organic compounds

10,11-Dihydrocarbamazepine–dimethyl sulfoxide (1/1)

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The title compound [systematic name: 10,11-dihydro-$5H$-dibenz[b,f]azepine-5-carboxamide–dimethyl sulfoxide (1/1)], $C_{15}H_{14}N_2O \cdot C_2H_6OS$, crystallizes with one disordered dihydrocarbamazepine (with the approximate ratio of occupancies being 81:19) and one solvent molecule in the asymmetric unit. In the crystal structure, dihydrocarbamazepine molecules form an $R_2^2(8)$ N—H⋯O hydrogen-bonded dimer arrangement with the dimethyl sulfoxide molecules forming an N—H⋯O hydrogen bond to the *anti*-oriented NH group of the carboxamide group of dihydrocarbamazepine.

Related literature

For details on experimental methods used to obtain this form, see: Florence *et al.* (2003); Florence, Johnston, Fernandes *et al.* (2006). For related crystal structures of 10,11-dihydrocarbamazepine, see: Bandoli *et al.* (1992); Cyr *et al.* (1987); Harrison *et al.* (2006); Leech *et al.* (2007); Florence, Leech *et al.* (2006). For solvates of the related dibenzazepine compound carbamazepine, see: Fleischman *et al.* (2003); Florence, Johnston, Price *et al.* (2006). For details of graph-set notation, see: Etter (1990).
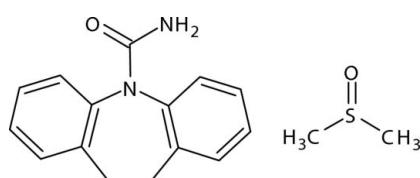
Experimental

Crystal data

$C_{15}H_{14}N_2O \cdot C_2H_6OS$	V = 1624.39 (8) Å³
M_r = 316.41	Z = 4
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
a = 10.2696 (3) Å	μ = 0.21 mm⁻¹
b = 6.8543 (2) Å	T = 123 (2) K
c = 23.3599 (6) Å	$0.24 \times 0.10 \times 0.07$ mm
β = 98.932 (2)°	

Data collection

Oxford Diffraction Gemini diffractometer	on Clark & Reid (1995)]
	T_{min} = 0.952, T_{max} = 0.986
Absorption correction: analytical [*CrysAlis RED* (Oxford Diffraction, 2000), based	16299 measured reflections
	3326 independent reflections
	2627 reflections with $I > 2\sigma(I)$
	R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.114	
S = 1.06	
3326 reflections	$\Delta\rho_{max}$ = 0.71 e Å⁻³
223 parameters	$\Delta\rho_{min}$ = −0.39 e Å⁻³
2 restraints	

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N2—H2A⋯O1[i]	0.85 (2)	2.05 (2)	2.8977 (19)	171 (2)
N2—H2B⋯O2	0.82 (2)	2.13 (2)	2.885 (2)	154 (2)
C16—H16C⋯O1[ii]	0.98	2.45	3.294 (2)	143

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2000); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2000); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *PLATON* (Spek, 2003).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2481).

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supplementary materials

10,11-Dihydrocarbamazepine-dimethyl sulfoxide (1/1)

A. Johnston, A. J. Florence, K. Shankland, C. K. Leech, N. Shankland and P. Fernandes

Comment

10,11-Dihydrocarbamazepine (DHC) is a recognized impurity in carbamazepine (CBZ), a dibenzazepine drug used to control seizures (Cyr *et al.*, 1987). DHC is known to crystallize in three polymorphic forms: monoclinic form I (Bandoli *et al.*, 1992), orthorhombic form II (Harrison *et al.*, 2006) and triclinic form III (Leech *et al.*, 2007*a*). The title compound, was produced during an automated parallel crystallization study (Florence, Johnston, Fernandes *et al.*, 2006) on DHC as part of a wider investigation into the predicted and experimental structures of CBZ (Florence, Johnston, Price *et al.*, 2006; Florence, Leech *et al.*, 2006) and related molecules (Leech *et al.*, 2007). The sample was identified as a new form using multi-sample foil transmission X-ray powder diffraction analysis (Florence *et al.*, 2003). Subsequent manual recrystallization, from a saturated dimethyl sulfoxide (DMSO) solution, by cooling to 273 K yielded single crystals suitable for X-ray diffraction. The molecular structure is shown in Fig. 1.

