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**(11*R*,12*S*)-16-Aminotetracyclo-
[6.6.2.0^{2,7}.0^{9,14}]hexadeca-
2(7),3,5,9(14),10,12-hexaen-15-ol**

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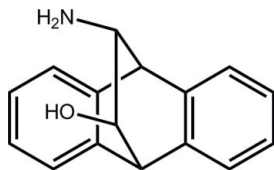
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.029; wR factor = 0.077; data-to-parameter ratio = 13.6.

In the title compound, $\text{C}_{16}\text{H}_{15}\text{NO}$, the dihedral angle between the outer benzene rings is $51.88(6)^\circ$, and each of the central six-membered rings has a boat conformation. The hydroxy and amino groups are *syn*, and the hydroxy H atom forms an intramolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bond. In the crystal, molecules assemble *via* $\text{C}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\pi$ interactions, consolidating a three-dimensional architecture.

Related literature

For chiral ligands in asymmetric catalytic reactions, see: Yamakuchi *et al.* (2005). For the synthesis of the title compound, see: Hashimoto *et al.* (1998); Matsunaga *et al.* (2005). For a related structure, see: Abdel-Aziz *et al.* (2012).



Experimental

Crystal data

$\text{C}_{16}\text{H}_{15}\text{NO}$	$V = 588.74(2) \text{ \AA}^3$
$M_r = 237.29$	$Z = 2$
Monoclinic, $P2_1$	Cu $K\alpha$ radiation
$a = 8.6224(2) \text{ \AA}$	$\mu = 0.65 \text{ mm}^{-1}$
$b = 7.1140(1) \text{ \AA}$	$T = 100 \text{ K}$
$c = 10.0210(2) \text{ \AA}$	$0.40 \times 0.30 \times 0.20 \text{ mm}$
$\beta = 106.707(2)^\circ$	

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Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector	4044 measured reflections
Absorption correction: multi-scan (<i>CrysAlis PRO</i> ; Agilent, 2012)	2375 independent reflections
$T_{\min} = 0.590$, $T_{\max} = 1.000$	2357 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.011$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.029$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.077$	$\Delta\rho_{\text{max}} = 0.20 \text{ e \AA}^{-3}$
$S = 1.06$	$\Delta\rho_{\text{min}} = -0.17 \text{ e \AA}^{-3}$
2375 reflections	Absolute structure: Flack (1983), 1060 Friedel pairs
175 parameters	Flack parameter: 0.0 (2)
1 restraint	

Table 1

Hydrogen-bond geometry (Å, °).

Cg1 and Cg2 are the centroids of the $\text{C1}-\text{C6}$ and $\text{C11}-\text{C16}$ benzene rings, respectively.

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1}o\cdots\text{N1}$	0.96 (3)	1.82 (2)	2.577 (2)	133 (2)
$\text{C5}-\text{H5}\cdots\text{O1}^i$	0.95	2.56	3.3506 (16)	141
$\text{C4}-\text{H4}\cdots\text{Cg1}^{ii}$	0.95	2.61	3.5064 (14)	158
$\text{C10}-\text{H10}\cdots\text{Cg2}^{iii}$	1.00	2.95	3.9212 (14)	164
$\text{C12}-\text{H12}\cdots\text{Cg1}^{iii}$	0.95	2.67	3.5159 (14)	149

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

Data collection: *CrysAlis PRO* (Agilent, 2012); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2558).

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supporting information

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(11*R*,12*S*)-16-Aminotetracyclo[6.6.2.0^{2,7}.0^{9,14}]hexadeca-2(7),3,5,9(14),10,12-hexaen-15-ol

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S1. Comment

The title compound was synthesized in relation to the development of chiral ligands for asymmetric catalytic reactions (Yamakuchi *et al.*, 2005) and in continuation of related structural studies (Abdel-Aziz *et al.*, 2012).

