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Synthesis, crystal structure and Hirshfeld surface analysis of 5-cyclopropyl-*N*-(2-hydroxyethyl)-1-(4-methylphenyl)-1*H*-1,2,3-triazole-4-carboxamide

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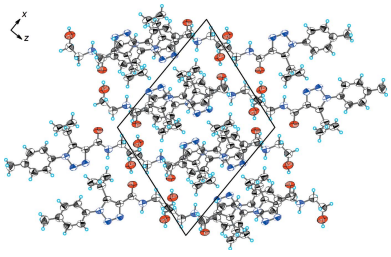
The title compound, $C_{15}H_{18}N_4O_2$, was obtained *via* a two-step synthesis (Dimroth reaction and amidation) for anticancer activity screening and was selected from a 1*H*-1,2,3-triazole-4-carboxamide library. The cyclopropyl ring is oriented almost perpendicular to the benzene ring [dihedral angle = 87.9 (1)°], while the dihedral angle between the mean plane of the cyclopropyl ring and that of the triazole ring is 55.6 (1)°. In the crystal, the molecules are linked by O—H···O and C—H···N interactions into infinite ribbons propagating in the [001] direction, which are interconnected by weak C—H···O interactions into layers. The intermolecular interactions were characterized *via* Hirshfeld surface analysis, which indicated that the largest fingerprint contact percentages are H···H (55.5%), N···H/H···N (15.4%), C···H/H···C (13.2%) and O···H/H···O (12.9%).

1. Chemical context

The 1,2,3-triazolyl-4-carboxamide motif is of great interest in drug discovery, especially in relation to anticancer and antimicrobial research. Besides the well-known drugs rufinamide and carboxyamidotriazole, several preclinical studies are ongoing. As an example of antitumour activity evaluations, libraries of 1,2,3-triazole-4-carboxamides containing podophyllotoxin (Reddy *et al.*, 2018), 1-*R*-*N*-[(1-*R*-1*H*-1,2,3-triazol-4-yl)methyl]-1*H*-1,2,3-triazole-4-carboxamides (Elamari *et al.*, 2013), 5-(trifluoromethyl)-1*H*-1,2,3-triazole-4-carboxamides (Wang *et al.*, 2018; Zhou *et al.*, 2014) and 1-benzyl-*N*-[2-(phenylamino)pyridin-3-yl]-1*H*-1,2,3-triazole-4-carboxamides (Prasad *et al.*, 2019) have been tested. Several 1,4,5-trisubstituted 1,2,3-triazole-4-carboxamides showed high affinity in the nanomolar concentration range toward Hsp90 associated with cell proliferation inhibition (Taddei *et al.*, 2014; Giannini *et al.*, 2015). Moreover, 4-[4-(hydrazinecarbonyl)-5-methyl-1*H*-1,2,3-triazol-1-yl]benzenesulfonamide was found to act as a COX-2 inhibitor (Bekheit *et al.*, 2021).

In our previous studies, new active compounds with a 1,2,3-triazolyl-4-carboxamide motif were reported (Shyyka *et al.*, 2019; Pokhodylo, Shyyka, Finiuk & Stoika, 2020; Pokhodylo, Slyvka & Pavlyuk, 2020). Additionally, 1,2,3-triazolyl-4-carboxamide derivatives were found to be inhibitors of the Wnt/β-catenin signalling pathway (Obianom *et al.*, 2019). In addition, compounds with this motif exhibited fungicidal (Wang *et al.*, 2014), antiviral (Krajczyk *et al.*, 2014) and antimicrobial (Pokhodylo *et al.*, 2021; Jadhav *et al.*, 2017) activities.



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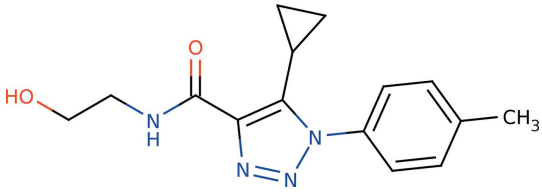
The most convenient synthetic path to diverse 1*H*-1,2,3-triazole-4-carboxamides is a two-step synthesis involving the Dimroth reaction of organic azides with β-ketoesters (Pokhodylo & Obushak, 2019) followed by amidation of the resulting 1*H*-1,2,3-triazole-4-carboxylic acids.

