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10,11-Dihydrocarbamazepine–formamide solvate (1/1)

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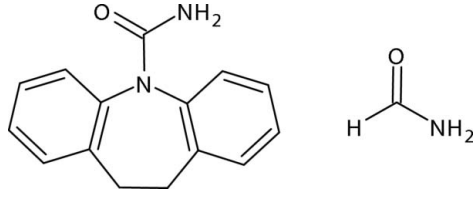
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.003 Å; R factor = 0.074; wR factor = 0.193; data-to-parameter ratio = 63.7.

In the title compound {systematic name: 10,11-dihydro-5*H*-dibenz[*b*,*f*]azepine-5-carboxamide–formamide solvate (1/1)}, $C_{15}H_{14}N_2O{\cdot}CH_3NO$, the dihydrocarbamazepine and formamide molecules are hydrogen bonded to form an $R_2^2(8)$ dimer, which is further connected to form a ladder motif.

Related literature

For details of experimental methods used to obtain this compound, see: Florence *et al.* (2003); Florence, Johnston, Fernandes *et al.* (2006). For related crystal structures, see: Bandoli *et al.* (1992); Cyr *et al.* (1987); Harrison *et al.* (2006); Leech *et al.* (2007); Fleischman *et al.* (2003); Florence, Johnston, Price *et al.* (2006); Florence, Leech *et al.* (2006); Johnston *et al.* (2006, 2007). For related literature, see: Etter (1990).



Experimental

Crystal data

$C_{15}H_{14}N_2O{\cdot}CH_3NO$
M_r = 283.33
Triclinic, $P\overline{1}$
a = 8.4690 (4) Å
b = 9.0215 (4) Å
c = 10.3137 (4) Å
α = 74.363 (3)°
β = 83.630 (3)°
γ = 70.847 (3)°
V = 716.61 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 120 (2) K
0.08 × 0.06 × 0.02 mm

Data collection

Bruker–Nonius 95mm CCD camera on κ-goniostat
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2007)
T_{min} = 0.624, T_{max} = 1
(expected range = 0.623–0.998)
16142 measured reflections
13441 independent reflections
7956 reflections with $I > 2\sigma(I)$
R_{int} = 0.106

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.194
S = 1.04
13441 reflections
211 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2N···O2	0.87 (2)	2.12 (2)	2.975 (2)	170.0 (19)
N2—H1N···O2^{i}	0.93 (2)	2.12 (2)	2.948 (2)	147.6 (17)
N3—H4N···O1	0.94 (2)	1.99 (2)	2.924 (2)	172.2 (19)
N3—H3N···O1^{ii}	0.88 (2)	2.10 (2)	2.945 (2)	159.7 (18)

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

Data collection: *COLLECT* (Nonius, 1998) and *DENZO* (Otwinowski & Minor, 1997); cell refinement: *COLLECT* and *DENZO*; data reduction: *DENZO*; 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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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WN2194).

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  doi:10.1107/S1600536807041049

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supplementary materials

Acta Cryst. (2007). E**63**, o3888-o3889 [**doi:10.1107/S1600536807041049**]

10,11-Dihydrocarbamazepine-formamide solvate (1/1)

A. Johnston, A. J. Florence, P. Fernandes, N. Shankland and A. R. Kennedy

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). The title compound was produced during an automated parallel crystallization study (Florence, Johnston, Fernandes *et al.*, 2006) on DHC as part of a wider study 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 formamide solution by slow evaporation at 298 K, yielded single crystals suitable for X-ray diffraction (Fig. 1).

The molecules adopt a hydrogen-bonded arrangement similar to that observed in the DHC acetic acid (1/1) (Johnston *et al.*, 2006) and DHC formic acid (1/1) solvates (Johnston *et al.*, 2007). Specifically, the DHC and formamide molecules are connected *via* N2—H2N···O2 and N3—H4N···O1 hydrogen bonds to form an $R_2^2(8)$ (Etter, 1990) dimer motif (Table 1). Two further hydrogen bonds, N2—H1···O2^{i} and N3—H3N···O1^{ii} form centrosymmetric $R_2^4(8)$ motifs that link the $R_2^2(8)$ DHC/formamide dimers in a ladder arrangement running parallel to the *c* axis (Fig. 2).