In the crystal structure, DHC molecules are partially disordered over two sites with the major component of this disorder being present at approximately 81%.

DHC molecules form a centrosymmetric hydrogen-bonded $R_2^2(8)$ dimer motif (Etter, 1990) *via* N2—H2A···O1^i (symmetry code as in the Hydrogen Bond Geometry table) contacts in contrast to the C(4) catameric configuration observed in each of the three polymorphic forms. In the title structure, the DHC molecules form a second N—H···O hydrogen bond to atom O2 of the solvent molecule (Fig. 2) to form a packing motif also observed in the CBZ DMSO solvate (Fleischman *et al.*, 2003).

Experimental

DHC was used as received from SigmaAldrich and a single-crystal sample was obtained by cooling a saturated DMSO solution, saturated at 298 K, to 273 K.

Refinement

For the major component of the disorder and for the undisordered part of the structure all non-hydrogen atoms were located from the direct method solution, all hydrogen atoms were visible in a Fourier difference map. The –NH$_2$ hydrogen atoms were located in a Fourier difference map and the atomic coordinates and U_{iso} values were refined freely. The rest of the hydrogen atoms were constrained to geometrically sensible positions in a riding-model approximation [*SHELXL97*; Sheldrick (1997)].

For the minor component of disorder, atoms C7A and C8A were visible in the Fourier difference map. The aromatic rings were constrained to a regular hexagon with C—C bond length of 1.39Å and all hydrogen atoms were constrained to geometrically sensible positions in a riding-model approximation [*SHELXL97*; Sheldrick (1997)]. All atoms in the minor

supplementary materials

component of the disorder were refined isotropically with U_{iso} constrained to be identical for all C atoms. The U_{iso} value for the H atoms were constrained to be 1.2 times the value for the C atoms.

Figures
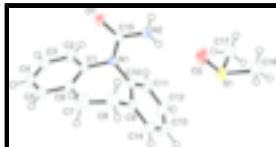
Fig. 1. The asymmetric unit showing 50% probablility displacement ellipsoids. Minor occupancy disordered atomic sites have been omitted for clarity.
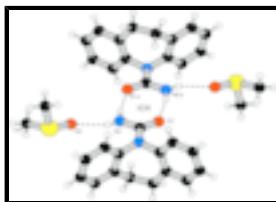
Fig. 2. The packing motif containing the centrosymmetric $R_2^2(8)$ dimer between DHC molecules, and the N—H···O contact between DHC and DMSO molecules. Hydrogen bonds are shown as dashed lines and minor disordered components have been omitted for clarity. [Symmetry code:(*a*) $2 − x, -y, 1 − z$].

10,11-dihydro-*5H*-dibenz[*b,f*]azepine-5-carboxamide–dimethyl sulfoxide (1/1)

Crystal data
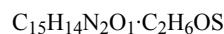
$C_{15}H_{14}N_2O_1·C_2H_6OS$	$F_{000} = 672$
$M_r = 316.41$	$D_x = 1.294$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 7414 reflections
$a = 10.2696$ (3) Å	$\theta = 2.6–28.6°$
$b = 6.8543$ (2) Å	$\mu = 0.21$ mm^{-1}
$c = 23.3599$ (6) Å	$T = 123$ (2) K
$\beta = 98.932$ (2)°	Block, colourless
$V = 1624.39$ (8) Å^3	$0.24 \times 0.10 \times 0.07$ mm
$Z = 4$	

Data collection

Oxford Diffraction Gemini diffractometer	3326 independent reflections
Radiation source: Enhance (Mu) X-ray Source	2627 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.029$
$T = 123(2)$ K	$\theta_{max} = 26.4°$
φ and ω scans	$\theta_{min} = 2.9°$
Absorption correction: analytical [CrysAlis RED (Oxford Diffraction, 2000), based on Clark & Reid (1995)]	$h = −10 \rightarrow 12$
$T_{min} = 0.952, T_{max} = 0.986$	$k = −8 \rightarrow 8$
16299 measured reflections	$l = −29 \rightarrow 29$

Refinement

Refinement on F^2	2 restraints
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.042$	$w = 1/[\sigma^2(F_o^2) + (0.0295P)^2 + 2.9523P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.114$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.06$	$\Delta\rho_{max} = 0.71$ e Å^{-3}
3326 reflections	$\Delta\rho_{min} = -0.39$ e Å^{-3}
223 parameters	Extinction correction: none

Special details

Experimental. Absorption correction: *CrysAlis RED* (Oxford Diffraction, 2000), analytical numeric absorption correction using a multifaceted crystal model based on expressions derived by Clark & Reid (1995). 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 > 2sigma(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.

