

Charlotte K. Leech,<sup>a</sup> Alastair J. Florence,<sup>b\*</sup> Kenneth Shankland,<sup>a</sup> Norman Shankland<sup>b</sup> and Andrea Johnston<sup>b</sup>

<sup>a</sup>ISIS Facility, Rutherford Appleton Laboratory, Chilton, Didcot, Oxon OX11 0QX, England, and <sup>b</sup>Solid-State Research Group, Strathclyde Institute of Pharmacy and Biomedical Sciences, University of Strathclyde, 27 Taylor Street, Glasgow G4 0NR, Scotland

Correspondence e-mail:  
alastair.florence@strath.ac.uk

#### Key indicators

Single-crystal X-ray study  
 T = 150 K  
 Mean  $\sigma(C-C)$  = 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>.

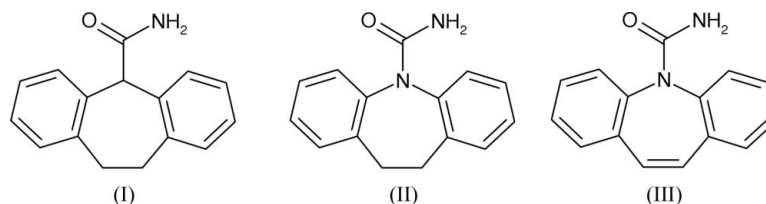
## A low-temperature redetermination of cyheptamide

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

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### Comment

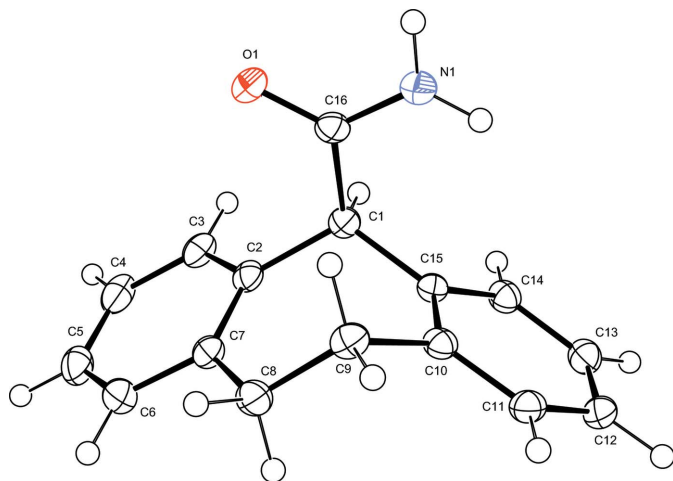
Cyheptamide, (I), is an analogue of dihydrocarbamazepine, (II), the latter being a recognized impurity (Cyr *et al.*, 1987) in the widely used antiepileptic drug carbamazepine, (III). The crystal structure of (I) was first reported by Codding *et al.* (1984) and the structure reported here (Fig. 1) is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



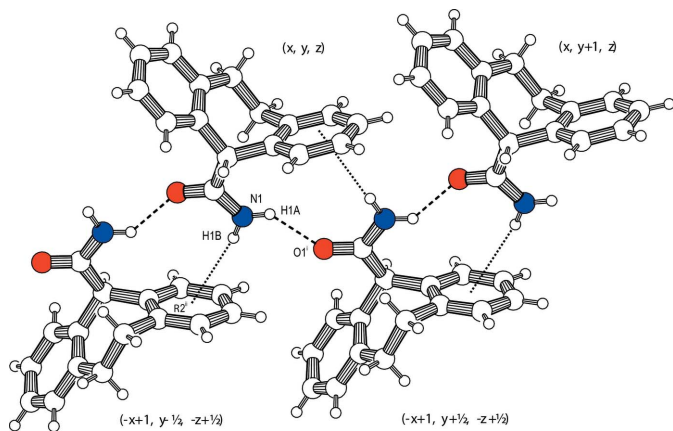
The intermolecular interactions in (I) combine to create the catemeric motif shown in Fig. 2, with the geometric parameters listed in Table 1. Infinite [010] chains of molecules are linked by an N1···O1<sup>i</sup> [symmetry code: (i)  $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$ ] hydrogen bond, supplemented by an N—H··· $\pi$  interaction, N1···Cg2<sup>ii</sup> [symmetry code: (ii)  $-x + 1, y - \frac{1}{2}, -z + \frac{1}{2}$ ], where Cg2 is the centroid of ring R2 (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,  $\beta = 96.47$  (3)°,  $V = 1192.4$  (8) Å<sup>3</sup> 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<sup>3</sup>