Experimental

DHC was used as received from SigmaAldrich and a single-crystal sample of the title compound was obtained by isothermal solvent evaporation of a saturated formamide solution at 298 K.

Refinement

All crystals examined were twinned by a 180° rotation about −1 1 0. The model reported utilized the *SHELX* HKLF 5 convention where the ratio of the two twin components refined to 0.518 (3):0.482 (3). H atoms in the formamide solvent and the NH_2 group of the DHC molecule were found by difference synthesis and refined isotropically; C—H = 1.026 (19) and N—H = 0.87 (2)–0.94 (2) Å. All other H atoms were constrained to idealized geometry with riding models: U_{iso}(H) = 1.2U_{eq}(C); C—H distances = 0.95 and 0.99 Å for CH and CH_2 groups, respectively.

Figures

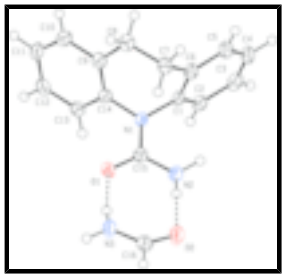

Fig. 1. The asymmetric unit of the title compound, showing 50% probability displacement ellipsoids. Hydrogen bonds are shown as dashed lines.

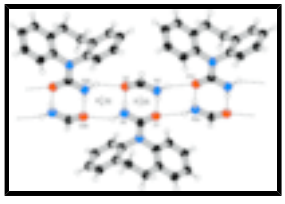

Fig. 2. Plot showing the hydrogen-bonded dimer arrangement in the title compound, with three $R_2^2(8)$ dimers linked in a ladder arrangement *via* two $R_4^2(8)$ motifs. Hydrogen bonds are shown as dashed lines. [Symmetry codes: (*b*) −*x* + 1, −*y*, −*z* + 2, (*c*) −*x* + 1, −*y*, −*z* + 1.]

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

Crystal data

$C_{15}H_{14}N_2O{\cdot}CH_3NO$	$Z = 2$
$M_r = 283.33$	$F_{000} = 300$
Triclinic, $P\overline{1}$	$D_x = 1.313$ Mg m^{-3}
$a = 8.4690$ (4) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 9.0215$ (4) Å	Cell parameters from 3115 reflections
$c = 10.3137$ (4) Å	$\theta = 2.9$–27.5º
$\alpha = 74.363$ (3)º	$\mu = 0.09$ mm^{-1}
$\beta = 83.630$ (3)º	$T = 120$ (2) K
$\gamma = 70.847$ (3)º	Slab, colourless
$V = 716.61$ (5) Å^3	0.08 × 0.06 × 0.02 mm

Data collection

Bruker Nonius 95mm CCD camera on κ-goniostat diffractometer	13441 independent reflections
Radiation source: Bruker Nonius FR591 rotating anode	7956 reflections with $I > 2\sigma(I)$
Monochromator: 10cm confocal mirrors	$R_{int} = 0.106$
Detector resolution: 9.091 pixels mm^{-1}	$\theta_{max} = 26.0$º
$T = 120(2)$ K	$\theta_{min} = 2.9$º
φ and ω scans	$h = -10 \rightarrow 10$
Absorption correction: multi-scan (SADABS; Sheldrick, 2007)	$k = -11 \rightarrow 11$
$T_{min} = 0.624$, $T_{max} = 1$	$l = -12 \rightarrow 12$
16142 measured reflections	

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0295P)^2 + 2.9523P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.074$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.194$	$\Delta\rho_{max} = 0.37$ e Å^{-3}
$S = 1.04$	$\Delta\rho_{min} = -0.33$ e Å^{-3}
13441 reflections	Extinction correction: none
211 parameters	

Special details

Experimental. Data collection at Soton service 2006src1503. Data treated as twinned (180 ° rot) about −1 1 0 hklf 5 file created. *SADABS* does not output Tmin and Tmax - but the ratio is reported as 0.62441.