In the title molecule (Fig. 1), the dihedral angle between the (C1–C6) and (C11–C16) benzene rings is 51.88 (6)°. The dihedral angles between these planes and the central C7–C10 residue are 66.96 (5) and 61.17 (5)°, respectively. Each of the central six-membered rings (C1,C6–C10) and (C7–C10,C15,C15) has a boat conformation. The hydroxy and amino groups are *syn*, and the hydroxy-H atom is aligned to form an intramolecular O—H···N hydrogen bond (Table 1).

In the crystal packing, molecules assemble into a three-dimensional architecture *via* C—H···O and C—H··· π interactions (Fig. 2 and Table 1).

S2. Experimental

The title compound was prepared following literature precedents (Hashimoto *et al.*, 1998; Matsunaga *et al.*, 2005). To 10,11,14,15-tetrahydro-9,10-[4,5]epoxazoloanthracen-13(9*H*)-one (2.0 ml), water (2 ml), ethanol (6 ml) and Ba(OH)₂·8H₂O (20 ml) were added. The mixture was heated at 413 K in a glass sealed tube for 72 h. The resulting solution was evaporated and extracted three times with chloroform (10 ml). The organic extract was dried and recrystallized from ethanol to afford the title compound.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions [C—H 0.95 to 1.00 Å, $U_{\text{iso}}(\text{H}) 1.2U_{\text{eq}}(\text{C})$] and were included in the refinement in the riding model approximation. The hydroxy- and amino-H atoms were located in a difference Fourier map and were refined freely.

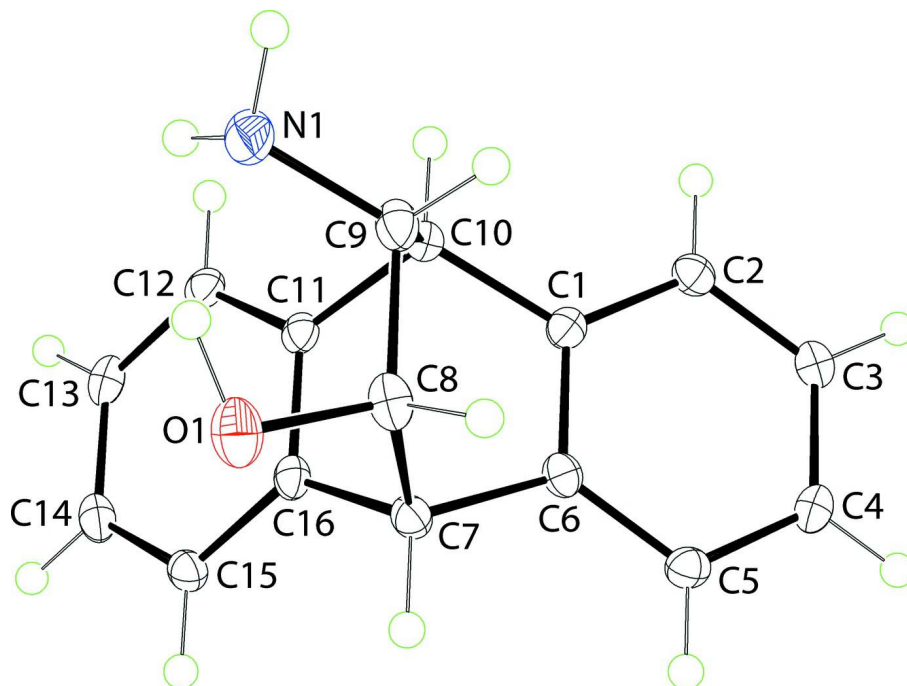


Figure 1

The molecular structure of the title compound showing the atom-labelling scheme and displacement ellipsoids at the 50% probability level.

