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A low-temperature redetermination of cyheptamide

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
R factor = 0.035
wR factor = 0.086
Data-to-parameter ratio = 10.8

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

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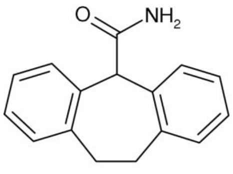
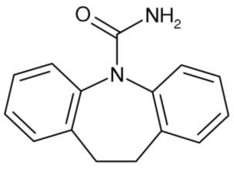
In the title compound [systematic name: 10,11-dihydro-5*H*-dibenz[*a*,*d*]cycloheptene-5-carboxamide], $C_{16}H_{15}NO$, N—H$\cdots$O and N—H$\cdots\pi$ interactions combine to create a catemeric motif that is also observed in crystal structures of the closely related compound dihydrocarbamazepine.

Comment

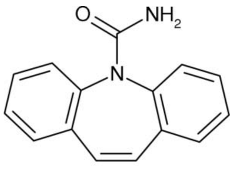
Cyheptamide, (I), is an analogue of dihydrocarbamazepine, (II), the latter being a recognized impurity (Cyr *et al.*, 1987) in the widely used antiepileptic drug carbamazepine, (III). The crystal structure of (I) was first reported by Codding *et al.* (1984) and the structure reported here (Fig. 1) is a low-temperature redetemination. This work 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 (III) and its analogues (Florence, Johnston, Price *et al.*, 2006).



(I)



(II)



(III)

The intermolecular interactions in (I) combine to create the catemeric motif shown in Fig. 2, with the geometric parameters listed in Table 1. Infinite [010] chains of molecules are linked by an N1$\cdots$O1^{i} [symmetry code: (i) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$] hydrogen bond, supplemented by an N—H$\cdots\pi$ interaction, N1$\cdots$*Cg*2^{ii} [symmetry code: (ii) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$], where *Cg*2 is the centroid of ring *R*2 (atoms C10–C15). The robustness of this motif is reflected in the fact that it is observed in all three polymorphic forms of (II) (monoclinic: Bandoli *et al.*, 1992; orthorhombic: Harrison *et al.*, 2006; triclinic: Leech *et al.*, 2006) and in a predicted crystal structure of (III) that is isostructural with the orthorhombic form of (II) (Florence, Leech *et al.*, 2006). It is notable also that the crystal structure of (I) is essentially isostructural with the monoclinic form of (II) [$P2_1/c$; a = 5.433 (3) Å, b = 9.129 (2) Å, c = 24.196 (5) Å, β = 96.47 (3)°, V = 1192.4 (8) Å^3 at T = 150 K; Leech, 2006].

Experimental

A single-crystal of the title compound was selected from the sample as supplied (Sigma–Aldrich Co.) without recrystallization.

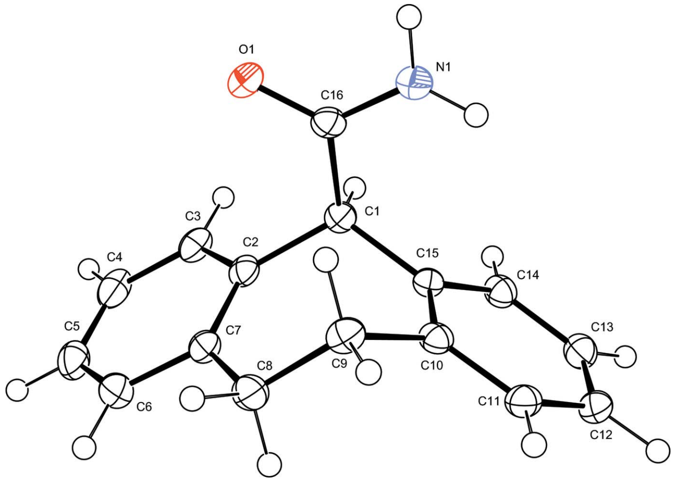


Figure 1
The molecular structure of (I), showing the atom-numbering scheme and 50% probability displacement ellipsoids.

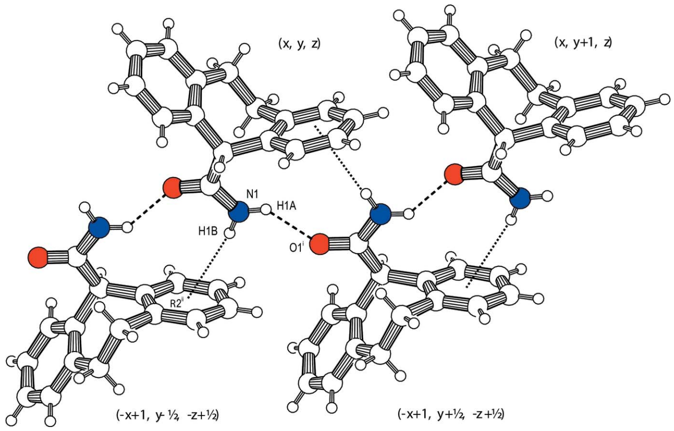


Figure 2
The catemeric motif of (I). Hydrogen bonds are indicated by dashed lines.

Crystal data

$C_{16}H_{15}NO$
$M_r = 237.29$
Monoclinic, $P2_1/c$
$a = 5.6035$ (7) Å
$b = 9.1716$ (11) Å
$c = 23.579$ (3) Å
$\beta = 96.752$ (12)°
$V = 1203.4$ (3) Å^3
$Z = 4$
$D_x = 1.310$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.08$ mm^{-1}
$T = 150$ (2) K
Block, colourless
0.26 × 0.17 × 0.16 mm

Data collection

Oxford Diffraction Gemini diffractometer
ω and φ scans
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006)
$T_{\min} = 0.956$, $T_{\max} = 0.983$
11433 measured reflections
2407 independent reflections
1928 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.029$
$\theta_{\max} = 26.4°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.086$
$S = 1.04$
2407 reflections
223 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0353P)^2 + 0.4305P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.18$ e Å^{-3}
$\Delta\rho_{\min} = -0.16$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.91 (2)	2.13 (2)	2.842 (2)	135 (1)
N1—H1B···$Cg2^{ii}$	0.93 (2)	2.78 (2)	3.676 (1)	162 (1)

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

All H atoms were located in a Fourier difference map and the atomic coordinates and U_{iso} parameters were refined freely. X—H distances refined to N1—H1A = 0.90 (2) Å, N1—H1B = 0.93 (2) Å, C1—H1 = 1.03 (2) and 0.96 (2)–1.01 (2) Å for aromatic H atoms and 0.98 (2)–1.00 (2) Å for the CH_2 H atoms.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Version 011105; Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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