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10,11-Dihydrocarbamazepine (form III)

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.041
wR factor = 0.101
Data-to-parameter ratio = 12.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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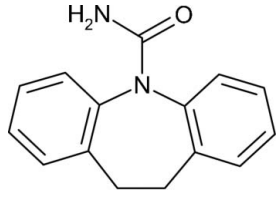
The title compound (systematic name: 10,11-dihydro-5*H*-dibenz[*b*,*f*]azepine-5-carboxamide), $C_{15}H_{14}N_2O$, is shown to crystallize as a triclinic polymorph with $Z' = 2$. N—H···O and N—H···π interactions combine to create a catemeric motif. The robustness of this motif is reflected in the fact that it is also observed in the previously published monoclinic and orthorhombic forms of the compound.

Comment

Dihydrocarbamazepine (DHC), (I), is a recognized impurity in carbamazepine, a dibenzazepine drug used to control seizures (Cyr *et al.*, 1987). DHC is known to crystallize in three polymorphic forms: monoclinic form I [$P2_1/c$; a = 5.505 (1) Å, b = 9.158 (2) Å, c = 24.266 (7) Å, β = 95.95 (2)° at T = 294 K; Bandoli *et al.*, 1992], orthorhombic form II [*Pbca*; a = 9.0592 (4) Å, b = 10.3156 (5) Å, c = 25.0534 (12) Å at T = 120 K; Harrison *et al.*, 2006] and triclinic form III (present work). It also forms a 1:1 solvate with acetic acid (Johnston *et al.*, 2006). The work reported here forms part of a wider investigation that couples automated parallel crystallization (Florence, Johnston, Fernandes *et al.*, 2006) with crystal structure prediction methodology to investigate the basic science underlying the solid-state diversity of carbamazepine and its analogues (Florence, Johnston, Price *et al.*, 2006).



(I)

There are two independent molecules in DHC form III (Fig. 1). The intermolecular interactions combine to create the catemeric motif shown in Fig. 2, with the geometric parameters listed in Table 1. Infinite [0$\bar{1}$0] chains of DHC molecules are linked by hydrogen bonds N4—H4*B*···O1 and N2—H2*B*···O2^{i} [symmetry code: (i) x, $y-1$, z], supplemented by N—H···π interactions, N2—H2*A*···*Cg*4 and N4—H4*A*···*Cg*2^{ii} [symmetry code: (ii) x, $y+1$, z], where *Cg*4 is the centroid of ring *R*4 (C29–C34) and *Cg*2 is the centroid of ring *R*2 (C9–C14). The robustness of this motif is reflected in the fact that it is observed in DHC form II [Fig. 2 of Harrison *et al.* (2006)], DHC form I [Fig. 3 of Bandoli *et al.* (1992)] and in a predicted carbamazepine crystal structure that is isostructural with DHC form II [Fig. 2 of Florence, Leech *et al.* (2006)]. This

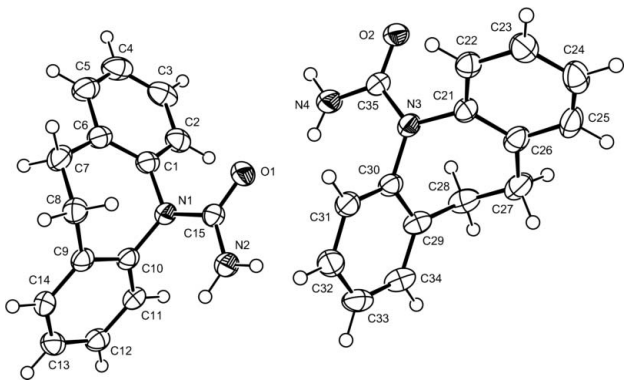


Figure 1
The asymmetric unit of DHC form III with 50% probability displacement ellipsoids.

