorph structure show that the asymmetric unit of the compound contains two independent molecules, which differ in the twist of the phenyl ring: the $N_{\text{pyrrole}}\text{—C(H}_2\text{)—C—C}$ torsion angles are -73.0 (3)° and 17.1 (3)°, respectively. And for the present one, the asymmetric unit contains just one molecule, in this molecule, the $N_{\text{pyrrole}}\text{—C(H}_2\text{)—C—C}$ torsion angle is -7.7 (3)°, the dihedral angle between the pyrrole plane and the benzene plane is 83.60 (2)°.

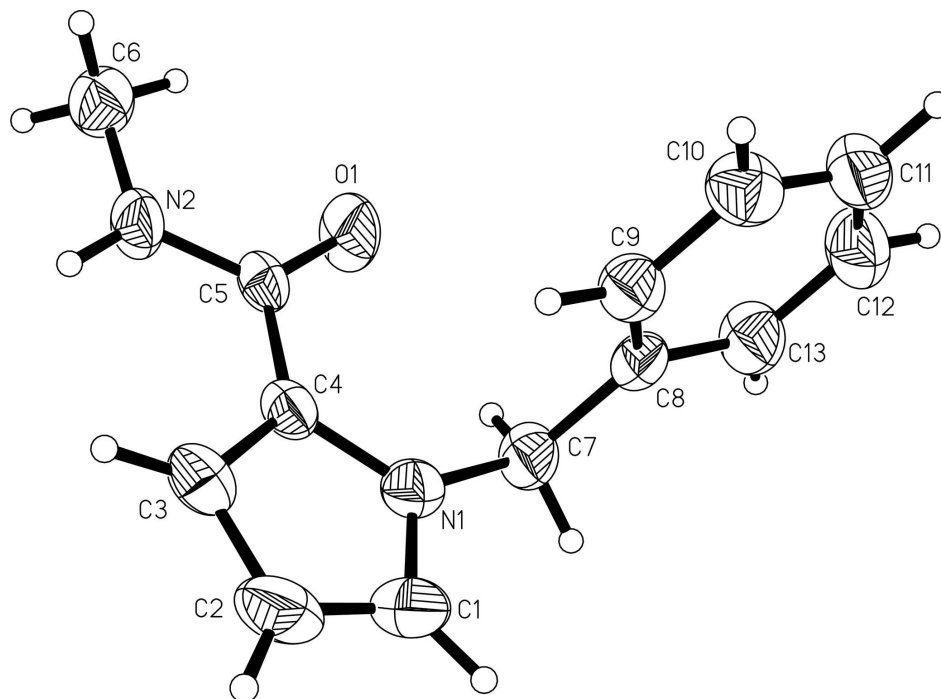
The crystal packings of these two structures are differences also. In the previous polymorph structure, molecules are linked through N—H···O hydrogen bonds, generating chains extending to the *a* axis (shown in Fig. 3). And for the present one, N—H···O hydrogen bonds link molecules together to forming chains extending to the *c* axis (shown in Fig. 2).

S2. Experimental

The title compound was synthesized according to the literature procedure (Zeng *et al.*, 2010). The product was dissolved in the mixture of ethanol / water (2:1 *v/v*), colorless crystals suitable for X-ray analysis were obtained over a period of five days by slow evaporation at room temperature of the solution.

S3. Refinement

All non-H atoms were refined with anisotropic displacement parameters. The H atoms were positioned geometrically [$\text{C—H} = 0.97$ Å for CH_2 , 0.96 Å for CH_3 , 0.93 Å for CH (aromatic) and $\text{N—H} = 0.86$ Å] and refined using a riding model, with $U_{\text{iso}} = 1.2U_{\text{eq}}$ ($1.5U_{\text{eq}}$ for the methyl group) of the parent atom.