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 $F^2 > 2$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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.66395 (16)	0.07647 (16)	0.62574 (12)	0.0278 (3)
N1	0.78725 (19)	0.21960 (19)	0.70960 (15)	0.0228 (4)
N2	0.6485 (2)	0.0536 (2)	0.85028 (17)	0.0318 (4)
C1	0.8002 (2)	0.2875 (2)	0.81820 (18)	0.0237 (5)
C2	0.9315 (2)	0.2155 (2)	0.90536 (18)	0.0269 (5)
H2	1.0145	0.1178	0.8963	0.032*
C3	0.9412 (3)	0.2868 (3)	1.00620 (19)	0.0305 (5)
H3	1.0299	0.2375	1.0680	0.037*
C4	0.8201 (3)	0.4309 (3)	1.01629 (19)	0.0339 (5)
H4	0.8263	0.4801	1.0854	0.041*
C5	0.6905 (3)	0.5036 (3)	0.92678 (19)	0.0303 (5)
H5	0.6095	0.6031	0.9339	0.036*
C6	0.6782 (2)	0.4316 (2)	0.82621 (19)	0.0268 (5)
C7	0.5462 (2)	0.5087 (2)	0.72184 (18)	0.0279 (5)
H7A	0.4567	0.5968	0.7510	0.034*
H7B	0.4959	0.4270	0.7134	0.034*
C8	0.6176 (2)	0.5775 (2)	0.58525 (19)	0.0287 (5)
H8A	0.5250	0.6264	0.5210	0.034*

H8B	0.6566	0.6663	0.5941	0.034*
C9	0.7598 (2)	0.4634 (2)	0.52278 (19)	0.0247 (5)
C10	0.8252 (2)	0.5318 (3)	0.39736 (19)	0.0273 (5)
H10	0.7784	0.6443	0.3579	0.033*
C11	0.9537 (3)	0.4415 (3)	0.33078 (19)	0.0293 (5)
H11	0.9953	0.4917	0.2469	0.035*
C12	1.0230 (3)	0.2771 (3)	0.38566 (19)	0.0294 (5)
H12	1.1098	0.2130	0.3386	0.035*
C13	0.9645 (2)	0.2072 (2)	0.50968 (19)	0.0249 (5)
H13	1.0139	0.0950	0.5491	0.030*
C14	0.8346 (2)	0.2986 (2)	0.57749 (18)	0.0223 (4)
C15	0.6970 (2)	0.1151 (2)	0.72422 (18)	0.0244 (5)
O2	0.36473 (18)	−0.07760 (18)	0.86899 (13)	0.0367 (4)
N3	0.3791 (2)	−0.0440 (2)	0.64367 (17)	0.0297 (4)
C16	0.3135 (3)	−0.0850 (3)	0.7648 (2)	0.0286 (5)
H1N	0.659 (3)	0.095 (2)	0.921 (2)	0.037 (6)*
H2N	0.575 (3)	0.004 (2)	0.859 (2)	0.032 (6)*
H3N	0.347 (3)	−0.065 (2)	0.573 (2)	0.036 (6)*