$Z = 4$   
 $D_x = 1.310$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 $\mu = 0.08$  mm<sup>-1</sup>  
 $T = 150$  (2) K  
 Block, colourless  
 $0.26 \times 0.17 \times 0.16$  mm

#### Data collection

Oxford Diffraction Gemini diffractometer  
 $\omega$  and  $\varphi$  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  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.029$   
 $\theta_{\text{max}} = 26.4^\circ$

#### 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  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.18 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.16 \text{ e \AA}^{-3}$

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1A\cdots O1^i$	0.91 (2)	2.13 (2)	2.842 (2)	135 (1)
$N1-H1B\cdots Cg2^{ii}$	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  $U_{\text{iso}}$  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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## supporting information

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10,11-dihydro-5*H*-dibenz[*a,d*]cycloheptene-5-carboxamide*Crystal data*

$C_{16}H_{15}NO$

$M_r = 237.29$

Monoclinic,  $P2_1/c$

Hall symbol: -P 2ybc

$a = 5.6035$  (7) Å

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$c = 23.579$  (3) Å

$\beta = 96.752$  (12)°

$V = 1203.4$  (3) Å<sup>3</sup>

$Z = 4$

$F(000) = 504$

$D_x = 1.310$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 5695 reflections

$\theta = 2.4$ – $28.0^\circ$

$\mu = 0.08$  mm<sup>-1</sup>

$T = 150$  K

Block, colourless

$0.26 \times 0.17 \times 0.16$  mm

*Data collection*

Oxford Diffraction Gemini  
diffractometer

Radiation source: Enhance (Mo) X-ray Source  
Graphite monochromator

Detector resolution: 15.9745 pixels mm<sup>-1</sup>

$\omega$  and  $\varphi$  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  $I > 2\sigma(I)$

$R_{\text{int}} = 0.029$

$\theta_{\max} = 26.4^\circ$ ,  $\theta_{\min} = 2.8^\circ$

$h = -6 \rightarrow 6$

$k = -11 \rightarrow 11$

$l = -29 \rightarrow 28$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.035$