**Figure 1**

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

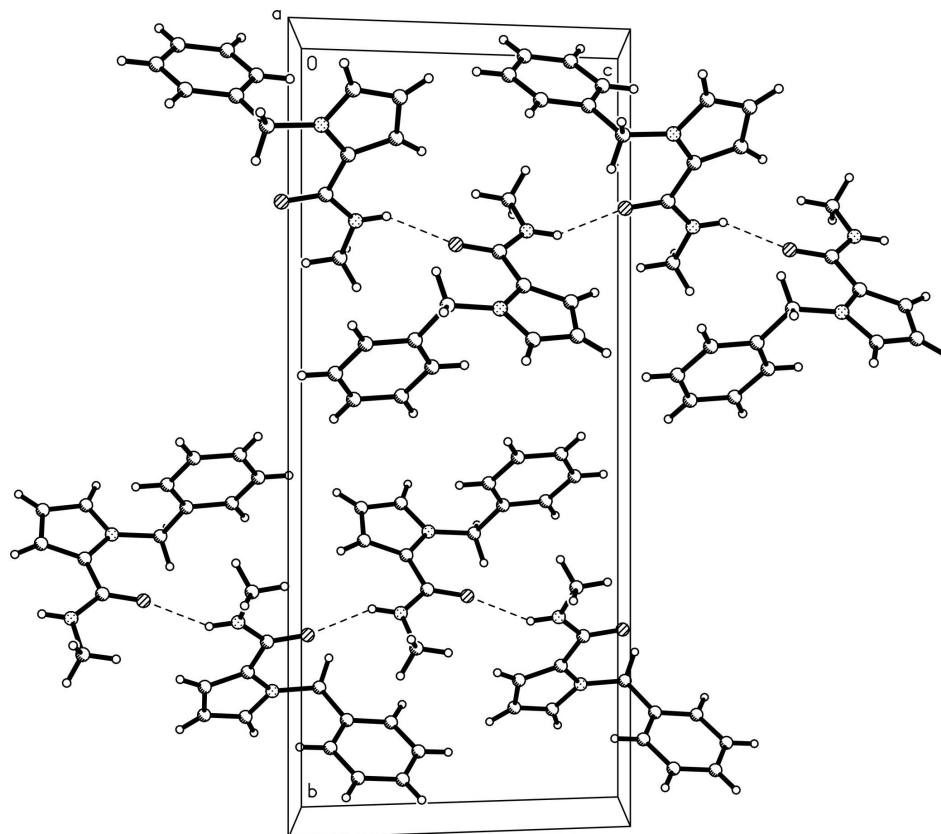


Figure 2

Crystal packing of (I) showing the chains formed by hydrogen bonds(dashed lines).