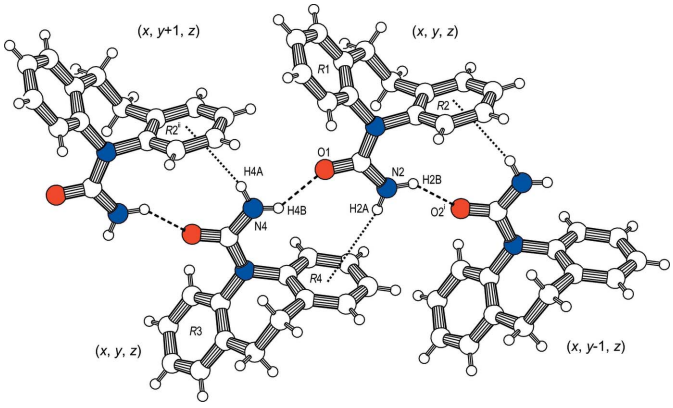


Figure 2
The DHC catemer in form III. Dashed and dotted lines indicate N—H···O and N—H···π interactions, respectively.

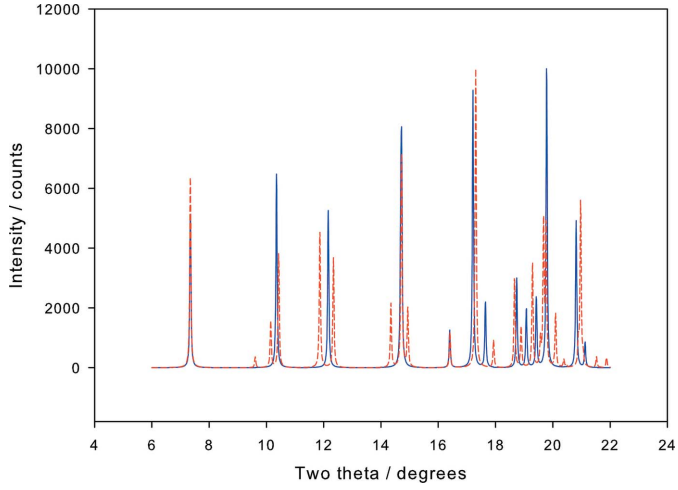


Figure 3
Calculated powder diffraction patterns (λ = 1.54 Å) for DHC form I (blue solid line) and form III (red dashed line).

motif is also observed in the crystal structure of cyheptamide (Leech *et al.*, 2007), an analogue of DHC.

The structures of DHC forms I and III are closely related, but certainly distinct, and there is no evidence of missing symmetry in the form III structure [using the *ADDSYM* algorithm in *PLATON* (Spek, 2003)]. Powder patterns calculated from single-crystal structures offer an effective means of distinguishing polymorphs (Karami *et al.*, 2006) and, in this case, the calculated patterns are quite different, reflecting the small but significant differences in both the lattice parameters and the atomic positions (Fig. 3).

Experimental

DHC was recrystallized from methanol solution by slow evaporation at room temperature to yield single crystals of form I (blocks), form II (hexagonal plates) and form III (needles).

Crystal data

$C_{15}H_{14}N_2O$
M_r = 238.28
Triclinic, $P\overline{1}$
a = 5.4233 (12) Å
b = 9.200 (5) Å
c = 24.189 (6) Å
α = 87.59 (3)°
β = 84.23 (2)°
γ = 88.93 (3)°
V = 1199.6 (8) Å^3
Z = 4
D_x = 1.319 Mg m^{-3}
Cu $K\alpha$ radiation
μ = 0.67 mm^{-1}
T = 150 (2) K
Needle, colourless
0.22 × 0.07 × 0.07 mm

Data collection

Oxford Diffraction Gemini diffractometer
ω and φ scans
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006)
T_{min} = 0.867, T_{max} = 0.955
12410 measured reflections
4297 independent reflections
2327 reflections with $I > 2\sigma(I)$
R_{int} = 0.044
θ_{max} = 67.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.101
S = 0.84
4297 reflections
341 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0472P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2B···O2^{i}	0.92 (2)	2.02 (3)	2.800 (3)	142.7 (19)
N4—H4B···O1	0.86 (2)	2.11 (3)	2.801 (3)	137.4 (19)
N2—H2A···Cg4	0.89 (3)	3.01 (3)	3.862 (3)	162 (2)
N4—H4A···Cg2^{ii}	0.90 (3)	2.89 (3)	3.765 (3)	166 (2)