Given the practical interest of 1-aryl-1*H*-1,2,3-triazole-4-carboxamides in anticancer and antimicrobial research, in the present paper, we report the molecular and crystal structure of the title compound $C_{15}H_{18}N_4O_2$, highlighting its molecular conformation and analysing the intermolecular interactions. The cyclopropyl substituent was selected as it meets the criteria of lead-oriented synthesis, increasing the number of sp^3-carbon atoms, but at the same time is conformationally restricted and occupies minimal volume among other C3-alkyl substituents. Moreover, the 5-cyclopropyltriazole fragment could appear as a bisected or perpendicular conformer.



2. Structural commentary

The title compound crystallizes in the monoclinic centrosymmetric space group $P2_1/c$, with one molecule in the asymmetric unit as shown in Fig. 1. The molecular structure possesses three conformational degrees of freedom due to free rotation about the C9—C10, C8—C11 and N1—C1 single bonds. The C10/N4/O1 amide group is turned slightly relative to the N1/N2/N3/C8/C9 triazole ring by 11.71 (4)°. Within the C11/C12/C13 cyclopropyl ring, the C—C bond lengths differ by an insignificant amount [C11—C12 = 1.488 (3), C11—C13 = 1.492 (3), C12—C13 = 1.471 (3) Å]. The cyclopropyl ring is oriented almost perpendicular to the C1–C6 benzene ring and the dihedral angle between these planes is 87.9 (1)°. The dihedral angle between the mean plane of the cyclopropyl ring and that of the triazole ring is 55.6 (1)°.

A similar location of the cyclopropyl ring relative to the 1,2,3-triazole ring was also observed in 5-cyclopropyl-1-(3-methoxyphenyl)-1*H*-1,2,3-triazole-4-carboxylic acid (Pokhodylo *et al.*, 2017), but in the structure of the related compound *N*-(4-chlorophenyl)-5-cyclopropyl-1-(4-methoxyphenyl)-1*H*-1,2,3-triazole-4-carboxamide (Pokhodylo & Slyvka *et al.*, 2020), the cyclopropyl ring is close to coplanar with the aryl substituent. An intramolecular N4—H4···N3 close contact (H···N = 2.37 Å; N—H···N = 106°) is observed.

The dihedral angle between the tolyl and 1,2,3-triazole rings in the title compound is 32.75 (7)°, which is comparable with the corresponding angle in 5-cyclopropyl-1-(3-methoxyphenyl)-1*H*-1,2,3-triazole-4-carboxylic acid [39.1 (2)°] but lower than in the structure of 5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-ylphosphonate [45.36 (6)°] (Pokhodylo, Shykka, Goreshnik *et al.*, 2020). Conversely, in the triazoles unsubstituted at the 5-position, [1-(3-bromo- or 4-fluorophenyl)-1*H*-1,2,3-triazol-4-yl]methyl methylphosphonate, these angle are 22.9 (3) and 15.7 (2)°, respectively (Pokhodylo, Shyyka *et al.*, 2019).

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O1^{i}	0.95 (3)	1.78 (3)	2.734 (2)	177 (3)
C11—H11···N3^{ii}	0.98	2.61	3.391 (2)	137
C5—H5···O2^{iii}	0.93	2.66	3.564 (3)	164

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

3. Supramolecular features

As shown in Fig. 2 and Table 1, the extended structure of the title compound features a number of directional intermolecular interactions. The molecules are linked by O2—H2···O1^{i} and C11—H11···N3^{ii} (see Table 1 for symmetry codes) interactions into an infinite ribbon propagating in the [001] direction. The ribbons are interconnected by a weak C5—H5···O2^{iii} interaction into layers (Fig. 3).

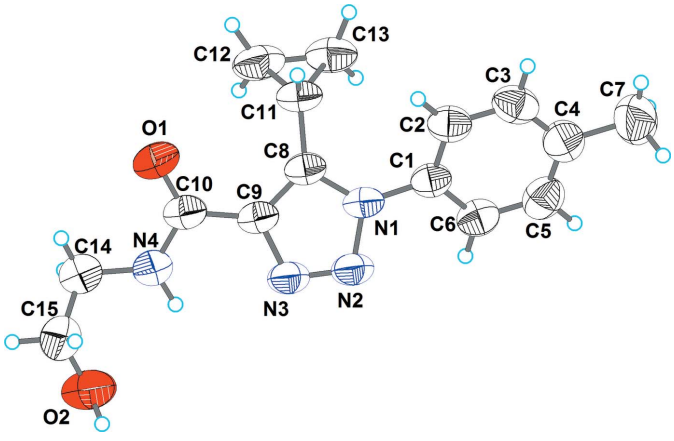


Figure 1
The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level.