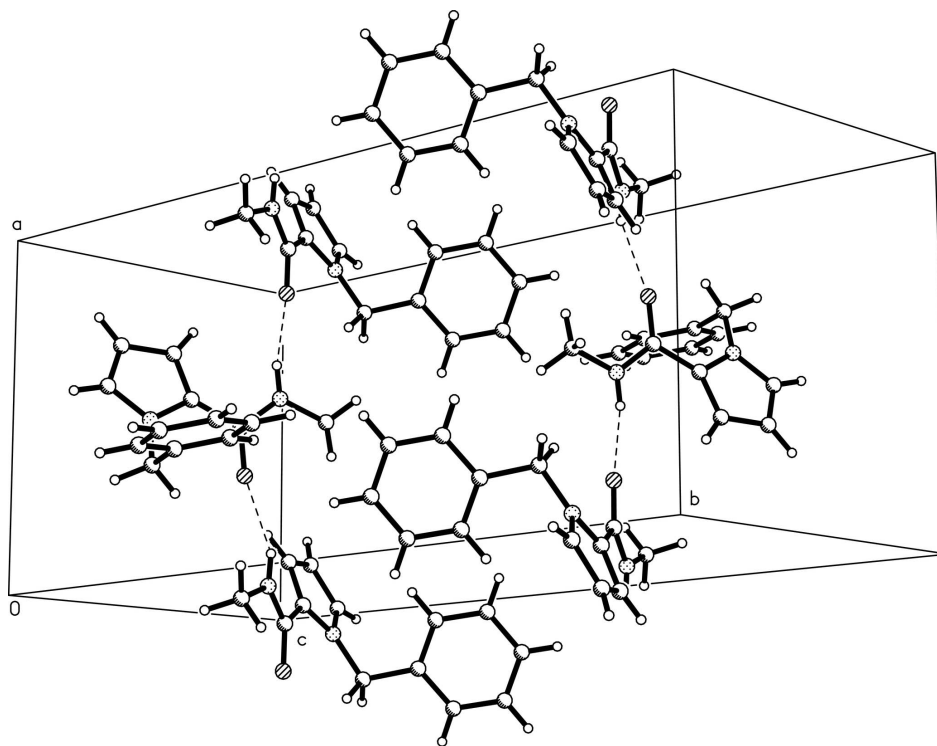


Figure 3

Crystal packing of the previous polymorph structure showing the chains formed by hydrogen bonds(dashed lines).

1-Benzyl-*N*-methyl-1*H*-pyrrole-2-carboxamide

Crystal data

$C_{13}H_{14}N_2O$

$M_r = 214.26$

Monoclinic, $P2_1/c$

$a = 5.4326$ (4) Å

$b = 22.5218$ (16) Å

$c = 9.7358$ (5) Å

$\beta = 101.676$ (6)°

$V = 1166.55$ (13) Å³

$Z = 4$

$F(000) = 456$

$D_x = 1.220$ Mg m⁻³

Melting point: 360 K

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1936 reflections

$\theta = 3.5$ – 29.2 °

$\mu = 0.08$ mm⁻¹

$T = 293$ K

Prism, colorless

$0.50 \times 0.28 \times 0.19$ mm

Data collection

Oxford Gemini S Ultra area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*CrysAlis PRO*; Oxford Diffraction, 2010)

$T_{\min} = 0.962$, $T_{\max} = 0.985$

5327 measured reflections

2504 independent reflections

1597 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.025$

$\theta_{\max} = 27.0$ °, $\theta_{\min} = 3.5$ °

$h = -6 \rightarrow 6$

$k = -28 \rightarrow 22$

$l = -12 \rightarrow 11$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.063$
 $wR(F^2) = 0.169$
 $S = 1.02$
 2504 reflections
 150 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 atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0629P)^2 + 0.3194P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.004$
 $\Delta\rho_{\max} = 0.17 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.16 \text{ e } \text{\AA}^{-3}$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C5	0.0889 (4)	0.28312 (9)	0.60936 (19)	0.0517 (5)
C4	0.2716 (4)	0.32716 (10)	0.6779 (2)	0.0543 (5)
N1	0.4311 (3)	0.35726 (9)	0.6084 (2)	0.0652 (5)
O1	0.0465 (4)	0.27429 (8)	0.48276 (15)	0.0877 (6)
N2	-0.0360 (4)	0.25434 (9)	0.6916 (2)	0.0688 (6)
C8	0.2613 (4)	0.39425 (10)	0.3667 (2)	0.0590 (6)
C7	0.4436 (5)	0.35414 (12)	0.4599 (2)	0.0729 (7)
H7A	0.4117	0.3135	0.4281	0.088*
H7B	0.6127	0.3643	0.4501	0.088*
C9	0.0783 (4)	0.42544 (11)	0.4135 (3)	0.0681 (6)
H9	0.0665	0.4229	0.5073	0.082*
C3	0.3192 (5)	0.34701 (11)	0.8140 (2)	0.0719 (7)
H3	0.2398	0.3342	0.8848	0.086*
C1	0.5712 (5)	0.39542 (13)	0.6999 (3)	0.0870 (8)
H1	0.6916	0.4213	0.6787	0.104*
C11	-0.0747 (6)	0.46500 (12)	0.1851 (3)	0.0847 (8)
H11	-0.1872	0.4888	0.1244	0.102*
C6	-0.2303 (5)	0.21166 (12)	0.6402 (3)	0.0819 (8)
H6A	-0.3890	0.2266	0.6541	0.123*
H6B	-0.1930	0.1750	0.6903	0.123*
H6C	-0.2385	0.2050	0.5420	0.123*
C12	0.1055 (6)	0.43425 (15)	0.1372 (3)	0.0962 (9)
H12	0.1160	0.4368	0.0432	0.115*
C10	-0.0895 (5)	0.46073 (12)	0.3229 (3)	0.0799 (7)