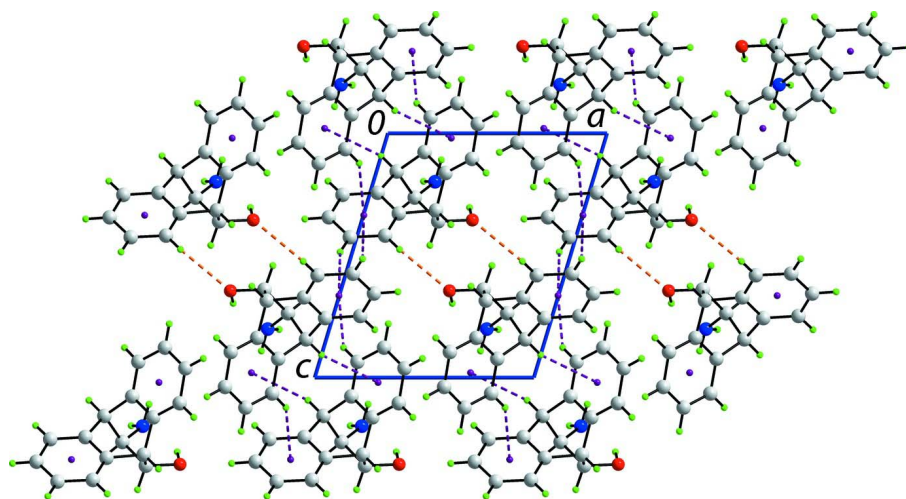


Figure 2

A view in projection down the *b* axis of the unit-cell contents for the title compound. The C—H...O and C—H... π interactions are shown as orange and purple dashed lines, respectively.

(11*R*,12*S*)-16-Aminotetracyclo[6.6.2.0^{2,7}.0^{9,14}]hexadeca- 2(7),3,5,9(14),10,12-hexaen-15-ol

Crystal data

C₁₆H₁₅NO

M_r = 237.29

Monoclinic, *P*2₁

Hall symbol: *P* 2₁*b*

a = 8.6224 (2) Å

b = 7.1140 (1) Å

$c = 10.0210 (2) \text{ \AA}$
 $\beta = 106.707 (2)^\circ$
 $V = 588.74 (2) \text{ \AA}^3$
 $Z = 2$
 $F(000) = 252$
 $D_x = 1.339 \text{ Mg m}^{-3}$
 Cu $K\alpha$ radiation, $\lambda = 1.54184 \text{ \AA}$

Cell parameters from 3312 reflections
 $\theta = 4.6\text{--}76.4^\circ$
 $\mu = 0.65 \text{ mm}^{-1}$
 $T = 100 \text{ K}$
 Prism, colourless
 $0.40 \times 0.30 \times 0.20 \text{ mm}$

Data collection

Agilent SuperNova Dual
 diffractometer with an Atlas detector
 Radiation source: SuperNova (Cu) X-ray
 Source
 Mirror monochromator
 Detector resolution: $10.4041 \text{ pixels mm}^{-1}$
 ω scan
 Absorption correction: multi-scan
 (CrysAlis PRO; Agilent, 2012)

$T_{\min} = 0.590, T_{\max} = 1.000$
 4044 measured reflections
 2375 independent reflections
 2357 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.011$
 $\theta_{\max} = 76.6^\circ, \theta_{\min} = 4.6^\circ$
 $h = -10 \rightarrow 10$
 $k = -8 \rightarrow 8$
 $l = -12 \rightarrow 7$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.077$
 $S = 1.06$
 2375 reflections
 175 parameters
 1 restraint
 Primary atom site location: structure-invariant
 direct methods
 Secondary atom site location: difference Fourier
 map

Hydrogen site location: inferred from
 neighbouring sites
 H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0469P)^2 + 0.1178P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.20 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.17 \text{ e \AA}^{-3}$
 Absolute structure: Flack (1983), 1060 Friedel
 pairs
 Absolute structure parameter: $0.0 (2)$