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. The structure was refined using a disordered model in which the relative occupancy of the major component equals 81.1 (6)%. The disordered model is justified by the fact the refined structure using an ordered model showed 1) large residual peaks of electron density close to the C7—C8 bond which were assigned as atoms C7a and C8a, and 2) anomalous adps especially for atoms C3 C4 C5 and C12 C13 C14. Inclusion of disorder yielded adp values within normal ranges for these atoms. Overall, the disordered model resulted in significant improvements in the Rfactors (wR2 = 0.1426 and R1 = 0.0551 with no disorder).

The major and minor components of the disorder are related conformers of DHC. The two conformers are related by an approximate inversion of the C6—C7—C8—C9 torsion angle, which leads to an associated shift in the position of the aromatic rings. This relationship can also be described as an approximate 180° rotation about the N1—C15 bond followed by an inversion of the whole molecule (note SG=P21/c contains an inversion center).

For the major component of the disorder and for the undisordered part of the structure all non-hydrogen atoms were located from the direct method solution, all hydrogen atoms were visible in the Fourier difference map, the NH2 hydrogen atoms located from the Fourier difference map and the atomic coordinated and U_{iso} refined freely, the rest of the hydrogen atoms were constrained to geometrically sensible positions with a riding model (*SHELX97*).

For the minor component of the disorder atoms C7A and C8A were visible in the Fourier difference map. The aromatic rings were constrained to a regular hexagon with C—C bond lenght of 1.39Å and all hydrogen atoms were constrained to geometrically sensible positions with a riding model (*SHELX97*). All atoms in the minor component of the disorder were refined isotropically with U_{iso} constrained to be identical for all the carbon atoms. The U_{iso} for the hydrogen atoms was constrained to be 1.2 times the value for the hydrogen atoms.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	y	z	$U_{iso}*/U_{eq}$	Occ. (<1)
O1	1.05951 (11)	0.08903 (17)	0.43941 (5)	0.0227 (3)	
N1	0.91771 (13)	0.2552 (2)	0.37293 (6)	0.0189 (3)	
C15	0.94794 (15)	0.1576 (2)	0.42506 (7)	0.0189 (3)	
N2	0.85194 (15)	0.1394 (2)	0.45751 (7)	0.0236 (3)	
H2A	0.871 (2)	0.077 (3)	0.4893 (10)	0.038 (6)*	
H2B	0.778 (2)	0.186 (3)	0.4494 (9)	0.034 (6)*	
C1	1.02405 (15)	0.3029 (3)	0.34188 (7)	0.0207 (4)	
C2A	1.0637 (9)	0.1930 (9)	0.2976 (3)	0.0203 (13)*	0.189 (6)
H2C	1.0215	0.0725	0.2866	0.024*	0.189 (6)
C3A	1.1650 (10)	0.2595 (14)	0.2696 (4)	0.0203 (13)*	0.189 (6)
H3A	1.1921	0.1844	0.2393	0.024*	0.189 (6)
C4A	1.2268 (7)	0.4358 (15)	0.2858 (4)	0.0203 (13)*	0.189 (6)
H4A	1.2961	0.4812	0.2666	0.024*	0.189 (6)
C5A	1.1872 (9)	0.5457 (12)	0.3300 (5)	0.0203 (13)*	0.189 (6)
H5A	1.2294	0.6662	0.3411	0.024*	0.189 (6)
C6A	1.0858 (8)	0.4793 (8)	0.3581 (3)	0.0203 (13)*	0.189 (6)
C2	1.0568 (3)	0.1613 (4)	0.30398 (11)	0.0264 (6)	0.811 (6)
H2	1.0115	0.0400	0.3006	0.032*	0.811 (6)
C3	1.1561 (3)	0.1974 (5)	0.27098 (11)	0.0300 (7)	0.811 (6)
H3	1.1799	0.1006	0.2454	0.036*	0.811 (6)
C4	1.2193 (2)	0.3749 (5)	0.27597 (11)	0.0293 (7)	0.811 (6)
H4	1.2868	0.4012	0.2534	0.035*	0.811 (6)
C5	1.1859 (2)	0.5136 (4)	0.31305 (13)	0.0313 (7)	0.811 (6)
H5	1.2310	0.6351	0.3157	0.038*	0.811 (6)
C6	1.0874 (3)	0.4829 (4)	0.34740 (11)	0.0272 (6)	0.811 (6)
C7	1.0555 (3)	0.6467 (4)	0.38578 (16)	0.0365 (7)	0.811 (6)
H7A	1.0392	0.7646	0.3613	0.044*	0.811 (6)
H7B	1.1357	0.6724	0.4142	0.044*	0.811 (6)
C8	0.9413 (3)	0.6269 (4)	0.41943 (13)	0.0293 (7)	0.811 (6)
H8A	0.9644	0.5291	0.4505	0.035*	0.811 (6)
H8B	0.9274	0.7533	0.4380	0.035*	0.811 (6)
C9	0.8147 (2)	0.5664 (3)	0.38213 (10)	0.0233 (5)	0.811 (6)
C10	0.80304 (16)	0.3762 (3)	0.36009 (7)	0.0217 (4)	
C11A	0.6944 (4)	0.2697 (6)	0.3343 (3)	0.0203 (13)*	0.189 (6)
H11A	0.7023	0.1334	0.3283	0.024*	0.189 (6)
C12A	0.5743 (3)	0.3626 (12)	0.3173 (4)	0.0203 (13)*	0.189 (6)
H12A	0.5001	0.2898	0.2996	0.024*	0.189 (6)
C13A	0.5629 (4)	0.5620 (12)	0.3261 (4)	0.0203 (13)*	0.189 (6)
H13A	0.4808	0.6255	0.3144	0.024*	0.189 (6)
C14A	0.6715 (7)	0.6686 (7)	0.3519 (4)	0.0203 (13)*	0.189 (6)
H14A	0.6636	0.8049	0.3579	0.024*	0.189 (6)
C9A	0.7916 (5)	0.5757 (3)	0.3689 (3)	0.0203 (13)*	0.189 (6)
C7A	1.0462 (10)	0.5949 (16)	0.4089 (4)	0.0203 (13)*	0.189 (6)
H7C	1.0466	0.5061	0.4423	0.024*	0.189 (6)
H7D	1.1127	0.6979	0.4204	0.024*	0.189 (6)