H10	-0.2130	0.4816	0.3561	0.096*
C13	0.2726 (5)	0.39933 (13)	0.2273 (3)	0.0830 (8)
H13	0.3960	0.3787	0.1933	0.100*
C2	0.5084 (5)	0.38987 (14)	0.8271 (3)	0.0911 (9)
H2	0.5783	0.4107	0.9080	0.109*
H2A	0.002 (5)	0.2599 (12)	0.781 (3)	0.092 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C5	0.0605 (12)	0.0601 (13)	0.0343 (9)	0.0096 (10)	0.0092 (8)	0.0024 (9)
C4	0.0544 (12)	0.0661 (13)	0.0408 (10)	0.0075 (10)	0.0060 (8)	0.0031 (9)
N1	0.0503 (10)	0.0815 (14)	0.0631 (11)	0.0025 (10)	0.0095 (9)	0.0076 (10)
O1	0.1194 (15)	0.1092 (14)	0.0364 (8)	-0.0265 (12)	0.0205 (8)	-0.0120 (8)
N2	0.0875 (15)	0.0786 (14)	0.0402 (10)	-0.0144 (11)	0.0126 (10)	0.0021 (10)
C8	0.0509 (12)	0.0652 (14)	0.0648 (13)	-0.0049 (10)	0.0206 (10)	0.0075 (11)
C7	0.0605 (14)	0.0972 (19)	0.0682 (14)	0.0108 (13)	0.0297 (11)	0.0171 (13)
C9	0.0659 (14)	0.0742 (15)	0.0658 (14)	0.0021 (12)	0.0176 (11)	0.0046 (12)
C3	0.0807 (17)	0.0841 (17)	0.0457 (12)	0.0007 (14)	0.0000 (11)	-0.0033 (11)
C1	0.0592 (15)	0.097 (2)	0.096 (2)	-0.0110 (14)	-0.0053 (14)	0.0067 (17)
C11	0.091 (2)	0.0721 (17)	0.0839 (19)	0.0001 (15)	0.0011 (15)	0.0179 (14)
C6	0.0880 (19)	0.0753 (17)	0.0811 (17)	-0.0126 (14)	0.0140 (14)	0.0068 (13)
C12	0.107 (2)	0.114 (2)	0.0689 (17)	0.005 (2)	0.0211 (16)	0.0241 (17)
C10	0.0772 (17)	0.0731 (16)	0.0878 (18)	0.0086 (14)	0.0127 (14)	-0.0016 (15)
C13	0.0811 (18)	0.103 (2)	0.0721 (16)	0.0089 (16)	0.0330 (14)	0.0104 (15)
C2	0.0860 (19)	0.097 (2)	0.0756 (18)	-0.0090 (17)	-0.0194 (15)	-0.0136 (16)

Geometric parameters (Å, °)

C5—O1	1.223 (2)	C3—C2	1.397 (4)
C5—N2	1.320 (3)	C3—H3	0.9300
C5—C4	1.465 (3)	C1—C2	1.355 (4)
C4—C3	1.372 (3)	C1—H1	0.9300
C4—N1	1.381 (3)	C11—C12	1.356 (4)
N1—C1	1.356 (3)	C11—C10	1.363 (4)
N1—C7	1.462 (3)	C11—H11	0.9300
N2—C6	1.440 (3)	C6—H6A	0.9600
N2—H2A	0.86 (3)	C6—H6B	0.9600
C8—C9	1.369 (3)	C6—H6C	0.9600
C8—C13	1.376 (3)	C12—C13	1.374 (4)
C8—C7	1.502 (3)	C12—H12	0.9300
C7—H7A	0.9700	C10—H10	0.9300
C7—H7B	0.9700	C13—H13	0.9300
C9—C10	1.384 (3)	C2—H2	0.9300
C9—H9	0.9300		
O1—C5—N2	121.1 (2)	C2—C3—H3	126.1
O1—C5—C4	122.79 (19)	C2—C1—N1	109.3 (2)