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

The amide H atoms were located in difference maps and their coordinates and U_{iso} parameters refined freely. All other H atoms were constrained to geometrically sensible positions in a riding model, with C—H = 0.95–0.99 Å and with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduc-

tion: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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supporting information

Acta Cryst. (2007). E**63**, o675–o677 [https://doi.org/10.1107/S1600536806053335]

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$M_r = 238.28$
Triclinic, $P\overline{1}$
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$a = 5.4233$ (12) Å
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$\alpha = 87.59$ (3)°
$\beta = 84.23$ (2)°
$\gamma = 88.93$ (3)°
$V = 1199.6$ (8) Å^3
$Z = 4$
$F(000) = 504$
$D_x = 1.319$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 2529 reflections
$\theta = 3.7$–72.8°
$\mu = 0.67$ mm^{-1}
$T = 150$ K
Needle, colourless
0.22 × 0.07 × 0.07 mm

Data collection

Oxford Diffraction Gemini diffractometer
Radiation source: Enhance (Cu) X-ray Source
Graphite monochromator
Detector resolution: 15.9745 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2006)
$T_{min} = 0.867$, $T_{max} = 0.955$
12410 measured reflections
4297 independent reflections
2327 reflections with $I > 2\sigma(I)$
$R_{int} = 0.044$
$\theta_{max} = 67.5°$, $\theta_{min} = 3.7°$
$h = -5\rightarrow6$
$k = -11\rightarrow10$
$l = -28\rightarrow28$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.101$
$S = 0.84$
4297 reflections
341 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
$w = 1/[\sigma^2(F_o^2) + (0.0472P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	−0.1609 (3)	0.31475 (16)	0.28334 (6)	0.0399 (4)
N1	−0.3307 (4)	0.11654 (19)	0.33152 (7)	0.0324 (4)
N2	−0.0524 (4)	0.0916 (2)	0.25291 (8)	0.0426 (5)
H2A	0.060 (6)	0.131 (3)	0.2277 (12)	0.069 (9)*
H2B	−0.042 (4)	−0.008 (3)	0.2558 (9)	0.034 (7)*
C1	−0.4478 (4)	0.2066 (2)	0.37405 (9)	0.0340 (5)
C2	−0.6367 (5)	0.3015 (2)	0.35965 (10)	0.0395 (6)
H2	−0.6800	0.3065	0.3225	0.047*
C3	−0.7613 (5)	0.3880 (3)	0.39872 (12)	0.0473 (7)
H3	−0.8903	0.4522	0.3887	0.057*
C4	−0.6964 (5)	0.3805 (3)	0.45266 (11)	0.0530 (8)
H4	−0.7793	0.4407	0.4798	0.064*
C5	−0.5110 (5)	0.2856 (3)	0.46687 (10)	0.0477 (7)
H5	−0.4709	0.2804	0.5042	0.057*
C6	−0.3802 (5)	0.1967 (2)	0.42832 (9)	0.0364 (6)
C7	−0.1833 (5)	0.0965 (3)	0.44960 (9)	0.0419 (6)
H7A	−0.2666	0.0288	0.4781	0.050*
H7B	−0.0712	0.1566	0.4687	0.050*
C8	−0.0225 (4)	0.0054 (2)	0.40825 (9)	0.0368 (5)
H8A	0.0679	0.0707	0.3799	0.044*
H8B	0.1015	−0.0509	0.4280	0.044*
C9	−0.1762 (4)	−0.0973 (2)	0.38008 (9)	0.0326 (5)
C10	−0.3336 (4)	−0.0384 (2)	0.34217 (9)	0.0314 (5)
C11	−0.4906 (4)	−0.1256 (2)	0.31713 (9)	0.0327 (5)
H11	−0.5975	−0.0840	0.2918	0.039*
C12	−0.4914 (4)	−0.2740 (2)	0.32916 (9)	0.0363 (6)
H12	−0.5995	−0.3344	0.3121	0.044*
C13	−0.3358 (5)	−0.3342 (3)	0.36575 (9)	0.0390 (6)
H13	−0.3369	−0.4362	0.3738	0.047*
C14	−0.1768 (4)	−0.2468 (2)	0.39110 (9)	0.0353 (5)
H14	−0.0685	−0.2894	0.4160	0.042*
C15	−0.1761 (4)	0.1812 (2)	0.28836 (9)	0.0314 (5)
O2	0.1329 (3)	0.81843 (16)	0.22084 (6)	0.0360 (4)
N3	0.3008 (4)	0.63384 (19)	0.16859 (7)	0.0317 (4)
N4	0.0134 (4)	0.5889 (2)	0.24465 (9)	0.0426 (5)