$wR(F^2) = 0.086$

$S = 1.04$

2407 reflections

223 parameters

0 restraints

Primary atom site location: structure-invariant  
direct methods

Secondary atom site location: difference Fourier  
map

Hydrogen site location: difference Fourier map

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<sup>-3</sup>

$\Delta\rho_{\min} = -0.16$  e Å<sup>-3</sup>

*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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.37581 (17)	0.71849 (10)	0.21007 (4)	0.0258 (2)
C1	0.1185 (2)	0.91677 (14)	0.17364 (6)	0.0188 (3)
N1	0.4738 (2)	0.94199 (15)	0.24450 (5)	0.0266 (3)
C2	0.0314 (2)	0.81878 (14)	0.12290 (6)	0.0198 (3)
C7	0.1170 (2)	0.82304 (14)	0.06934 (6)	0.0217 (3)
C11	0.3338 (2)	1.27115 (15)	0.11228 (6)	0.0221 (3)
C16	0.3378 (2)	0.85046 (14)	0.21032 (6)	0.0201 (3)
C15	0.1420 (2)	1.07749 (14)	0.15933 (6)	0.0181 (3)
C9	0.4629 (2)	1.01088 (15)	0.09812 (6)	0.0219 (3)
C3	-0.1529 (2)	0.72033 (15)	0.13157 (7)	0.0239 (3)
C14	-0.0075 (2)	1.18044 (15)	0.18046 (6)	0.0216 (3)
C8	0.3149 (3)	0.92184 (16)	0.05173 (6)	0.0242 (3)
C6	0.0123 (3)	0.72846 (16)	0.02645 (7)	0.0292 (3)
C4	-0.2550 (3)	0.62948 (16)	0.08836 (7)	0.0296 (4)
C10	0.3137 (2)	1.12316 (15)	0.12427 (6)	0.0194 (3)
C12	0.1827 (3)	1.37355 (16)	0.13308 (6)	0.0245 (3)
C13	0.0110 (3)	1.32744 (16)	0.16701 (6)	0.0246 (3)
C5	-0.1717 (3)	0.63338 (16)	0.03540 (7)	0.0319 (4)
H3	-0.207 (3)	0.7207 (17)	0.1690 (8)	0.031 (4)*
H9B	0.594 (3)	1.0627 (16)	0.0810 (6)	0.022 (4)*
H9A	0.537 (2)	0.9436 (16)	0.1282 (7)	0.018 (4)*
H11	0.455 (3)	1.3047 (17)	0.0885 (7)	0.028 (4)*
H8B	0.239 (3)	0.9875 (17)	0.0219 (7)	0.024 (4)*
H6	0.076 (3)	0.7306 (18)	-0.0120 (8)	0.036 (5)*
H4	-0.388 (3)	0.5642 (18)	0.0965 (7)	0.032 (4)*
H14	-0.129 (3)	1.1473 (17)	0.2044 (7)	0.027 (4)*
H8A	0.422 (3)	0.8596 (18)	0.0326 (7)	0.031 (4)*
H1	-0.013 (3)	0.9104 (15)	0.2006 (7)	0.022 (4)*
H12	0.204 (3)	1.4789 (18)	0.1245 (7)	0.030 (4)*
H13	-0.097 (3)	1.3978 (18)	0.1810 (7)	0.032 (4)*
H5	-0.237 (3)	0.5704 (18)	0.0052 (8)	0.032 (4)*
H1B	0.596 (3)	0.900 (2)	0.2693 (8)	0.042 (5)*
H1A	0.452 (3)	1.040 (2)	0.2440 (8)	0.040 (5)*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0307 (5)	0.0183 (5)	0.0277 (6)	0.0030 (4)	-0.0002 (4)	0.0034 (4)
C1	0.0190 (6)	0.0194 (7)	0.0186 (7)	0.0007 (5)	0.0051 (5)	0.0008 (5)
N1	0.0313 (7)	0.0229 (7)	0.0237 (7)	-0.0003 (5)	-0.0049 (5)	0.0015 (5)
C2	0.0188 (6)	0.0168 (7)	0.0232 (7)	0.0047 (5)	0.0001 (5)	0.0006 (5)
C7	0.0235 (7)	0.0184 (7)	0.0224 (8)	0.0055 (5)	0.0000 (5)	0.0002 (5)
C11	0.0224 (7)	0.0228 (7)	0.0204 (8)	-0.0014 (6)	-0.0005 (6)	0.0029 (5)
C16	0.0230 (7)	0.0219 (8)	0.0162 (7)	-0.0004 (5)	0.0055 (5)	0.0027 (5)
C15	0.0187 (6)	0.0184 (7)	0.0166 (7)	0.0008 (5)	-0.0006 (5)	0.0002 (5)
C9	0.0210 (7)	0.0228 (7)	0.0231 (8)	0.0023 (6)	0.0066 (6)	0.0033 (6)
C3	0.0212 (7)	0.0194 (7)	0.0310 (9)	0.0032 (5)	0.0023 (6)	0.0033 (6)
C14	0.0213 (7)	0.0234 (7)	0.0202 (7)	0.0020 (6)	0.0023 (6)	-0.0020 (5)
C8	0.0292 (8)	0.0244 (8)	0.0202 (8)	0.0044 (6)	0.0080 (6)	0.0001 (6)
C6	0.0374 (8)	0.0254 (8)	0.0235 (8)	0.0059 (6)	-0.0022 (7)	-0.0020 (6)
C4	0.0247 (7)	0.0190 (7)	0.0428 (10)	0.0005 (6)	-0.0053 (7)	0.0012 (6)
C10	0.0181 (6)	0.0219 (7)	0.0176 (7)	0.0014 (5)	-0.0009 (5)	0.0002 (5)
C12	0.0291 (7)	0.0190 (7)	0.0236 (8)	0.0013 (6)	-0.0044 (6)	0.0020 (6)
C13	0.0254 (7)	0.0228 (7)	0.0245 (8)	0.0068 (6)	-0.0012 (6)	-0.0033 (6)
C5	0.0360 (8)	0.0220 (8)	0.0341 (9)	0.0022 (6)	-0.0116 (7)	-0.0057 (6)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

