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A tetraazamacrocycle with benzyl substituents

Jong-Ha Choi,[a]‡ William Clegg,[a]* Ross W. Harrington,[a] Hyang-Mi Yoon[b] and Yong Pyo Hong[b]

[a]School of Natural Sciences (Chemistry), Bedson Building, University of Newcastle upon Tyne, Newcastle upon Tyne NE1 7RU, England, and [b]Department of Chemistry, Andong National University, Andong 760-749, South Korea

‡ Permanent address: Department of Chemistry, Andong National University, Andong 760-749, South Korea. E-mail: jhchoi@andong.ac.kr

Correspondence e-mail: w.clegg@ncl.ac.uk

The molecule of the title compound, 2,13-dibenzyl-5,16-dimethyl-2,6,13,17-tetraazatricyclo[16.4.0^{1,18}.0^{7,12}]docosane, $C_{34}H_{52}N_4$, is centrosymmetric. The 14-membered macrocycle adopts the stable *trans*-III (*RRSS*) configuration, with one benzyl group above and the other below the macrocycle mean plane. The crystal structure is stabilized by N—H···N hydrogen bonds. There is no intermolecular π–π interaction between benzyl planes, which are almost 6Å apart.

Comment

The 14-membered cyclam (1,4,8,11-tetraazacyclotetradecane) ligand and its substituted derivatives are involved in diverse application fields, such as catalysis, enzyme mimics, chemical sensors, selective metal ion recovery, pharmacology and therapy (Meyer *et al.*, 1998, and references therein). Metal–cyclam adducts have a moderately flexible structure, and can adopt both planar (*trans*) and folded (*cis*) configurations (Poon *et al.*, 1980). There are five configurational *trans* isomers for the N₄-donor set of cyclam which differ in the chirality of the *sec*-NH centres. The *trans*-V configuration can fold to form a *cis*-V isomer. We previously described the spectroscopic and ligand-field properties based on the emission, far-IR and electronic spectroscopy of chromium(III) complexes with 14-membered cyclam derivatives and two auxiliary ligands (Choi, 2000a,b; Choi, Oh, Suzuki & Kaizaki, 2004; Choi, Oh, Linder & Schönherr, 2004; Choi, Oh, Lim & Park, 2004). The modification of C- and/or N-configurational isomers of polyaza macrocyclic ligands to control the chemical and physical properties of metal complexes has been of considerable interest (Dong & Lindoy, 2001). The 14-membered cyclam containing two 1,2-diaminocyclohexanediamine subunits occurs in both *cis*- and *trans*-configurations (Kang & Jeong, 2003).

Octahedral transition metal complexes with cyclam derivatives display UV–visible electronic absorption bands. The *d–d* transitions are symmetry forbidden, so extinction coefficients are relatively small. Thus, it is necessary to prepare new systems with ligand-based chromophores with higher extinction coefficients (Bernhardt & Riley, 2002). Benzyl groups are introduced as possible internal sensitizers of the macrocyclic ligand and its metal complexes. Recently, the synthesis and chemical properties of tetraaza marcrocycles containing two pendant arms and their nickel(II) and copper(II) complexes have been reported (Kang & Kim, 2003). However, the structures of (I) and its complexes have not previously been determined by X-ray crystallography. We report here the crystal structure of the title macrocycle, (I), with the aim of confirming the stereochemistry of the attached groups and

gaining further insight into its coordination properties for various transition metal ions.
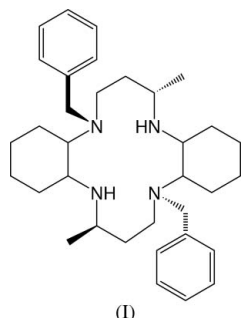
Selected bond lengths and angles are listed in Table 1. A perspective drawing of the centrosymmetric molecular structure is depicted in Fig. 1. Bond distances and angles are in normal ranges. The crystal structure of (I) shows a configuration with one benzyl group above and the other below the macrocycle mean plane, this being the sterically least-hindered configuration. The cyclam ligand has the *trans*-III (*RRSS*) form, consistent with a crystallographic centre of symmetry. The four N atoms are exactly coplanar as a result of the centrosymmetry of the molecule. The $(CH_2)_4$ part of the cyclohexane subunit is *anti* with respect to the macrocycle plane. The intramolecular hydrogen bond between secondary N2—H2*N* and tertiary N1 lends some rigidity to the cyclam ring. The closest intermolecular distance between benzyl rings is >5.8Å, which is not within the range associated with π–π interactions (Munakata *et al.*, 1994). We can anticipate that a related new macrocycle, containing two naphthylmethyl pendant arms, will adopt the most stable *trans*-III configuration, in which the two H atoms on the secondary amines and the two naphthylmethyl groups are likewise oriented on opposite sides of the macrocycle plane. The crystal structure of a copper(II) complex with the title ligand will be reported later (Choi *et al.*, 2006).