H4B	0.015 (4)	0.496 (3)	0.2416 (9)	0.036 (7)*
H4A	−0.077 (5)	0.626 (3)	0.2740 (11)	0.047 (7)*
C21	0.4437 (4)	0.7339 (2)	0.13104 (9)	0.0334 (5)
C22	0.6176 (4)	0.8181 (2)	0.15319 (9)	0.0340 (5)
H22	0.6339	0.8105	0.1919	0.041*
C23	0.7667 (5)	0.9128 (2)	0.11940 (10)	0.0424 (6)
H23	0.8865	0.9691	0.1346	0.051*
C24	0.7391 (5)	0.9241 (3)	0.06339 (11)	0.0462 (7)
H24	0.8374	0.9906	0.0399	0.055*
C25	0.5707 (5)	0.8400 (3)	0.04142 (10)	0.0451 (7)
H25	0.5588	0.8477	0.0025	0.054*
C26	0.4141 (5)	0.7425 (3)	0.07420 (9)	0.0373 (6)
C27	0.2361 (5)	0.6556 (3)	0.04489 (9)	0.0462 (7)
H27A	0.3361	0.5938	0.0183	0.055*
H27B	0.1409	0.7252	0.0227	0.055*
C28	0.0496 (5)	0.5573 (3)	0.07958 (10)	0.0446 (6)
H28A	−0.0582	0.6171	0.1054	0.053*
H28B	−0.0568	0.5110	0.0546	0.053*
C29	0.1772 (4)	0.4415 (3)	0.11223 (9)	0.0379 (6)
C30	0.3084 (4)	0.4827 (2)	0.15580 (9)	0.0323 (5)
C31	0.4467 (4)	0.3825 (2)	0.18449 (9)	0.0347 (5)
H31	0.5378	0.4129	0.2134	0.042*
C32	0.4515 (5)	0.2380 (3)	0.17082 (10)	0.0410 (6)
H32	0.5462	0.1688	0.1903	0.049*
C33	0.3174 (5)	0.1941 (3)	0.12854 (10)	0.0431 (6)
H33	0.3190	0.0947	0.1194	0.052*
C34	0.1818 (5)	0.2949 (3)	0.09973 (10)	0.0422 (6)
H34	0.0903	0.2639	0.0710	0.051*
C35	0.1453 (4)	0.6875 (2)	0.21248 (9)	0.0313 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0472 (11)	0.0317 (9)	0.0390 (9)	−0.0038 (7)	0.0038 (8)	0.0007 (7)
N1	0.0380 (11)	0.0314 (10)	0.0269 (9)	−0.0025 (8)	0.0013 (8)	−0.0021 (7)
N2	0.0540 (15)	0.0362 (13)	0.0346 (11)	−0.0022 (10)	0.0115 (10)	−0.0024 (9)
C1	0.0359 (13)	0.0307 (12)	0.0342 (12)	−0.0077 (10)	0.0047 (10)	−0.0034 (9)
C2	0.0373 (14)	0.0347 (13)	0.0457 (14)	−0.0063 (11)	0.0001 (11)	−0.0013 (10)
C3	0.0377 (14)	0.0351 (14)	0.0665 (18)	−0.0028 (11)	0.0090 (13)	−0.0044 (12)
C4	0.0562 (18)	0.0423 (15)	0.0559 (17)	−0.0066 (13)	0.0231 (14)	−0.0146 (12)
C5	0.0583 (18)	0.0451 (15)	0.0381 (14)	−0.0129 (13)	0.0084 (12)	−0.0099 (11)
C6	0.0397 (14)	0.0346 (13)	0.0339 (12)	−0.0102 (11)	0.0057 (10)	−0.0055 (10)
C7	0.0509 (16)	0.0455 (15)	0.0305 (12)	−0.0125 (12)	−0.0062 (11)	−0.0037 (10)
C8	0.0368 (13)	0.0387 (13)	0.0357 (13)	−0.0056 (11)	−0.0070 (10)	−0.0010 (10)
C9	0.0334 (13)	0.0361 (13)	0.0270 (12)	−0.0037 (10)	0.0042 (10)	−0.0030 (9)
C10	0.0334 (12)	0.0340 (12)	0.0258 (11)	−0.0016 (10)	0.0039 (9)	−0.0046 (9)
C11	0.0326 (13)	0.0384 (13)	0.0268 (11)	−0.0004 (10)	−0.0002 (10)	−0.0052 (9)
C12	0.0366 (14)	0.0372 (14)	0.0349 (12)	−0.0079 (11)	0.0013 (11)	−0.0065 (10)