O1—C16	1.2291 (16)	C9—C8	1.529 (2)
C1—C15	1.5214 (18)	C9—H9B	1.000 (15)
C1—C2	1.5296 (19)	C9—H9A	0.994 (15)
C1—C16	1.5423 (18)	C3—C4	1.386 (2)
C1—H1	1.028 (15)	C3—H3	0.966 (18)
N1—C16	1.3381 (18)	C14—C13	1.392 (2)
N1—H1B	0.93 (2)	C14—H14	0.983 (16)
N1—H1A	0.904 (19)	C8—H8B	0.985 (16)
C2—C7	1.403 (2)	C8—H8A	0.978 (17)
C2—C3	1.405 (2)	C6—C5	1.385 (2)
C7—C6	1.407 (2)	C6—H6	1.013 (19)
C7—C8	1.527 (2)	C4—C5	1.384 (2)
C11—C12	1.392 (2)	C4—H4	0.993 (17)
C11—C10	1.3937 (19)	C12—C13	1.388 (2)
C11—H11	0.982 (16)	C12—H12	0.996 (16)
C15—C14	1.3927 (19)	C13—H13	0.967 (17)
C15—C10	1.4044 (19)	C5—H5	0.956 (17)
C9—C10	1.5034 (19)		
C15—C1—C2	115.08 (11)	C4—C3—C2	121.75 (15)
C15—C1—C16	114.98 (11)	C4—C3—H3	121.7 (10)
C2—C1—C16	111.51 (11)	C2—C3—H3	116.5 (10)
C15—C1—H1	106.2 (8)	C13—C14—C15	120.75 (13)
C2—C1—H1	105.3 (8)	C13—C14—H14	120.3 (9)

C16—C1—H1	102.2 (8)	C15—C14—H14	118.9 (9)
C16—N1—H1B	116.4 (11)	C7—C8—C9	118.20 (12)
C16—N1—H1A	123.0 (12)	C7—C8—H8B	106.8 (9)
H1B—N1—H1A	120.5 (16)	C9—C8—H8B	109.8 (9)
C7—C2—C3	118.94 (13)	C7—C8—H8A	106.6 (9)
C7—C2—C1	125.19 (12)	C9—C8—H8A	109.3 (10)
C3—C2—C1	115.86 (13)	H8B—C8—H8A	105.4 (13)
C2—C7—C6	118.15 (13)	C5—C6—C7	122.24 (15)
C2—C7—C8	126.67 (12)	C5—C6—H6	119.7 (10)
C6—C7—C8	115.17 (13)	C7—C6—H6	118.1 (10)
C12—C11—C10	121.24 (14)	C5—C4—C3	119.58 (14)
C12—C11—H11	118.8 (9)	C5—C4—H4	122.1 (10)
C10—C11—H11	119.9 (9)	C3—C4—H4	118.3 (10)
O1—C16—N1	122.28 (13)	C11—C10—C15	119.15 (12)
O1—C16—C1	120.84 (12)	C11—C10—C9	121.46 (13)
N1—C16—C1	116.76 (12)	C15—C10—C9	119.31 (12)
C14—C15—C10	119.41 (12)	C13—C12—C11	119.30 (13)
C14—C15—C1	120.41 (12)	C13—C12—H12	121.2 (9)
C10—C15—C1	120.18 (11)	C11—C12—H12	119.5 (9)
C10—C9—C8	112.23 (11)	C12—C13—C14	120.12 (13)
C10—C9—H9B	108.1 (8)	C12—C13—H13	119.7 (10)
C8—C9—H9B	109.1 (9)	C14—C13—H13	120.2 (10)
C10—C9—H9A	109.9 (8)	C4—C5—C6	119.32 (14)
C8—C9—H9A	109.0 (8)	C4—C5—H5	121.0 (10)
H9B—C9—H9A	108.4 (11)	C6—C5—H5	119.7 (10)

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1A...O1 <sup>i</sup>	0.91 (2)	2.13 (2)	2.842 (2)	135 (1)
N1—H1B...Cg2 <sup>ii</sup>	0.93 (2)	2.78 (2)	3.676 (1)	162 (1)

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