C8A	0.9104 (8)	0.6895 (16)	0.3947 (5)	0.0203 (13)*	0.189 (6)
H8C	0.8902	0.7477	0.4311	0.024*	0.189 (6)
H8D	0.9191	0.7991	0.3680	0.024*	0.189 (6)
C11	0.6906 (2)	0.3133 (5)	0.32519 (11)	0.0319 (6)	0.811 (6)
H11	0.6850	0.1841	0.3103	0.038*	0.811 (6)
C12	0.5857 (2)	0.4407 (6)	0.31199 (11)	0.0406 (8)	0.811 (6)
H12	0.5074	0.3989	0.2880	0.049*	0.811 (6)
C13	0.5945 (3)	0.6268 (5)	0.33340 (12)	0.0397 (8)	0.811 (6)
H13	0.5221	0.7134	0.3241	0.048*	0.811 (6)
C14	0.7070 (3)	0.6901 (4)	0.36816 (12)	0.0338 (7)	0.811 (6)
H14	0.7112	0.8197	0.3828	0.041*	0.811 (6)
S1	0.43861 (4)	0.26487 (8)	0.43255 (2)	0.03643 (17)	
O2	0.57765 (12)	0.2223 (2)	0.46042 (6)	0.0429 (4)	
C17	0.3694 (2)	0.0384 (4)	0.40614 (10)	0.0453 (6)	
H17A	0.4117	−0.0051	0.3736	0.068*	
H17B	0.2746	0.0546	0.3931	0.068*	
H17C	0.3836	−0.0591	0.4372	0.068*	
C16	0.34591 (19)	0.2887 (3)	0.49031 (10)	0.0412 (5)	
H16A	0.3713	0.4093	0.5117	0.062*	
H16B	0.3635	0.1768	0.5165	0.062*	
H16C	0.2517	0.2932	0.4746	0.062*	