C13	0.0441 (15)	0.0312 (13)	0.0392 (13)	−0.0055 (11)	0.0080 (11)	−0.0003 (10)
C14	0.0404 (14)	0.0362 (14)	0.0282 (12)	−0.0003 (11)	0.0008 (10)	0.0016 (9)
C15	0.0337 (13)	0.0316 (13)	0.0294 (12)	0.0006 (10)	−0.0060 (10)	−0.0016 (9)
O2	0.0403 (10)	0.0321 (9)	0.0348 (9)	−0.0014 (7)	0.0021 (7)	−0.0070 (6)
N3	0.0353 (11)	0.0344 (11)	0.0248 (9)	−0.0027 (8)	0.0012 (8)	−0.0037 (7)
N4	0.0537 (14)	0.0351 (13)	0.0365 (12)	−0.0049 (10)	0.0111 (10)	−0.0069 (9)
C21	0.0358 (13)	0.0339 (13)	0.0287 (11)	0.0033 (10)	0.0054 (10)	−0.0026 (9)
C22	0.0330 (13)	0.0352 (13)	0.0328 (12)	0.0053 (10)	0.0015 (10)	−0.0026 (9)
C23	0.0386 (14)	0.0380 (14)	0.0479 (15)	0.0009 (11)	0.0074 (12)	−0.0004 (11)
C24	0.0449 (16)	0.0420 (15)	0.0471 (15)	0.0056 (12)	0.0125 (12)	0.0073 (11)
C25	0.0547 (17)	0.0483 (16)	0.0281 (12)	0.0176 (13)	0.0101 (12)	0.0049 (10)
C26	0.0399 (14)	0.0416 (14)	0.0297 (12)	0.0117 (11)	−0.0008 (10)	−0.0040 (10)
C27	0.0565 (17)	0.0532 (16)	0.0294 (12)	0.0151 (13)	−0.0074 (12)	−0.0078 (11)
C28	0.0433 (15)	0.0501 (16)	0.0428 (14)	0.0063 (12)	−0.0110 (12)	−0.0168 (11)
C29	0.0372 (14)	0.0413 (14)	0.0352 (12)	0.0035 (11)	0.0006 (10)	−0.0118 (10)
C30	0.0341 (13)	0.0332 (13)	0.0289 (11)	−0.0028 (10)	0.0034 (10)	−0.0065 (9)
C31	0.0368 (13)	0.0366 (13)	0.0306 (12)	−0.0033 (10)	−0.0015 (10)	−0.0034 (10)
C32	0.0417 (15)	0.0384 (14)	0.0407 (14)	0.0055 (11)	0.0056 (11)	−0.0014 (10)
C33	0.0439 (15)	0.0369 (14)	0.0476 (15)	0.0017 (12)	0.0048 (12)	−0.0148 (11)
C34	0.0385 (14)	0.0479 (15)	0.0411 (14)	0.0023 (12)	−0.0023 (11)	−0.0173 (11)
C35	0.0309 (12)	0.0353 (13)	0.0277 (11)	−0.0010 (10)	−0.0018 (9)	−0.0052 (9)