H4N	0.475 (3)	−0.010 (3)	0.631 (2)	0.043 (7)*
H16	0.214 (2)	−0.127 (2)	0.7653 (18)	0.028 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0350 (8)	0.0344 (9)	0.0213 (7)	−0.0180 (7)	−0.0003 (6)	−0.0100 (6)
N1	0.0284 (9)	0.0260 (10)	0.0166 (8)	−0.0136 (8)	0.0009 (7)	−0.0039 (7)
N2	0.0453 (12)	0.0383 (12)	0.0207 (10)	−0.0262 (10)	0.0015 (9)	−0.0065 (9)
C1	0.0292 (11)	0.0288 (12)	0.0173 (10)	−0.0165 (10)	0.0022 (9)	−0.0043 (9)
C2	0.0290 (11)	0.0297 (12)	0.0235 (11)	−0.0142 (10)	−0.0022 (9)	−0.0023 (9)
C3	0.0292 (12)	0.0422 (14)	0.0228 (11)	−0.0193 (11)	−0.0046 (9)	−0.0009 (10)
C4	0.0455 (14)	0.0485 (15)	0.0197 (11)	−0.0292 (12)	0.0012 (10)	−0.0106 (10)
C5	0.0314 (12)	0.0349 (13)	0.0270 (12)	−0.0131 (10)	0.0051 (9)	−0.0106 (10)
C6	0.0289 (12)	0.0341 (13)	0.0217 (11)	−0.0166 (10)	0.0014 (9)	−0.0064 (9)
C7	0.0272 (11)	0.0329 (12)	0.0247 (11)	−0.0095 (9)	−0.0027 (9)	−0.0079 (9)
C8	0.0259 (11)	0.0300 (12)	0.0296 (12)	−0.0072 (10)	−0.0027 (9)	−0.0075 (9)
C9	0.0240 (11)	0.0312 (12)	0.0236 (11)	−0.0130 (9)	−0.0074 (9)	−0.0066 (9)
C10	0.0291 (12)	0.0299 (12)	0.0242 (11)	−0.0128 (10)	−0.0076 (9)	−0.0014 (9)
C11	0.0337 (12)	0.0433 (14)	0.0179 (10)	−0.0239 (11)	−0.0006 (9)	−0.0041 (10)
C12	0.0312 (12)	0.0369 (14)	0.0259 (12)	−0.0131 (10)	0.0015 (9)	−0.0150 (10)
C13	0.0279 (11)	0.0252 (11)	0.0241 (11)	−0.0103 (9)	−0.0035 (9)	−0.0068 (9)
C14	0.0248 (11)	0.0277 (12)	0.0196 (10)	−0.0150 (9)	−0.0023 (8)	−0.0052 (9)
C15	0.0285 (11)	0.0251 (12)	0.0204 (11)	−0.0105 (9)	−0.0021 (9)	−0.0037 (9)
O2	0.0442 (9)	0.0517 (10)	0.0228 (8)	−0.0262 (8)	0.0016 (7)	−0.0103 (7)
N3	0.0329 (11)	0.0411 (12)	0.0199 (10)	−0.0177 (9)	0.0018 (8)	−0.0088 (8)
C16	0.0294 (12)	0.0319 (13)	0.0272 (12)	−0.0128 (10)	−0.0004 (10)	−0.0079 (10)