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.49638 (11)	0.50034 (15)	0.64491 (10)	0.0241 (2)
N1	0.71887 (14)	0.29044 (18)	0.80145 (14)	0.0240 (3)
C1	0.96576 (14)	0.71372 (17)	0.75647 (12)	0.0142 (2)
C2	1.12329 (14)	0.73301 (18)	0.74782 (12)	0.0158 (2)
H2	1.2081	0.6575	0.8038	0.019*
C3	1.15501 (14)	0.86460 (19)	0.65592 (12)	0.0160 (2)
H3	1.2620	0.8777	0.6488	0.019*
C4	1.03187 (15)	0.97688 (19)	0.57454 (12)	0.0168 (2)
H4	1.0557	1.0684	0.5142	0.020*
C5	0.87298 (15)	0.95569 (18)	0.58111 (12)	0.0160 (2)
H5	0.7882	1.0306	0.5244	0.019*
C6	0.84091 (15)	0.82368 (17)	0.67170 (12)	0.0144 (3)
C7	0.67531 (14)	0.77274 (18)	0.68480 (12)	0.0151 (2)
H7	0.5883	0.8555	0.6263	0.018*
C8	0.65031 (15)	0.56368 (19)	0.63948 (13)	0.0180 (3)
H8	0.6555	0.5528	0.5414	0.022*
C9	0.78995 (15)	0.43912 (18)	0.73668 (13)	0.0181 (3)

H9	0.8514	0.3787	0.6773	0.022*
C10	0.90784 (14)	0.57079 (17)	0.84339 (13)	0.0150 (3)
H10	0.9998	0.4997	0.9070	0.018*
C11	0.80732 (14)	0.67230 (17)	0.92286 (12)	0.0141 (2)
C12	0.82683 (14)	0.66176 (18)	1.06492 (13)	0.0161 (2)
H12	0.9093	0.5848	1.1225	0.019*
C13	0.72441 (15)	0.76507 (18)	1.12256 (12)	0.0181 (3)
H13	0.7377	0.7591	1.2199	0.022*
C14	0.60311 (15)	0.87664 (18)	1.03815 (13)	0.0178 (3)
H14	0.5346	0.9479	1.0782	0.021*
C15	0.58155 (14)	0.88446 (17)	0.89508 (13)	0.0159 (2)
H15	0.4978	0.9597	0.8374	0.019*
C16	0.68295 (14)	0.78190 (18)	0.83726 (12)	0.0145 (2)
H1o	0.526 (3)	0.384 (4)	0.696 (2)	0.048 (6)*
H1n	0.720 (3)	0.316 (4)	0.891 (3)	0.060 (7)*
H2n	0.772 (3)	0.179 (4)	0.803 (2)	0.043 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0167 (4)	0.0288 (6)	0.0270 (5)	-0.0084 (4)	0.0067 (4)	-0.0030 (4)
N1	0.0266 (6)	0.0152 (5)	0.0352 (6)	-0.0026 (5)	0.0170 (5)	-0.0005 (5)
C1	0.0170 (5)	0.0130 (6)	0.0131 (5)	-0.0007 (5)	0.0051 (4)	-0.0011 (4)
C2	0.0156 (5)	0.0163 (6)	0.0153 (5)	0.0009 (4)	0.0043 (4)	-0.0019 (4)
C3	0.0143 (5)	0.0184 (6)	0.0161 (5)	-0.0018 (5)	0.0059 (4)	-0.0029 (5)
C4	0.0200 (6)	0.0178 (6)	0.0141 (5)	-0.0025 (5)	0.0072 (4)	0.0004 (5)
C5	0.0178 (6)	0.0161 (6)	0.0135 (5)	0.0023 (5)	0.0036 (4)	0.0000 (4)
C6	0.0143 (5)	0.0156 (6)	0.0141 (5)	-0.0006 (4)	0.0051 (4)	-0.0023 (4)
C7	0.0127 (5)	0.0183 (6)	0.0146 (5)	0.0013 (5)	0.0042 (4)	0.0007 (5)
C8	0.0167 (6)	0.0211 (7)	0.0179 (6)	-0.0030 (5)	0.0075 (5)	-0.0039 (5)
C9	0.0201 (6)	0.0146 (6)	0.0229 (6)	-0.0029 (5)	0.0114 (5)	-0.0029 (5)
C10	0.0148 (5)	0.0141 (6)	0.0171 (6)	0.0006 (4)	0.0064 (4)	0.0017 (4)
C11	0.0149 (5)	0.0120 (6)	0.0162 (6)	-0.0023 (4)	0.0059 (4)	0.0001 (4)
C12	0.0168 (5)	0.0137 (6)	0.0177 (6)	-0.0019 (5)	0.0051 (4)	0.0020 (4)
C13	0.0222 (6)	0.0191 (6)	0.0152 (5)	-0.0057 (5)	0.0088 (5)	-0.0011 (5)
C14	0.0181 (6)	0.0158 (6)	0.0230 (6)	-0.0034 (5)	0.0113 (5)	-0.0040 (5)
C15	0.0132 (5)	0.0146 (6)	0.0206 (6)	-0.0005 (5)	0.0058 (4)	0.0002 (5)
C16	0.0142 (5)	0.0144 (6)	0.0164 (5)	-0.0027 (5)	0.0065 (4)	-0.0003 (5)