Experimental

The title macrocycle was prepared according to a published procedure (Kang *et al.*, 1991). To a solution of 5,16-dimethyl-2,6,13,17-tetraazatricyclo[14.4.0^{1,18}.0^{7,12}]docosane (8.814 g, 2.42 mmol) in methanol (10 ml) were added benzyl bromide (0.838 g, 4.90 mmol) and a solution containing Na_2CO_3 (0.520 g, 4.90 mmol) in water (4 ml). The solution was refluxed for 24 h and cooled to room temperature, and the resultant white solid was filtered off and washed with cold water. The crude compound was recrystallized from tetrahydrofuran to give colourless crystals suitable for X-ray analysis (yield 0.828 g, 66.3%). The IR spectrum (KBr) showed peaks at 3264 (N—H), 3063 and 3027 (aromatic C—H), 1603 and 1484 cm^{-1} (aromatic C=C). Analysis found: C 79.71, H 10.48, N 10.99%; $C_{34}H_{52}N_4$ requires: C 79.02, H 10.14, N 10.84%. The new macrocycle containing two naphthylmethyl pendant arms was also prepared by a similar method, except that 1-chloromethylnaphthalene was used instead of benzyl bromide.
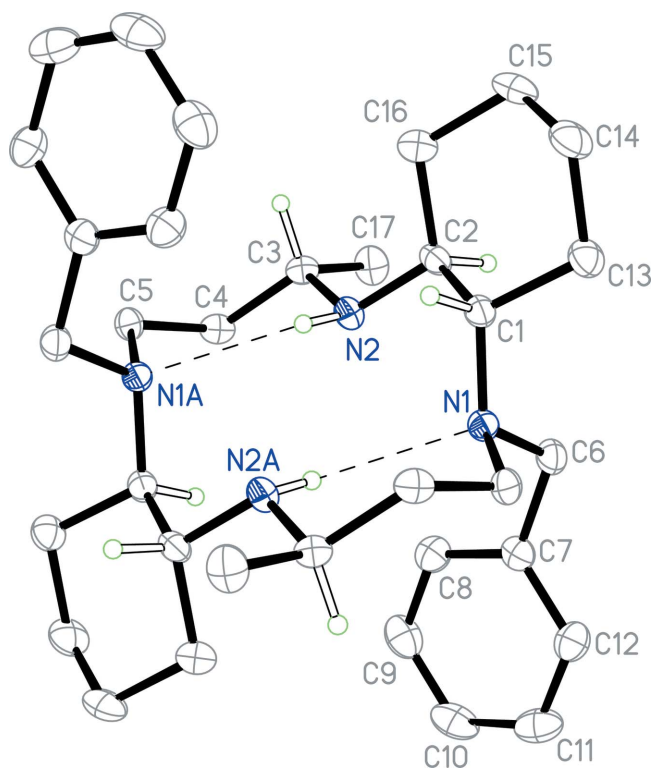
Figure 1
The molecular structure with 50% probability displacement ellipsoids. H atoms have been omitted, except for those on N atoms and major asymmetric centres. The dashed lines represent N—H···N hydrogen bonds. The suffix A corresponds to symmetry code (i) in Tables 1 and 2.