Geometric parameters (Å, °)

O1—C15	1.245 (2)	C7—H7B	0.9900

N1—C15	1.366 (2)	C8—C9	1.515 (3)
N1—C14	1.438 (2)	C8—H8A	0.9900
N1—C1	1.444 (2)	C8—H8B	0.9900
N2—C15	1.345 (2)	C9—C14	1.393 (3)
N2—H1N	0.93 (2)	C9—C10	1.413 (3)
N2—H2N	0.87 (2)	C10—C11	1.369 (3)
C1—C2	1.378 (3)	C10—H10	0.9500
C1—C6	1.386 (3)	C11—C12	1.384 (3)
C2—C3	1.384 (3)	C11—H11	0.9500
C2—H2	0.9500	C12—C13	1.382 (3)
C3—C4	1.389 (3)	C12—H12	0.9500
C3—H3	0.9500	C13—C14	1.387 (3)
C4—C5	1.383 (3)	C13—H13	0.9500
C4—H4	0.9500	O2—C16	1.228 (2)
C5—C6	1.393 (3)	N3—C16	1.318 (3)
C5—H5	0.9500	N3—H3N	0.88 (2)
C6—C7	1.499 (3)	N3—H4N	0.94 (2)
C7—C8	1.524 (3)	C16—H16	1.026 (19)
C7—H7A	0.9900		
?···?	?		
C15—N1—C14	120.22 (14)	C7—C8—H8A	107.8
C15—N1—C1	121.60 (15)	C9—C8—H8B	107.8
C14—N1—C1	116.19 (14)	C7—C8—H8B	107.8
C15—N2—H1N	120.6 (13)	H8A—C8—H8B	107.1
C15—N2—H2N	117.3 (13)	C14—C9—C10	116.65 (19)
H1N—N2—H2N	117.9 (19)	C14—C9—C8	126.67 (17)
C2—C1—C6	122.17 (18)	C10—C9—C8	116.67 (19)
C2—C1—N1	121.25 (18)	C11—C10—C9	122.3 (2)
C6—C1—N1	116.52 (17)	C11—C10—H10	118.9
C1—C2—C3	119.3 (2)	C9—C10—H10	118.9
C1—C2—H2	120.3	C10—C11—C12	119.96 (18)
C3—C2—H2	120.3	C10—C11—H11	120.0
C2—C3—C4	119.43 (19)	C12—C11—H11	120.0
C2—C3—H3	120.3	C13—C12—C11	119.20 (19)
C4—C3—H3	120.3	C13—C12—H12	120.4
C5—C4—C3	120.70 (19)	C11—C12—H12	120.4
C5—C4—H4	119.6	C12—C13—C14	120.90 (19)
C3—C4—H4	119.6	C12—C13—H13	119.5
C4—C5—C6	120.3 (2)	C14—C13—H13	119.5
C4—C5—H5	119.8	C13—C14—C9	120.99 (17)
C6—C5—H5	119.8	C13—C14—N1	117.68 (18)
C1—C6—C5	118.01 (18)	C9—C14—N1	121.21 (17)
C1—C6—C7	119.05 (17)	O1—C15—N2	121.61 (18)
C5—C6—C7	122.8 (2)	O1—C15—N1	121.52 (17)
C6—C7—C8	111.57 (16)	N2—C15—N1	116.85 (16)
C6—C7—H7A	109.3	C16—N3—H3N	121.0 (13)
C8—C7—H7A	109.3	C16—N3—H4N	121.2 (13)
C6—C7—H7B	109.3	H3N—N3—H4N	116.8 (19)

C8—C7—H7B	109.3	O2—C16—N3	124.7 (2)
H7A—C7—H7B	108.0	O2—C16—H16	121.8 (10)
C9—C8—C7	118.16 (17)	N3—C16—H16	113.5 (10)
C9—C8—H8A	107.8		
C15—N1—C1—C2	93.5 (2)	C14—C9—C10—C11	1.1 (3)
C14—N1—C1—C2	−102.6 (2)	C8—C9—C10—C11	179.94 (17)
C15—N1—C1—C6	−89.2 (2)	C9—C10—C11—C12	0.5 (3)
C14—N1—C1—C6	74.8 (2)	C10—C11—C12—C13	−2.0 (3)
C6—C1—C2—C3	1.3 (3)	C11—C12—C13—C14	1.9 (3)
N1—C1—C2—C3	178.50 (16)	C12—C13—C14—C9	−0.2 (3)
C1—C2—C3—C4	−1.1 (3)	C12—C13—C14—N1	−176.34 (16)
C2—C3—C4—C5	0.0 (3)	C10—C9—C14—C13	−1.3 (2)
C3—C4—C5—C6	1.0 (3)	C8—C9—C14—C13	−179.96 (18)
C2—C1—C6—C5	−0.3 (3)	C10—C9—C14—N1	174.74 (15)
N1—C1—C6—C5	−177.66 (16)	C8—C9—C14—N1	−4.0 (3)
C2—C1—C6—C7	175.70 (17)	C15—N1—C14—C13	−75.5 (2)
N1—C1—C6—C7	−1.6 (3)	C1—N1—C14—C13	120.26 (19)
C4—C5—C6—C1	−0.8 (3)	C15—N1—C14—C9	108.3 (2)
C4—C5—C6—C7	−176.68 (18)	C1—N1—C14—C9	−55.9 (2)
C1—C6—C7—C8	−70.4 (2)	C14—N1—C15—O1	5.3 (3)
C5—C6—C7—C8	105.5 (2)	C1—N1—C15—O1	168.59 (18)
C6—C7—C8—C9	57.7 (2)	C14—N1—C15—N2	−176.03 (18)
C7—C8—C9—C14	−0.1 (3)	C1—N1—C15—N2	−12.7 (3)
C7—C8—C9—C10	−178.80 (16)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2N···O2	0.87 (2)	2.12 (2)	2.975 (2)	170.0 (19)
N2—H1N···O2^{i}	0.93 (2)	2.12 (2)	2.948 (2)	147.6 (17)
N3—H4N···O1	0.94 (2)	1.99 (2)	2.924 (2)	172.2 (19)
N3—H3N···O1^{ii}	0.88 (2)	2.10 (2)	2.945 (2)	159.7 (18)