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0168 (6)	0.0253 (6)	0.0260 (6)	0.0016 (5)	0.0037 (5)	0.0045 (5)
N1	0.0165 (7)	0.0214 (7)	0.0193 (7)	0.0003 (5)	0.0043 (5)	0.0004 (6)
C15	0.0188 (8)	0.0153 (8)	0.0223 (8)	−0.0026 (6)	0.0023 (6)	−0.0015 (7)
N2	0.0182 (8)	0.0283 (8)	0.0250 (8)	0.0038 (6)	0.0055 (6)	0.0081 (7)
C1	0.0156 (8)	0.0288 (9)	0.0171 (8)	0.0000 (7)	0.0005 (6)	0.0027 (7)
C2	0.0210 (11)	0.0389 (14)	0.0193 (11)	−0.0028 (10)	0.0036 (8)	−0.0018 (10)
C3	0.0252 (12)	0.0462 (18)	0.0188 (11)	0.0003 (12)	0.0038 (8)	−0.0010 (12)
C4	0.0195 (11)	0.0475 (19)	0.0209 (12)	−0.0017 (11)	0.0031 (9)	0.0063 (11)
C5	0.0226 (11)	0.0370 (15)	0.0339 (16)	−0.0070 (11)	0.0031 (12)	0.0069 (12)
C6	0.0211 (11)	0.0333 (13)	0.0270 (13)	−0.0001 (9)	0.0031 (10)	0.0036 (10)
C7	0.0360 (14)	0.0286 (14)	0.0456 (18)	−0.0076 (11)	0.0091 (12)	−0.0032 (12)
C8	0.0363 (14)	0.0229 (13)	0.0293 (14)	−0.0010 (10)	0.0072 (11)	−0.0035 (11)
C9	0.0259 (12)	0.0280 (12)	0.0183 (11)	0.0073 (9)	0.0103 (9)	0.0088 (9)
C10	0.0207 (8)	0.0290 (9)	0.0164 (8)	0.0042 (7)	0.0064 (6)	0.0042 (7)
C11	0.0231 (12)	0.0472 (16)	0.0242 (12)	0.0054 (10)	−0.0003 (9)	−0.0023 (11)
C12	0.0234 (12)	0.068 (2)	0.0288 (14)	0.0095 (13)	−0.0005 (10)	0.0042 (15)
C13	0.0337 (15)	0.0553 (19)	0.0314 (14)	0.0211 (14)	0.0092 (12)	0.0109 (13)
C14	0.0388 (15)	0.0398 (15)	0.0258 (14)	0.0160 (11)	0.0142 (11)	0.0118 (11)
S1	0.0212 (3)	0.0496 (3)	0.0390 (3)	0.0011 (2)	0.00652 (19)	0.0107 (2)
O2	0.0173 (7)	0.0631 (10)	0.0486 (9)	−0.0003 (6)	0.0059 (6)	0.0105 (8)
C17	0.0315 (11)	0.0599 (15)	0.0430 (12)	0.0056 (10)	0.0013 (9)	−0.0136 (11)
C16	0.0253 (10)	0.0509 (14)	0.0488 (12)	−0.0079 (9)	0.0098 (9)	−0.0113 (10)

supplementary materials

Geometric parameters (Å, °)