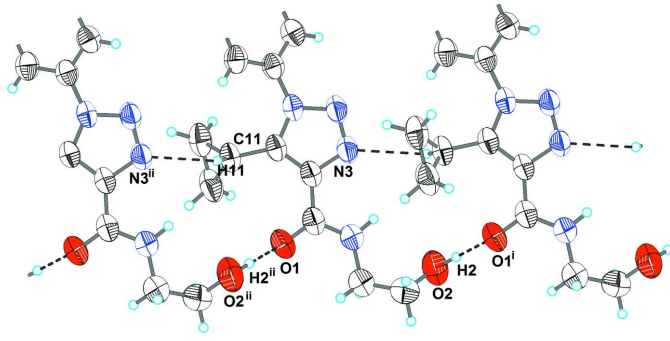


Figure 2
The hydrogen-bonded ribbon in the title compound. Hydrogen bonds are shown as dashed lines. The symmetry codes are as in Table 1.

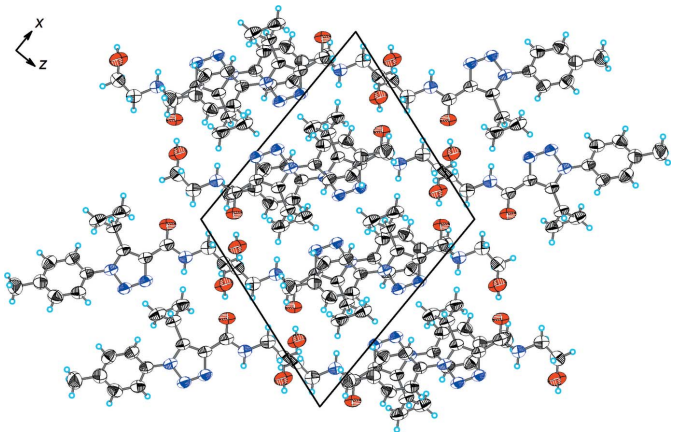


Figure 3
A view along the *b*-axis direction of the crystal packing of the title compound.

4. Hirshfeld surface analysis

The significant interactions among the molecules of the title compound can be visualized qualitatively through Hirshfeld surface analysis (Spackman & Jayatilaka, 2009). The mapping of the normalized contact distance (d_{norm}) was performed using the *CrystalExplorer* software (Turner *et al.*, 2017). The most prominent interactions (short contact areas) are indicated on the Hirshfeld surfaces in red, whereas long contacts are shown in blue. Fingerprint plots were produced to show the intermolecular surface bond distances with the regions highlighted for O···H/H···O and N···H/H···N interactions (Fig. 4). The contributions to the surface area for such contacts are 12.9% and 15.4%, respectively. The relatively low percentage of C···H/H···C contacts (13.2%) indicates the small contribution of C—H···π interactions for consolidating the crystal packing. The contribution to the surface area for H···H contacts is 55.5%.

5. Database survey

The most closest related compounds containing a similar 1-aryl-1*H*-1,2,3-triazole-4-carboxamide skeleton to the title compound but with different substituents on the amide are: *N*-(4-chlorophenyl)-5-cyclopropyl-1-(4-methoxyphenyl)-1*H*-1,2,3-triazole-4-carboxamide (Pokhodylo & Slyvka *et al.*, 2020), (*S*)-1-(4-chlorophenyl)-*N*-(1-hydroxy-3-phenylpropan-2-yl)-5-methyl-1*H*-1,2,3-triazole-4-carboxamide (I) [Cambridge Structural Database (Version 2021.1; Groom *et al.*, 2016) refcode ZIPSEY; Shen *et al.*, 2013], 1-(4-chlorophenyl)-5-methyl-*N*-[(3-phenyl-1,2-oxazol-5-yl)methyl]-1*H*-1,2,3-triazole-4-carboxamide (II) (LELHOB; Niu *et al.*, 2013), (5-methyl-1-[8-(trifluoromethyl)quinolin-4-yl]-1*H*-1,2,3-triazol-4-yl)morpholino)methanone (III) (LOHWIP; Anuradha *et al.*, 2008) and 1-(3-amino-5-(3-hydroxy-3-methylbut-1-yn-1-yl)phenyl)-*N*-butyl-1*H*-1,2,3-triazole-4-carboxamide (IV) (BEBJEZ; Li *et al.*, 2012).