Crystal data

$C_{34}H_{52}N_4$	$Z = 1$
$M_r = 516.80$	$D_x = 1.155$ Mg m^{-3}
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 8.8976\ (12)$ Å	Cell parameters from 5357
$b = 9.2438\ (12)$ Å	reflections
$c = 9.2535\ (12)$ Å	$\theta = 2.3$–$28.7°$
$\alpha = 82.467\ (2)°$	$\mu = 0.07$ mm^{-1}
$\beta = 87.605\ (2)°$	$T = 150\ (2)$ K
$\gamma = 79.982\ (2)°$	Block, colourless
$V = 742.87\ (17)$ Å^3	$0.56 \times 0.55 \times 0.50$ mm

Data collection

Bruker SMART 1K CCD diffractometer	2363 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.024$
Absorption correction: none	$\theta_{max} = 25.0°$
5375 measured reflections	$h = -10 \rightarrow 10$
2590 independent reflections	$k = -10 \rightarrow 10$
	$l = -10 \rightarrow 10$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0312P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.064$	$+ 0.5246P]$
$wR(F^2) = 0.131$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.27$	$(\Delta/\sigma)_{max} < 0.001$
2590 reflections	$\Delta\rho_{max} = 0.27$ e Å^{-3}
178 parameters	$\Delta\rho_{min} = -0.19$ e Å^{-3}
H atoms treated by a mixture of independent and constrained refinement	Extinction correction: *SHELXTL*
	Extinction coefficient: 0.015 (4)

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Table 1
Selected geometric parameters (Å, °).

N1—C5[i]	1.463 (3)	N2—C2	1.463 (3)
N1—C6	1.468 (3)	N2—C3	1.466 (3)
N1—C1	1.474 (3)		
C5[i]—N1—C6	110.47 (15)	C6—N1—C1	113.44 (15)
C5[i]—N1—C1	113.92 (15)	C2—N2—C3	116.53 (16)

Symmetry code: (i) $-x+1, -y+1, -z+1$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N2—H2$N\cdots$N1[i]	0.86 (2)	2.32 (2)	3.025 (2)	139.9 (19)

Symmetry code: (i) $-x+1, -y+1, -z+1$.

The H atom bonded to N2 was located in a difference map and refined freely. Other H atoms were positioned geometrically, with C—H distances of 0.95–1.00Å, and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and local programs.

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supporting information

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2,13-dibenzyl-5,16-dimethyl-2,6,13,17- tetraazatricyclo[16.4.0^{1,18}.0^{7,12}]docosane

Crystal data

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$M_r = 516.80$	$F(000) = 284$
Triclinic, $P\overline{1}$	$D_x = 1.155$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 8.8976$ (12) Å	Cell parameters from 5357 reflections
$b = 9.2438$ (12) Å	$\theta = 2.3$–28.7°
$c = 9.2535$ (12) Å	$\mu = 0.07$ mm^{-1}
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$\beta = 87.605$ (2)°	Block, colourless
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Radiation source: sealed tube	$R_{int} = 0.024$
Graphite monochromator	$\theta_{max} = 25.0°$, $\theta_{min} = 2.2°$
ω scans	$h = -10 \rightarrow 10$
5375 measured reflections	$k = -10 \rightarrow 10$
2590 independent reflections	$l = -10 \rightarrow 10$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.064$	
$wR(F^2) = 0.131$	$w = 1/[\sigma^2(F_o^2) + (0.0312P)^2 + 0.5246P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.27$	
2590 reflections	$(\Delta/\sigma)_{max} < 0.001$
178 parameters	$\Delta\rho_{max} = 0.27$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.19$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: SHELXTL, Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.015 (4)

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.