Geometric parameters (Å, º)

O1—C15	1.233 (3)	O2—C35	1.229 (3)
N1—C15	1.392 (3)	N3—C35	1.390 (3)
N1—C10	1.438 (3)	N3—C30	1.436 (3)
N1—C1	1.439 (3)	N3—C21	1.444 (3)
N2—C15	1.337 (3)	N4—C35	1.339 (3)
N2—H2A	0.89 (3)	N4—H4B	0.86 (2)
N2—H2B	0.92 (2)	N4—H4A	0.90 (3)
C1—C2	1.394 (3)	C21—C22	1.393 (3)
C1—C6	1.397 (3)	C21—C26	1.399 (3)
C2—C3	1.378 (3)	C22—C23	1.383 (3)
C2—H2	0.9500	C22—H22	0.9500
C3—C4	1.384 (4)	C23—C24	1.377 (3)
C3—H3	0.9500	C23—H23	0.9500
C4—C5	1.380 (4)	C24—C25	1.369 (4)
C4—H4	0.9500	C24—H24	0.9500
C5—C6	1.397 (3)	C25—C26	1.410 (3)
C5—H5	0.9500	C25—H25	0.9500
C6—C7	1.513 (3)	C26—C27	1.512 (3)
C7—C8	1.527 (3)	C27—C28	1.531 (3)
C7—H7A	0.9900	C27—H27A	0.9900
C7—H7B	0.9900	C27—H27B	0.9900
C8—C9	1.500 (3)	C28—C29	1.500 (3)
C8—H8A	0.9900	C28—H28A	0.9900
C8—H8B	0.9900	C28—H28B	0.9900