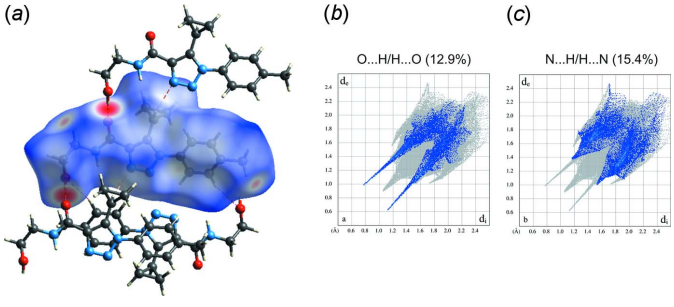


Figure 4
(*a*) Hirshfeld surface for the title compound mapped with d_{norm} over the range −0.68 to 1.46 showing the O—H···O, C—H···N and C—H···O hydrogen-bonded contacts. Fingerprint plots resolved into (*b*) O···H/H···O and (*c*) N···H/H···N contacts. Neighbouring molecules associated with close contacts are also shown.

Compounds (I) and (II) crystallize in the monoclinic crystal system with space groups $P2_1$ and $P2_1/c$, respectively, while compounds (III) and (IV) crystallize in the triclinic space group $P\overline{1}$. Structure (I) contains two crystallographically independent molecules, the hydroxyl groups of which participate in intermolecular O—H···O hydrogen bonds. In contrast to the molecular structure of title compound, the torsion angles between the phenyl rings and triazole rings in (I) are −45.2 (6)° (C5—C6—N1—N2) and 39.9 (6)° (C1′—C6′—N1′—N2′); the analogous value in (II) is 19.2 (2)°. In structure (II), the carboxamide groups connect neighbouring molecules into infinite chains by means of N—H···O hydrogen bonds. The molecules in structures (III) and (IV) are connected by N—H···O(oxazol) contacts. Similarly to (I) and (II), structure (III) contains a 5-methyl substituent at the triazole ring; as a result of the significant steric hindrance of 8-(trifluoromethyl)quinoline, the dihedral angle between the rings is 54.7°. The phenyl and triazole rings in (IV) are close to coplanar (7.5°), while the hydroxyl, carboxamide and amino groups participate in O—H···O and N—H···O hydrogen bonds. Finally, two copper(I) π-complexes of compositions $[Cu(C_{12}H_{13}N_5O)(NO_3)]\cdot 0.5H_2O$ and $[Cu(C_{12}H_{13}N_5O)(CF_3COO)]$ ($C_{12}H_{13}N_5O$ is *N*-allyl-5-amino-1-phenyl-1*H*-1,2,3-triazole-4-carboxamide) were obtained by electrochemical synthesis (ZEQTOG and ZEQTUM; Slyvka *et al.*, 2012). Crystals of these compounds are monoclinic, space group *C*2/*c*: in both structures, the *N*-allyl-1*H*-1,2,3-triazole-4-carboxamide motif acts as a bridging chelating ligand and forms with the copper(I) atoms infinite chains containing $[CuC_4NO]$ seven-membered rings.

6. Synthesis and crystallization

5-Cyclopropyl-1-*p*-tolyl-1*H*-1,2,3-triazole-4-carboxylic acid (Pokhodylo *et al.*, 2017) (1.22 g, 5.00 mmol) was added to a solution of 1,1′-carbonyldiimidazole (CDI, 0.81 g, 5.0 mmol) in dry acetonitrile (5 ml) and the mixture was kept for 30 min at 323 K. Then, 0.3 ml of 2-aminoethanol (0.31 g, 5.00 mmol) was added, and the mixture was heated at 343 K for 1 h. After cooling to room temperature, water (30 ml) was added. The precipitate was filtered off, washed with water on a filter, crystallized from diluted ethanol solution, and dried in air to give the title compound as colourless crystals, m.p. 396–397 K.

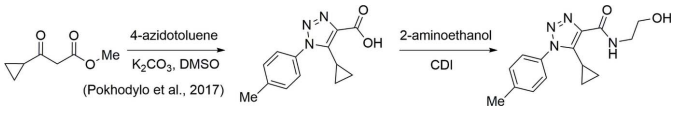


Figure 5
Synthesis of 5-cyclopropyl-*N*-(2-hydroxyethyl)-1-(*p*-tolyl)-1*H*-1,2,3-triazole-4-carboxamide.