N2—C5—C4	116.09 (18)	C2—C1—H1	125.4
C3—C4—N1	107.4 (2)	N1—C1—H1	125.4
C3—C4—C5	129.7 (2)	C12—C11—C10	119.5 (3)
N1—C4—C5	122.89 (17)	C12—C11—H11	120.3
C1—N1—C4	108.3 (2)	C10—C11—H11	120.3
C1—N1—C7	123.2 (2)	N2—C6—H6A	109.5
C4—N1—C7	128.4 (2)	N2—C6—H6B	109.5
C5—N2—C6	123.2 (2)	H6A—C6—H6B	109.5
C5—N2—H2A	119.7 (19)	N2—C6—H6C	109.5
C6—N2—H2A	117.1 (18)	H6A—C6—H6C	109.5
C9—C8—C13	117.8 (2)	H6B—C6—H6C	109.5
C9—C8—C7	122.8 (2)	C11—C12—C13	120.2 (3)
C13—C8—C7	119.4 (2)	C11—C12—H12	119.9
N1—C7—C8	114.28 (19)	C13—C12—H12	119.9
N1—C7—H7A	108.7	C11—C10—C9	120.4 (3)
C8—C7—H7A	108.7	C11—C10—H10	119.8
N1—C7—H7B	108.7	C9—C10—H10	119.8
C8—C7—H7B	108.7	C12—C13—C8	121.4 (3)
H7A—C7—H7B	107.6	C12—C13—H13	119.3
C8—C9—C10	120.8 (2)	C8—C13—H13	119.3
C8—C9—H9	119.6	C1—C2—C3	107.3 (2)
C10—C9—H9	119.6	C1—C2—H2	126.3
C4—C3—C2	107.7 (2)	C3—C2—H2	126.3
C4—C3—H3	126.1		
O1—C5—C4—C3	171.6 (2)	C13—C8—C9—C10	0.1 (4)
N2—C5—C4—C3	-6.1 (3)	C7—C8—C9—C10	-178.4 (2)
O1—C5—C4—N1	-7.8 (3)	N1—C4—C3—C2	0.4 (3)
N2—C5—C4—N1	174.51 (19)	C5—C4—C3—C2	-179.1 (2)
C3—C4—N1—C1	-0.8 (3)	C4—N1—C1—C2	1.0 (3)
C5—C4—N1—C1	178.7 (2)	C7—N1—C1—C2	176.9 (2)
C3—C4—N1—C7	-176.4 (2)	C10—C11—C12—C13	-0.4 (5)
C5—C4—N1—C7	3.0 (3)	C12—C11—C10—C9	0.2 (4)
O1—C5—N2—C6	-0.7 (4)	C8—C9—C10—C11	-0.1 (4)
C4—C5—N2—C6	177.0 (2)	C11—C12—C13—C8	0.5 (5)
C1—N1—C7—C8	-90.4 (3)	C9—C8—C13—C12	-0.3 (4)
C4—N1—C7—C8	84.7 (3)	C7—C8—C13—C12	178.3 (3)
C9—C8—C7—N1	-7.7 (3)	N1—C1—C2—C3	-0.7 (3)
C13—C8—C7—N1	173.8 (2)	C4—C3—C2—C1	0.2 (3)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2A \cdots O1 ⁱ	0.86 (3)	2.08 (3)	2.852 (2)	149 (2)

Symmetry code: (i) $x, -y+1/2, z+1/2$.