C9—C14	1.390 (3)	C29—C34	1.394 (3)
C9—C10	1.400 (3)	C29—C30	1.398 (3)
C10—C11	1.380 (3)	C30—C31	1.385 (3)
C11—C12	1.384 (3)	C31—C32	1.382 (3)
C11—H11	0.9500	C31—H31	0.9500
C12—C13	1.376 (3)	C32—C33	1.391 (3)
C12—H12	0.9500	C32—H32	0.9500
C13—C14	1.393 (3)	C33—C34	1.382 (4)
C13—H13	0.9500	C33—H33	0.9500
C14—H14	0.9500	C34—H34	0.9500
C15—N1—C10	121.98 (18)	C35—N3—C30	122.25 (18)
C15—N1—C1	118.82 (18)	C35—N3—C21	119.51 (18)
C10—N1—C1	117.67 (17)	C30—N3—C21	117.98 (17)
C15—N2—H2A	117.4 (18)	C35—N4—H4B	127.7 (15)
C15—N2—H2B	127.7 (14)	C35—N4—H4A	114.1 (15)
H2A—N2—H2B	113 (2)	H4B—N4—H4A	118 (2)
C2—C1—C6	121.0 (2)	C22—C21—C26	121.2 (2)
C2—C1—N1	117.7 (2)	C22—C21—N3	117.6 (2)
C6—C1—N1	121.3 (2)	C26—C21—N3	121.2 (2)
C3—C2—C1	120.7 (3)	C23—C22—C21	120.8 (2)
C3—C2—H2	119.6	C23—C22—H22	119.6
C1—C2—H2	119.6	C21—C22—H22	119.6
C2—C3—C4	119.3 (3)	C24—C23—C22	119.0 (3)
C2—C3—H3	120.4	C24—C23—H23	120.5
C4—C3—H3	120.4	C22—C23—H23	120.5
C5—C4—C3	119.9 (2)	C25—C24—C23	120.3 (2)
C5—C4—H4	120.1	C25—C24—H24	119.9
C3—C4—H4	120.1	C23—C24—H24	119.9
C4—C5—C6	122.4 (3)	C24—C25—C26	122.7 (2)
C4—C5—H5	118.8	C24—C25—H25	118.6
C6—C5—H5	118.8	C26—C25—H25	118.6
C5—C6—C1	116.7 (2)	C21—C26—C25	116.0 (2)
C5—C6—C7	116.9 (2)	C21—C26—C27	126.4 (2)
C1—C6—C7	126.3 (2)	C25—C26—C27	117.6 (2)
C6—C7—C8	118.9 (2)	C26—C27—C28	119.1 (2)
C6—C7—H7A	107.6	C26—C27—H27A	107.6
C8—C7—H7A	107.6	C28—C27—H27A	107.6
C6—C7—H7B	107.6	C26—C27—H27B	107.6
C8—C7—H7B	107.6	C28—C27—H27B	107.6
H7A—C7—H7B	107.0	H27A—C27—H27B	107.0
C9—C8—C7	111.3 (2)	C29—C28—C27	111.6 (2)
C9—C8—H8A	109.4	C29—C28—H28A	109.3
C7—C8—H8A	109.4	C27—C28—H28A	109.3
C9—C8—H8B	109.4	C29—C28—H28B	109.3
C7—C8—H8B	109.4	C27—C28—H28B	109.3
H8A—C8—H8B	108.0	H28A—C28—H28B	108.0
C14—C9—C10	118.5 (2)	C34—C29—C30	118.0 (2)

C14—C9—C8	123.4 (2)	C34—C29—C28	123.3 (2)
C10—C9—C8	118.1 (2)	C30—C29—C28	118.7 (2)
C11—C10—C9	121.2 (2)	C31—C30—C29	121.4 (2)
C11—C10—N1	121.3 (2)	C31—C30—N3	121.1 (2)
C9—C10—N1	117.6 (2)	C29—C30—N3	117.5 (2)
C10—C11—C12	119.6 (2)	C32—C31—C30	119.6 (2)
C10—C11—H11	120.2	C32—C31—H31	120.2
C12—C11—H11	120.2	C30—C31—H31	120.2
C13—C12—C11	120.1 (2)	C31—C32—C33	119.9 (2)
C13—C12—H12	119.9	C31—C32—H32	120.1
C11—C12—H12	119.9	C33—C32—H32	120.1
C12—C13—C14	120.5 (2)	C34—C33—C32	120.1 (2)
C12—C13—H13	119.8	C34—C33—H33	119.9
C14—C13—H13	119.8	C32—C33—H33	119.9
C9—C14—C13	120.1 (2)	C33—C34—C29	121.0 (2)
C9—C14—H14	119.9	C33—C34—H34	119.5
C13—C14—H14	119.9	C29—C34—H34	119.5
O1—C15—N2	122.9 (2)	O2—C35—N4	122.7 (2)
O1—C15—N1	120.5 (2)	O2—C35—N3	121.01 (19)
N2—C15—N1	116.6 (2)	N4—C35—N3	116.2 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*B*···O2^{i}	0.92 (2)	2.02 (3)	2.800 (3)	142.7 (19)
N4—H4*B*···O1	0.86 (2)	2.11 (3)	2.801 (3)	137.4 (19)
N2—H2*A*···*Cg*4	0.89 (3)	3.01 (3)	3.862 (3)	162 (2)
N4—H4*A*···*Cg*2^{ii}	0.90 (3)	2.89 (3)	3.765 (3)	166 (2)

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