The reaction scheme is shown in Fig. 5. IR (KBr, ν, cm^{-1}): 1685 (C═O); 3370 (N—H). ^{1}H NMR: (400 MHz, DMSO-d_6): δ = 0.85–0.91 (*m*, 2H, CH_2), 0.98–1.02 (*m*, 2H, CH_2), 1.95–1.99 (*m*, 1H, CH), 2.46 (*c*, 3H, CH_3), 3.37 (*q*, *J* = 5.8 Hz, 2H, CH_2N), 3.54 (*q*, *J* = 5.8 Hz, 2H, CH_2O), 4.58 (*t*, *J* = 6.0, Hz, 1H, OH), 7.37 (*d*, *J* = 7.6 Hz, 2H, H_{Ar}-3,5), 7.43 (*d*, *J* = 7.6 Hz, 2H, H_{Ar}-2,6), 8.14 (*t*, *J* = 5.4 Hz, 1H, NH). ^{13}C NMR: (101 MHz, DMSO-d_6): δ = 5.3 (CH), 8.2 (2 × CH_2), 21.1 (CH_3), 42.3 (CH_2N), 59.5 (CH_2O), 126.5 (2 × CH_{Ar}-2,6), 130.1 (2 × CH_{Ar}-3,5), 133.7 (C_{Ar}-1), 137.2 ($C_{Triazole}$-4), 139.2 (C_{Ar}-4), 144.6 ($C_{Triazole}$-5), 161.8 (C═O). MS, *m*/*z* = 287 (M^++1). Calculated for $C_{15}H_{18}N_4O_2$, (%): C 62.92; H 6.34, N 19.57. Found (%): C 62.83; H 6.57, N 19.32.

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. N-bound and O-bound H atoms were located in difference-Fourier maps and refined isotropically. C-bound H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93–0.98 Å and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C-methyl).

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{18}N_4O_2$
M_r	286.33
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	293
a, *b*, *c* (Å)	14.3158 (6), 8.3972 (3), 13.0871 (4)
β (°)	108.040 (4)
V (Å^3)	1495.90 (10)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.5 × 0.4 × 0.06
Data collection	
Diffractometer	Oxford Diffraction Xcalibur3 CCD
Absorption correction	Multi-scan (*CrysAlis RED*; Oxford Diffraction, 2004)
T_{min}, T_{max}	0.935, 0.988
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8400, 2632, 1621
R_{int}	0.032
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.047, 0.099, 1.05
No. of reflections	2632
No. of parameters	199
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.14, −0.20

Computer programs: *CrysAlis CCD* and *CrysAlis RED* (Oxford Diffraction, 2004), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

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supporting information

Acta Cryst. (2021). E**77**, 1043-1047 [https://doi.org/10.1107/S2056989021009774]

Synthesis, crystal structure and Hirshfeld surface analysis of 5-cyclopropyl-*N*-(2-hydroxyethyl)-1-(4-methylphenyl)-1*H*-1,2,3-triazole-4-carboxamide

Nazariy T. Pokhodylo, Yurii Slyvka and Volodymyr Pavlyuk

Computing details

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2004); cell refinement: *CrysAlis CCD* (Oxford Diffraction, 2004); data reduction: *CrysAlis RED* (Oxford Diffraction, 2004); program(s) used to solve structure: ShelXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015b); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

5-Cyclopropyl-*N*-(2-hydroxyethyl)-1-(4-methylphenyl)-1*H*-1,2,3-triazole-4-carboxamide

Crystal data

$C_{15}H_{18}N_4O_2$
$M_r = 286.33$
Monoclinic, $P2_1/c$
$a = 14.3158$ (6) Å
$b = 8.3972$ (3) Å
$c = 13.0871$ (4) Å
$\beta = 108.040$ (4)°
$V = 1495.90$ (10) Å^3
$Z = 4$
$F(000) = 608$
$D_x = 1.271$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1679 reflections
$\theta = 2.9$–26.4°
$\mu = 0.09$ mm^{-1}
$T = 293$ K
Lamina, clear colourless
0.5 × 0.4 × 0.06 mm