O1—C15	1.2357 (19)	C9—C14	1.392 (3)
N1—C15	1.382 (2)	C9—C10	1.399 (3)
N1—C10	1.434 (2)	C10—C11	1.376 (3)
N1—C1	1.439 (2)	C10—C11A	1.3900
C15—N2	1.339 (2)	C10—C9A	1.3900
N2—H2A	0.85 (2)	C11A—C12A	1.3900
N2—H2B	0.82 (2)	C11A—H11A	0.9500
C1—C2	1.390 (3)	C12A—C13A	1.3900
C1—C2A	1.3900	C12A—H12A	0.9500
C1—C6A	1.3900	C13A—C14A	1.3900
C1—C6	1.391 (3)	C13A—H13A	0.9500
C2A—C3A	1.3900	C14A—C9A	1.3900
C2A—H2C	0.9500	C14A—H14A	0.9500
C3A—C4A	1.3900	C9A—C8A	1.494 (7)
C3A—H3A	0.9500	C7A—C8A	1.527 (14)
C4A—C5A	1.3900	C7A—H7C	0.9900
C4A—H4A	0.9500	C7A—H7D	0.9900
C5A—C6A	1.3900	C8A—H8C	0.9900
C5A—H5A	0.9500	C8A—H8D	0.9900
C6A—C7A	1.533 (8)	C11—C12	1.384 (4)
C2—C3	1.393 (3)	C11—H11	0.9500
C2—H2	0.9500	C12—C13	1.368 (4)
C3—C4	1.376 (4)	C12—H12	0.9500
C3—H3	0.9500	C13—C14	1.375 (4)
C4—C5	1.365 (4)	C13—H13	0.9500
C4—H4	0.9500	C14—H14	0.9500
C5—C6	1.401 (3)	S1—O2	1.5030 (14)
C5—H5	0.9500	S1—C16	1.776 (2)
C6—C7	1.504 (3)	S1—C17	1.777 (2)
C7—C8	1.516 (4)	C17—H17A	0.9800
C7—H7A	0.9900	C17—H17B	0.9800
C7—H7B	0.9900	C17—H17C	0.9800
C8—C9	1.507 (3)	C16—H16A	0.9800
C8—H8A	0.9900	C16—H16B	0.9800
C8—H8B	0.9900	C16—H16C	0.9800
?···?	?		
C15—N1—C10	121.95 (13)	C11—C10—C9A	108.3 (2)
C15—N1—C1	118.02 (13)	C11A—C10—C9A	120.0
C10—N1—C1	115.22 (13)	C11—C10—C9	121.72 (19)
O1—C15—N2	122.95 (16)	C11A—C10—C9	131.7 (2)
O1—C15—N1	119.68 (14)	C11—C10—N1	121.96 (19)
N2—C15—N1	117.36 (14)	C11A—C10—N1	111.4 (2)
C15—N2—H2A	116.5 (14)	C9A—C10—N1	128.6 (2)
C15—N2—H2B	124.7 (15)	C9—C10—N1	116.20 (17)
H2A—N2—H2B	119 (2)	C10—C11A—C12A	120.0
C2—C1—C6A	129.5 (3)	C10—C11A—H11A	120.0

C2A—C1—C6A	120.0	C12A—C11A—H11A	120.0
C2—C1—C6	121.50 (18)	C13A—C12A—C11A	120.0
C2A—C1—C6	111.3 (3)	C13A—C12A—H12A	120.0
C2—C1—N1	116.10 (17)	C11A—C12A—H12A	120.0
C2A—C1—N1	125.7 (3)	C12A—C13A—C14A	120.0
C6A—C1—N1	114.2 (3)	C12A—C13A—H13A	120.0
C6—C1—N1	122.35 (17)	C14A—C13A—H13A	120.0
C1—C2A—C3A	120.0	C13A—C14A—C9A	120.0
C1—C2A—H2C	120.0	C13A—C14A—H14A	120.0
C3A—C2A—H2C	120.0	C9A—C14A—H14A	120.0
C2A—C3A—C4A	120.0	C14A—C9A—C10	120.0
C2A—C3A—H3A	120.0	C14A—C9A—C8A	120.6 (5)
C4A—C3A—H3A	120.0	C10—C9A—C8A	119.4 (5)
C3A—C4A—C5A	120.0	C8A—C7A—C6A	113.2 (8)
C3A—C4A—H4A	120.0	C8A—C7A—H7C	108.9
C5A—C4A—H4A	120.0	C6A—C7A—H7C	108.9
C6A—C5A—C4A	120.0	C8A—C7A—H7D	108.9
C6A—C5A—H5A	120.0	C6A—C7A—H7D	108.9
C4A—C5A—H5A	120.0	H7C—C7A—H7D	107.7
C5A—C6A—C1	120.0	C9A—C8A—C7A	121.8 (8)
C5A—C6A—C7A	120.2 (5)	C9A—C8A—H8C	106.9
C1—C6A—C7A	119.7 (5)	C7A—C8A—H8C	106.9
C1—C2—C3	119.9 (2)	C9A—C8A—H8D	106.9
C1—C2—H2	120.0	C7A—C8A—H8D	106.9
C3—C2—H2	120.0	H8C—C8A—H8D	106.7
C4—C3—C2	119.1 (2)	C10—C11—C12	119.2 (2)
C4—C3—H3	120.4	C10—C11—H11	120.4
C2—C3—H3	120.4	C12—C11—H11	120.4
C5—C4—C3	120.5 (2)	C13—C12—C11	120.1 (2)
C5—C4—H4	119.7	C13—C12—H12	119.9
C3—C4—H4	119.7	C11—C12—H12	119.9
C4—C5—C6	122.2 (2)	C12—C13—C14	120.8 (2)
C4—C5—H5	118.9	C12—C13—H13	119.6
C6—C5—H5	118.9	C14—C13—H13	119.6
C1—C6—C5	116.7 (2)	C13—C14—C9	120.7 (2)
C1—C6—C7	125.1 (2)	C13—C14—H14	119.7
C5—C6—C7	118.2 (2)	C9—C14—H14	119.7
C6—C7—C8	120.0 (2)	O2—S1—C16	105.90 (10)
C6—C7—H7A	107.3	O2—S1—C17	106.44 (10)
C8—C7—H7A	107.3	C16—S1—C17	96.39 (10)
C6—C7—H7B	107.3	S1—C17—H17A	109.5
C8—C7—H7B	107.3	S1—C17—H17B	109.5
H7A—C7—H7B	106.9	H17A—C17—H17B	109.5
C9—C8—C7	113.1 (2)	S1—C17—H17C	109.5
C9—C8—H8A	109.0	H17A—C17—H17C	109.5
C7—C8—H8A	109.0	H17B—C17—H17C	109.5
C9—C8—H8B	109.0	S1—C16—H16A	109.5
C7—C8—H8B	109.0	S1—C16—H16B	109.5
H8A—C8—H8B	107.8	H16A—C16—H16B	109.5