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

Fig. 1

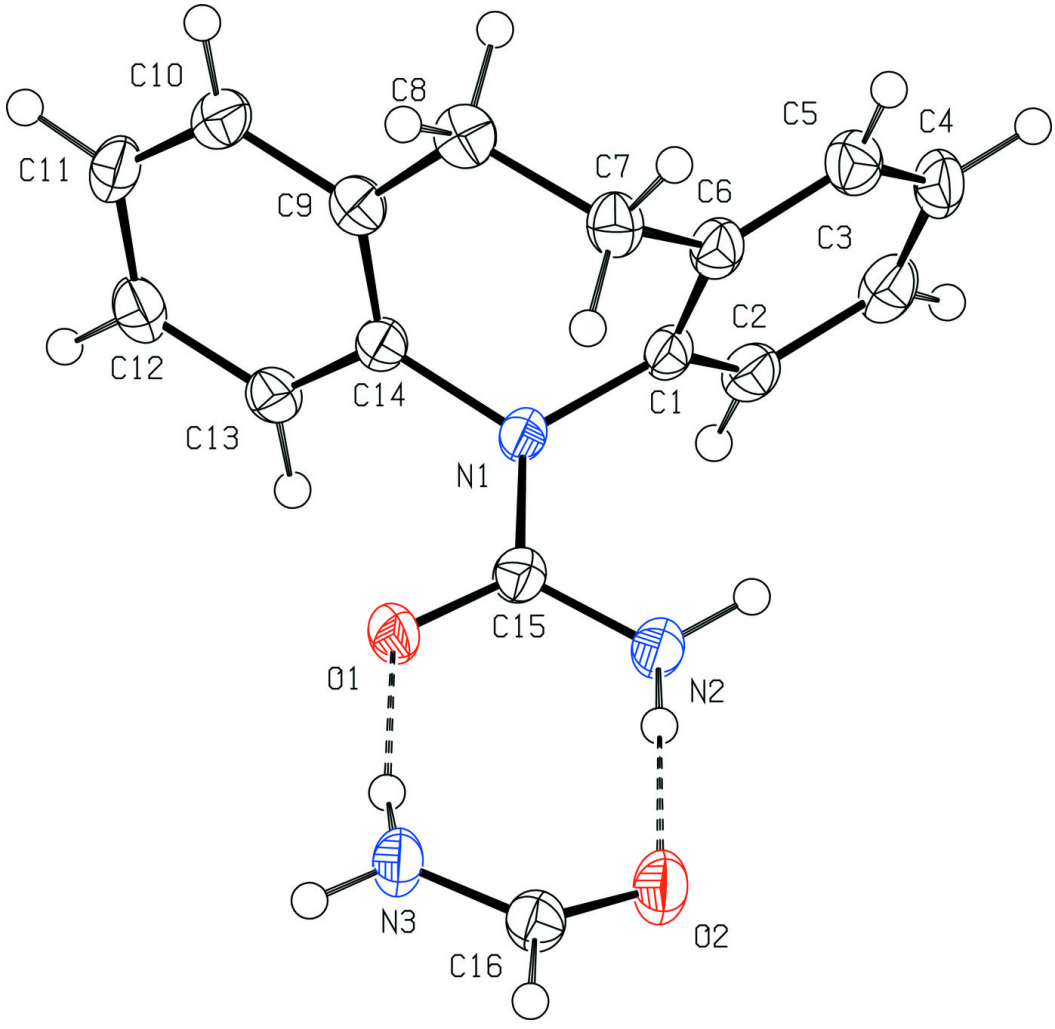

Fig. 2