Data collection

Oxford Diffraction Xcalibur3 CCD diffractometer
ω scans
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2004)
$T_{min} = 0.935$, $T_{max} = 0.988$
8400 measured reflections
2632 independent reflections
1621 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.9°$
$h = -17\rightarrow17$
$k = -9\rightarrow9$
$l = -15\rightarrow8$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.099$
$S = 1.05$
2632 reflections
199 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.035P)^2 + 0.180P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.14$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ 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. 1. Fixed Uiso At 1.2 times of: All C(H) groups, All C(H,H) groups At 1.5 times of: All C(H,H,H) groups 2.a Ternary CH refined with riding coordinates: C11(H11) 2.b Secondary CH2 refined with riding coordinates: C12(H12A,H12B), C13(H13A,H13B), C14(H14A,H14B), C15(H15A,H15B) 2.c Aromatic/amide H refined with riding coordinates: C2(H2A), C3(H3), C5(H5), C6(H6) 2.d Idealised Me refined as rotating group: C7(H7A,H7B,H7C)

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.13456 (11)	0.3294 (2)	0.60427 (12)	0.0974 (5)
O2	0.06831 (14)	0.3928 (2)	0.21808 (14)	0.1042 (6)
H2	0.092 (2)	0.318 (4)	0.177 (3)	0.171 (14)*
N1	0.44653 (12)	0.35154 (18)	0.67420 (11)	0.0570 (4)
N2	0.43955 (13)	0.3492 (2)	0.56776 (12)	0.0687 (5)
N3	0.34622 (13)	0.3512 (2)	0.51377 (12)	0.0680 (5)
N4	0.14491 (15)	0.3789 (3)	0.44072 (15)	0.0846 (6)
H4	0.1840 (16)	0.397 (3)	0.4005 (17)	0.090 (7)*
C1	0.54283 (14)	0.3518 (2)	0.75104 (14)	0.0573 (5)
C2	0.56125 (17)	0.2755 (2)	0.84840 (16)	0.0660 (6)
H2A	0.510965	0.223701	0.866145	0.079*
C3	0.65554 (19)	0.2771 (3)	0.91933 (17)	0.0755 (6)
H3	0.667851	0.226965	0.985570	0.091*
C4	0.73224 (17)	0.3510 (3)	0.89461 (18)	0.0754 (6)
C5	0.71121 (17)	0.4244 (3)	0.79535 (19)	0.0768 (6)
H5	0.761557	0.474207	0.776551	0.092*
C6	0.61784 (16)	0.4254 (2)	0.72389 (17)	0.0658 (5)
H6	0.605361	0.475465	0.657611	0.079*
C7	0.83533 (18)	0.3513 (4)	0.9729 (2)	0.1156 (10)
H7A	0.853395	0.457928	0.997559	0.173*
H7B	0.880342	0.311663	0.937725	0.173*
H7C	0.837630	0.284323	1.033110	0.173*
C8	0.35570 (14)	0.3551 (2)	0.68719 (14)	0.0546 (5)
C9	0.29293 (14)	0.3532 (2)	0.58406 (14)	0.0583 (5)
C10	0.18463 (16)	0.3519 (2)	0.54485 (16)	0.0671 (5)
C11	0.33673 (14)	0.3634 (2)	0.79130 (14)	0.0616 (5)
H11	0.329955	0.260032	0.823068	0.074*
C12	0.27363 (19)	0.4924 (3)	0.81202 (17)	0.0882 (7)
H12A	0.249069	0.572020	0.756496	0.106*
H12B	0.228996	0.465129	0.851979	0.106*
C13	0.37951 (19)	0.4924 (3)	0.87037 (17)	0.0791 (7)
H13A	0.399859	0.464995	0.946092	0.095*
H13B	0.419936	0.571906	0.850592	0.095*
C14	0.03984 (18)	0.3804 (3)	0.38523 (18)	0.0979 (8)
H14A	0.016031	0.489258	0.378087	0.117*