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

	x	y	z	U_{iso}*/U_{eq}
N1	0.63153 (18)	0.26660 (17)	0.59479 (18)	0.0170 (4)
N2	0.49804 (19)	0.42433 (19)	0.33715 (18)	0.0191 (4)
H2N	0.508 (2)	0.503 (3)	0.372 (2)	0.018 (6)*
C1	0.7304 (2)	0.2942 (2)	0.4658 (2)	0.0182 (5)
H1	0.7706	0.3866	0.4766	0.022*
C2	0.6433 (2)	0.3248 (2)	0.3222 (2)	0.0194 (5)
H2	0.6195	0.2283	0.2999	0.023*
C3	0.4077 (2)	0.4704 (2)	0.2050 (2)	0.0195 (5)
H3	0.4706	0.5203	0.1283	0.023*
C4	0.2659 (2)	0.5800 (2)	0.2386 (2)	0.0203 (5)
H4A	0.2132	0.5362	0.3255	0.024*
H4B	0.1958	0.5943	0.1557	0.024*
C5	0.2963 (2)	0.7312 (2)	0.2666 (2)	0.0200 (5)
H5A	0.3623	0.7682	0.1864	0.024*
H5B	0.1980	0.8007	0.2632	0.024*
C6	0.5653 (2)	0.1314 (2)	0.5978 (2)	0.0217 (5)
H6A	0.6381	0.0455	0.6427	0.026*
H6B	0.5476	0.1148	0.4968	0.026*
C7	0.4164 (2)	0.1427 (2)	0.6833 (2)	0.0205 (5)
C8	0.2895 (2)	0.2395 (2)	0.6271 (2)	0.0254 (5)
H8	0.2971	0.2973	0.5352	0.030*
C9	0.1521 (3)	0.2528 (3)	0.7035 (3)	0.0309 (6)
H9	0.0658	0.3192	0.6636	0.037*
C10	0.1393 (3)	0.1699 (3)	0.8378 (3)	0.0326 (6)
H10	0.0445	0.1790	0.8899	0.039*
C11	0.2645 (3)	0.0745 (3)	0.8952 (3)	0.0321 (6)
H11	0.2565	0.0180	0.9879	0.039*
C12	0.4027 (3)	0.0601 (2)	0.8186 (2)	0.0257 (5)
H12	0.4886	−0.0067	0.8588	0.031*
C13	0.8702 (2)	0.1735 (2)	0.4506 (2)	0.0239 (5)
H13A	0.9238	0.1483	0.5445	0.029*
H13B	0.8366	0.0831	0.4263	0.029*
C14	0.9794 (2)	0.2261 (3)	0.3314 (3)	0.0297 (5)
H14A	1.0186	0.3124	0.3591	0.036*
H14B	1.0674	0.1460	0.3215	0.036*
C15	0.8990 (2)	0.2694 (3)	0.1867 (2)	0.0297 (5)
H15A	0.8761	0.1793	0.1509	0.036*
H15B	0.9678	0.3145	0.1142	0.036*
C16	0.7505 (2)	0.3790 (2)	0.2000 (2)	0.0244 (5)

H16A	0.6968	0.3958	0.1062	0.029*
H16B	0.7753	0.4749	0.2194	0.029*
C17	0.3638 (3)	0.3360 (2)	0.1489 (2)	0.0273 (5)
H17A	0.4553	0.2755	0.1120	0.041*
H17B	0.2907	0.3690	0.0701	0.041*
H17C	0.3172	0.2770	0.2286	0.041*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0163 (9)	0.0163 (8)	0.0188 (9)	−0.0033 (7)	0.0009 (7)	−0.0028 (7)
N2	0.0194 (9)	0.0177 (9)	0.0203 (9)	−0.0011 (7)	−0.0003 (7)	−0.0055 (7)
C1	0.0161 (10)	0.0162 (10)	0.0225 (11)	−0.0034 (8)	0.0019 (8)	−0.0034 (8)
C2	0.0186 (11)	0.0188 (10)	0.0214 (11)	−0.0025 (8)	0.0038 (8)	−0.0071 (8)
C3	0.0198 (11)	0.0229 (11)	0.0163 (10)	−0.0054 (8)	0.0004 (8)	−0.0023 (8)
C4	0.0168 (10)	0.0272 (11)	0.0173 (10)	−0.0047 (9)	−0.0018 (8)	−0.0028 (8)
C5	0.0165 (10)	0.0191 (11)	0.0221 (11)	0.0018 (8)	−0.0014 (8)	−0.0002 (8)
C6	0.0208 (11)	0.0166 (10)	0.0274 (12)	−0.0028 (8)	0.0019 (9)	−0.0025 (8)
C7	0.0212 (11)	0.0188 (10)	0.0242 (11)	−0.0077 (8)	0.0007 (9)	−0.0072 (8)
C8	0.0244 (12)	0.0253 (12)	0.0269 (12)	−0.0065 (9)	−0.0015 (9)	−0.0017 (9)
C9	0.0201 (12)	0.0310 (13)	0.0428 (14)	−0.0035 (9)	−0.0009 (10)	−0.0105 (11)
C10	0.0216 (12)	0.0448 (15)	0.0370 (14)	−0.0142 (11)	0.0091 (10)	−0.0178 (11)
C11	0.0354 (14)	0.0399 (14)	0.0254 (12)	−0.0197 (11)	0.0050 (10)	−0.0048 (10)
C12	0.0263 (12)	0.0224 (11)	0.0291 (12)	−0.0074 (9)	−0.0030 (9)	−0.0005 (9)
C13	0.0185 (11)	0.0240 (11)	0.0283 (12)	−0.0014 (9)	0.0003 (9)	−0.0035 (9)
C14	0.0180 (11)	0.0340 (13)	0.0361 (13)	−0.0021 (9)	0.0062 (10)	−0.0063 (10)
C15	0.0231 (12)	0.0383 (13)	0.0283 (12)	−0.0050 (10)	0.0111 (9)	−0.0100 (10)
C16	0.0240 (11)	0.0292 (12)	0.0206 (11)	−0.0058 (9)	0.0023 (9)	−0.0046 (9)
C17	0.0286 (12)	0.0261 (12)	0.0280 (12)	−0.0032 (9)	−0.0066 (9)	−0.0064 (9)