C14—C9—C10	117.5 (2)	S1—C16—H16C	109.5
C14—C9—C8	123.4 (2)	H16A—C16—H16C	109.5
C10—C9—C8	119.06 (19)	H16B—C16—H16C	109.5
C10—N1—C1—C2	−116.0 (2)	C3—C4—C5—C6	0.1 (4)
C10—N1—C1—C6	61.5 (2)	C4—C5—C6—C1	−0.2 (4)
C15—N1—C1—C2	88.1 (2)	C4—C5—C6—C7	−178.5 (3)
C15—N1—C1—C6	−94.5 (2)	C1—C6—C7—C8	−3.8 (4)
C1—N1—C10—C9	−72.6 (2)	C5—C6—C7—C8	174.3 (3)
C1—N1—C10—C11	103.6 (2)	C6—C7—C8—C9	−51.3 (4)
C15—N1—C10—C9	82.3 (2)	C7—C8—C9—C10	70.4 (3)
C15—N1—C10—C11	−101.5 (2)	C7—C8—C9—C14	−109.7 (3)
C1—N1—C15—O1	−10.6 (2)	C8—C9—C10—N1	−3.0 (3)
C1—N1—C15—N2	170.45 (15)	C8—C9—C10—C11	−179.1 (2)
C10—N1—C15—O1	−164.93 (15)	C14—C9—C10—N1	177.14 (19)
C10—N1—C15—N2	16.2 (2)	C14—C9—C10—C11	1.0 (3)
N1—C1—C2—C3	178.2 (2)	C8—C9—C14—C13	179.4 (3)
C6—C1—C2—C3	0.8 (4)	C10—C9—C14—C13	−0.8 (4)
N1—C1—C6—C5	−177.6 (2)	N1—C10—C11—C12	−176.6 (2)
N1—C1—C6—C7	0.6 (4)	C9—C10—C11—C12	−0.7 (3)
C2—C1—C6—C5	−0.2 (4)	C10—C11—C12—C13	0.2 (4)
C2—C1—C6—C7	177.9 (3)	C11—C12—C13—C14	0.0 (4)
C1—C2—C3—C4	−0.9 (4)	C12—C13—C14—C9	0.3 (4)
C2—C3—C4—C5	0.4 (4)		

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1[i]	0.85 (2)	2.05 (2)	2.8977 (19)	171 (2)
N2—H2B···O2	0.82 (2)	2.13 (2)	2.885 (2)	154 (2)
C16—H16C···O1[ii]	0.98	2.45	3.294 (2)	143

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

Fig. 1

Fig. 2