H14B	0.005972	0.321759	0.427025	0.117*
C15	0.01844 (17)	0.3080 (3)	0.27817 (18)	0.0946 (8)
H15A	0.039484	0.197635	0.285111	0.114*
H15B	−0.051722	0.310769	0.241585	0.114*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0884 (11)	0.1413 (16)	0.0775 (10)	−0.0021 (10)	0.0476 (9)	0.0143 (9)
O2	0.1267 (15)	0.1162 (15)	0.0851 (12)	−0.0073 (11)	0.0552 (11)	−0.0083 (10)
N1	0.0729 (11)	0.0568 (10)	0.0487 (9)	−0.0034 (8)	0.0296 (8)	−0.0040 (8)
N2	0.0769 (13)	0.0840 (13)	0.0532 (10)	−0.0042 (10)	0.0317 (9)	−0.0090 (9)
N3	0.0760 (13)	0.0828 (13)	0.0525 (9)	−0.0047 (10)	0.0302 (9)	−0.0081 (8)
N4	0.0674 (13)	0.1313 (18)	0.0594 (12)	−0.0073 (11)	0.0258 (10)	0.0050 (11)
C1	0.0700 (14)	0.0492 (12)	0.0575 (12)	0.0002 (10)	0.0271 (11)	−0.0064 (10)
C2	0.0834 (17)	0.0572 (13)	0.0609 (13)	−0.0025 (11)	0.0273 (12)	−0.0031 (10)
C3	0.0962 (19)	0.0669 (15)	0.0621 (13)	0.0136 (13)	0.0228 (14)	−0.0030 (11)
C4	0.0750 (16)	0.0746 (15)	0.0752 (15)	0.0140 (13)	0.0209 (12)	−0.0165 (13)
C5	0.0738 (17)	0.0738 (16)	0.0895 (17)	0.0014 (12)	0.0349 (14)	−0.0096 (13)
C6	0.0758 (15)	0.0599 (13)	0.0684 (13)	0.0026 (11)	0.0322 (12)	0.0003 (10)
C7	0.0830 (19)	0.155 (3)	0.1007 (19)	0.0267 (17)	0.0160 (15)	−0.0231 (18)
C8	0.0729 (13)	0.0448 (11)	0.0535 (11)	−0.0021 (10)	0.0304 (10)	−0.0007 (9)
C9	0.0719 (14)	0.0575 (13)	0.0538 (12)	−0.0048 (10)	0.0315 (10)	−0.0014 (10)
C10	0.0796 (15)	0.0702 (14)	0.0591 (13)	−0.0042 (12)	0.0328 (12)	−0.0001 (11)
C11	0.0893 (15)	0.0516 (12)	0.0536 (11)	−0.0055 (11)	0.0364 (10)	−0.0003 (10)
C12	0.116 (2)	0.0920 (18)	0.0739 (15)	0.0236 (15)	0.0541 (15)	0.0002 (12)
C13	0.116 (2)	0.0663 (15)	0.0657 (13)	−0.0099 (13)	0.0447 (14)	−0.0104 (11)
C14	0.0787 (18)	0.147 (2)	0.0738 (15)	0.0057 (15)	0.0315 (13)	−0.0002 (15)
C15	0.0722 (16)	0.125 (2)	0.0884 (18)	−0.0097 (14)	0.0268 (14)	−0.0072 (15)

Geometric parameters (Å, º)

O1—C10	1.224 (2)	C6—H6	0.9300
O2—H2	0.95 (3)	C7—H7A	0.9600
O2—C15	1.408 (3)	C7—H7B	0.9600
N1—N2	1.3655 (19)	C7—H7C	0.9600
N1—C1	1.433 (2)	C8—C9	1.370 (2)
N1—C8	1.363 (2)	C8—C11	1.471 (2)
N2—N3	1.304 (2)	C9—C10	1.475 (3)
N3—C9	1.365 (2)	C11—H11	0.9800
N4—H4	0.89 (2)	C11—C12	1.488 (3)
N4—C10	1.324 (3)	C11—C13	1.492 (3)
N4—C14	1.454 (3)	C12—H12A	0.9700
C1—C2	1.377 (3)	C12—H12B	0.9700
C1—C6	1.378 (3)	C12—C13	1.471 (3)
C2—H2A	0.9300	C13—H13A	0.9700
C2—C3	1.381 (3)	C13—H13B	0.9700
C3—H3	0.9300	C14—H14A	0.9700

C3—C4	1.384 (3)	C14—H14B	0.9700
C4—C5	1.384 (3)	C14—C15	1.470 (3)
C4—C7	1.514 (3)	C15—H15A	0.9700
C5—H5	0.9300	C15—H15B	0.9700
C5—C6	1.374 (3)		