Geometric parameters (Å, º)

N1—C5^i	1.463 (3)	C7—C12	1.391 (3)
N1—C6	1.468 (3)	C8—C9	1.381 (3)
N1—C1	1.474 (3)	C8—H8	0.950
N2—C2	1.463 (3)	C9—C10	1.383 (3)
N2—C3	1.466 (3)	C9—H9	0.950
N2—H2N	0.86 (2)	C10—C11	1.374 (3)
C1—C13	1.535 (3)	C10—H10	0.950
C1—C2	1.535 (3)	C11—C12	1.389 (3)
C1—H1	1.000	C11—H11	0.950
C2—C16	1.538 (3)	C12—H12	0.950
C2—H2	1.000	C13—C14	1.526 (3)
C3—C17	1.523 (3)	C13—H13A	0.990
C3—C4	1.523 (3)	C13—H13B	0.990
C3—H3	1.000	C14—C15	1.519 (3)
C4—C5	1.524 (3)	C14—H14A	0.990
C4—H4A	0.990	C14—H14B	0.990

C4—H4B	0.990	C15—C16	1.530 (3)
C5—N1ⁱ	1.463 (3)	C15—H15A	0.990
C5—H5A	0.990	C15—H15B	0.990
C5—H5B	0.990	C16—H16A	0.990
C6—C7	1.509 (3)	C16—H16B	0.990
C6—H6A	0.990	C17—H17A	0.980
C6—H6B	0.990	C17—H17B	0.980
C7—C8	1.386 (3)	C17—H17C	0.980
C5ⁱ—N1—C6	110.47 (15)	C9—C8—C7	120.7 (2)
C5ⁱ—N1—C1	113.92 (15)	C9—C8—H8	119.7
C6—N1—C1	113.44 (15)	C7—C8—H8	119.7
C2—N2—C3	116.53 (16)	C8—C9—C10	120.4 (2)
C2—N2—H2N	112.9 (14)	C8—C9—H9	119.8
C3—N2—H2N	107.0 (14)	C10—C9—H9	119.8
N1—C1—C13	115.25 (16)	C11—C10—C9	119.5 (2)
N1—C1—C2	113.08 (16)	C11—C10—H10	120.2
C13—C1—C2	108.70 (16)	C9—C10—H10	120.2
N1—C1—H1	106.4	C10—C11—C12	120.3 (2)
C13—C1—H1	106.4	C10—C11—H11	119.8
C2—C1—H1	106.4	C12—C11—H11	119.8
N2—C2—C1	110.90 (16)	C11—C12—C7	120.5 (2)
N2—C2—C16	114.93 (17)	C11—C12—H12	119.8
C1—C2—C16	108.23 (16)	C7—C12—H12	119.8
N2—C2—H2	107.5	C14—C13—C1	110.68 (17)
C1—C2—H2	107.5	C14—C13—H13A	109.5
C16—C2—H2	107.5	C1—C13—H13A	109.5
N2—C3—C17	110.22 (17)	C14—C13—H13B	109.5
N2—C3—C4	109.32 (16)	C1—C13—H13B	109.5
C17—C3—C4	110.73 (17)	H13A—C13—H13B	108.1
N2—C3—H3	108.8	C15—C14—C13	110.71 (18)
C17—C3—H3	108.8	C15—C14—H14A	109.5
C4—C3—H3	108.8	C13—C14—H14A	109.5
C3—C4—C5	114.91 (16)	C15—C14—H14B	109.5
C3—C4—H4A	108.5	C13—C14—H14B	109.5
C5—C4—H4A	108.5	H14A—C14—H14B	108.1
C3—C4—H4B	108.5	C14—C15—C16	111.85 (18)
C5—C4—H4B	108.5	C14—C15—H15A	109.2
H4A—C4—H4B	107.5	C16—C15—H15A	109.2
N1ⁱ—C5—C4	115.19 (16)	C14—C15—H15B	109.2
N1ⁱ—C5—H5A	108.5	C16—C15—H15B	109.2
C4—C5—H5A	108.5	H15A—C15—H15B	107.9
N1ⁱ—C5—H5B	108.5	C15—C16—C2	112.86 (18)
C4—C5—H5B	108.5	C15—C16—H16A	109.0
H5A—C5—H5B	107.5	C2—C16—H16A	109.0
N1—C6—C7	111.16 (16)	C15—C16—H16B	109.0
N1—C6—H6A	109.4	C2—C16—H16B	109.0
C7—C6—H6A	109.4	H16A—C16—H16B	107.8

N1—C6—H6B	109.4	C3—C17—H17A	109.5
C7—C6—H6B	109.4	C3—C17—H17B	109.5
H6A—C6—H6B	108.0	H17A—C17—H17B	109.5
C8—C7—C12	118.6 (2)	C3—C17—H17C	109.5
C8—C7—C6	119.49 (19)	H17A—C17—H17C	109.5
C12—C7—C6	121.91 (19)	H17B—C17—H17C	109.5
C5ⁱ—N1—C1—C13	66.6 (2)	N1—C6—C7—C8	69.6 (2)
C6—N1—C1—C13	−61.0 (2)	N1—C6—C7—C12	−109.4 (2)
C5ⁱ—N1—C1—C2	−167.55 (16)	C12—C7—C8—C9	−0.5 (3)
C6—N1—C1—C2	64.9 (2)	C6—C7—C8—C9	−179.55 (19)
C3—N2—C2—C1	176.19 (16)	C7—C8—C9—C10	0.3 (3)
C3—N2—C2—C16	53.0 (2)	C8—C9—C10—C11	0.3 (3)
N1—C1—C2—N2	43.0 (2)	C9—C10—C11—C12	−0.6 (3)
C13—C1—C2—N2	172.36 (16)	C10—C11—C12—C7	0.4 (3)
N1—C1—C2—C16	169.96 (16)	C8—C7—C12—C11	0.1 (3)
C13—C1—C2—C16	−60.7 (2)	C6—C7—C12—C11	179.18 (19)
C2—N2—C3—C17	61.3 (2)	N1—C1—C13—C14	−169.24 (17)
C2—N2—C3—C4	−176.72 (16)	C2—C1—C13—C14	62.7 (2)
N2—C3—C4—C5	69.9 (2)	C1—C13—C14—C15	−57.7 (2)
C17—C3—C4—C5	−168.49 (17)	C13—C14—C15—C16	52.0 (3)
C3—C4—C5—N1ⁱ	−71.9 (2)	C14—C15—C16—C2	−52.8 (2)
C5ⁱ—N1—C6—C7	77.4 (2)	N2—C2—C16—C15	−178.66 (17)
C1—N1—C6—C7	−153.27 (17)	C1—C2—C16—C15	56.8 (2)

Symmetry code: (i) −x+1, −y+1, −z+1.

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2N···N1ⁱ	0.86 (2)	2.32 (2)	3.025 (2)	139.9 (19)

Symmetry code: (i) −x+1, −y+1, −z+1.