C15—O2—H2	108 (2)	N3—C9—C8	109.33 (17)
N2—N1—C1	117.80 (15)	N3—C9—C10	120.82 (17)
C8—N1—N2	110.87 (15)	C8—C9—C10	129.85 (16)
C8—N1—C1	131.32 (15)	O1—C10—N4	122.1 (2)
N3—N2—N1	106.95 (14)	O1—C10—C9	122.64 (19)
N2—N3—C9	109.13 (15)	N4—C10—C9	115.29 (17)
C10—N4—H4	119.3 (14)	C8—C11—H11	115.0
C10—N4—C14	124.32 (19)	C8—C11—C12	119.99 (17)
C14—N4—H4	116.4 (14)	C8—C11—C13	121.51 (16)
C2—C1—N1	121.04 (18)	C12—C11—H11	115.0
C2—C1—C6	120.41 (19)	C12—C11—C13	59.16 (14)
C6—C1—N1	118.51 (17)	C13—C11—H11	115.0
C1—C2—H2A	120.5	C11—C12—H12A	117.7
C1—C2—C3	119.0 (2)	C11—C12—H12B	117.7
C3—C2—H2A	120.5	H12A—C12—H12B	114.8
C2—C3—H3	119.1	C13—C12—C11	60.56 (14)
C2—C3—C4	121.9 (2)	C13—C12—H12A	117.7
C4—C3—H3	119.1	C13—C12—H12B	117.7
C3—C4—C7	121.3 (2)	C11—C13—H13A	117.7
C5—C4—C3	117.5 (2)	C11—C13—H13B	117.7
C5—C4—C7	121.2 (2)	C12—C13—C11	60.28 (14)
C4—C5—H5	119.2	C12—C13—H13A	117.7
C6—C5—C4	121.7 (2)	C12—C13—H13B	117.7
C6—C5—H5	119.2	H13A—C13—H13B	114.9
C1—C6—H6	120.2	N4—C14—H14A	109.5
C5—C6—C1	119.6 (2)	N4—C14—H14B	109.5
C5—C6—H6	120.2	N4—C14—C15	110.52 (19)
C4—C7—H7A	109.5	H14A—C14—H14B	108.1
C4—C7—H7B	109.5	C15—C14—H14A	109.5
C4—C7—H7C	109.5	C15—C14—H14B	109.5
H7A—C7—H7B	109.5	O2—C15—C14	109.4 (2)
H7A—C7—H7C	109.5	O2—C15—H15A	109.8
H7B—C7—H7C	109.5	O2—C15—H15B	109.8
N1—C8—C9	103.70 (15)	C14—C15—H15A	109.8
N1—C8—C11	124.99 (17)	C14—C15—H15B	109.8
C9—C8—C11	131.29 (17)	H15A—C15—H15B	108.3

N1—N2—N3—C9	−0.6 (2)	C2—C3—C4—C5	0.0 (3)
N1—C1—C2—C3	179.09 (16)	C2—C3—C4—C7	−179.8 (2)
N1—C1—C6—C5	−178.65 (17)	C3—C4—C5—C6	0.5 (3)
N1—C8—C9—N3	−1.0 (2)	C4—C5—C6—C1	0.0 (3)
N1—C8—C9—C10	178.57 (19)	C6—C1—C2—C3	1.5 (3)

N1—C8—C11—C12	125.2 (2)	C7—C4—C5—C6	−179.7 (2)
N1—C8—C11—C13	55.1 (3)	C8—N1—N2—N3	0.0 (2)
N2—N1—C1—C2	−146.14 (17)	C8—N1—C1—C2	34.7 (3)
N2—N1—C1—C6	31.5 (2)	C8—N1—C1—C6	−147.61 (19)
N2—N1—C8—C9	0.61 (19)	C8—C9—C10—O1	−10.8 (3)
N2—N1—C8—C11	−177.98 (16)	C8—C9—C10—N4	168.3 (2)
N2—N3—C9—C8	1.0 (2)	C8—C11—C12—C13	−111.0 (2)
N2—N3—C9—C10	−178.57 (17)	C8—C11—C13—C12	108.5 (2)
N3—C9—C10—O1	168.7 (2)	C9—C8—C11—C12	−53.0 (3)
N3—C9—C10—N4	−12.2 (3)	C9—C8—C11—C13	−123.1 (2)
N4—C14—C15—O2	−58.8 (3)	C10—N4—C14—C15	−141.9 (2)
C1—N1—N2—N3	−179.30 (16)	C11—C8—C9—N3	177.49 (18)
C1—N1—C8—C9	179.77 (18)	C11—C8—C9—C10	−3.0 (3)
C1—N1—C8—C11	1.2 (3)	C14—N4—C10—O1	−1.9 (3)
C1—C2—C3—C4	−1.0 (3)	C14—N4—C10—C9	179.0 (2)
C2—C1—C6—C5	−1.0 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O1^{i}	0.95 (3)	1.78 (3)	2.734 (2)	177 (3)
C11—H11···N3^{ii}	0.98	2.61	3.391 (2)	137
C5—H5···O2^{iii}	0.93	2.66	3.564 (3)	164

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