Geometric parameters (Å, °)

O1—C8	1.4174 (15)	C7—C8	1.5518 (17)
O1—H1o	0.96 (3)	C7—H7	1.0000
N1—C9	1.4644 (17)	C8—C9	1.5841 (18)
N1—H1n	0.91 (3)	C8—H8	1.0000
N1—H2n	0.91 (3)	C9—C10	1.5586 (17)
C1—C2	1.3924 (16)	C9—H9	1.0000
C1—C6	1.4024 (16)	C10—C11	1.5177 (16)

C1—C10	1.5140 (16)	C10—H10	1.0000
C2—C3	1.3944 (18)	C11—C12	1.3867 (17)
C2—H2	0.9500	C11—C16	1.4017 (16)
C3—C4	1.3902 (17)	C12—C13	1.3954 (18)
C3—H3	0.9500	C12—H12	0.9500
C4—C5	1.3985 (17)	C13—C14	1.3895 (18)
C4—H4	0.9500	C13—H13	0.9500
C5—C6	1.3881 (17)	C14—C15	1.3928 (18)
C5—H5	0.9500	C14—H14	0.9500
C6—C7	1.5150 (16)	C15—C16	1.3869 (17)
C7—C16	1.5116 (15)	C15—H15	0.9500
C8—O1—H1o	100.4 (13)	C7—C8—H8	108.7
C9—N1—H1n	113.9 (17)	C9—C8—H8	108.7
C9—N1—H2n	110.8 (14)	N1—C9—C10	113.79 (11)
H1n—N1—H2n	107 (2)	N1—C9—C8	109.61 (10)
C2—C1—C6	120.00 (11)	C10—C9—C8	108.43 (10)
C2—C1—C10	126.23 (11)	N1—C9—H9	108.3
C6—C1—C10	113.60 (10)	C10—C9—H9	108.3
C3—C2—C1	119.17 (11)	C8—C9—H9	108.3
C3—C2—H2	120.4	C1—C10—C11	108.35 (10)
C1—C2—H2	120.4	C1—C10—C9	105.47 (10)
C2—C3—C4	120.76 (11)	C11—C10—C9	106.73 (10)
C2—C3—H3	119.6	C1—C10—H10	112.0
C4—C3—H3	119.6	C11—C10—H10	112.0
C3—C4—C5	120.25 (11)	C9—C10—H10	112.0
C3—C4—H4	119.9	C12—C11—C16	120.26 (11)
C5—C4—H4	119.9	C12—C11—C10	126.43 (11)
C6—C5—C4	119.06 (11)	C16—C11—C10	113.29 (10)
C6—C5—H5	120.5	C11—C12—C13	119.47 (11)
C4—C5—H5	120.5	C11—C12—H12	120.3
C5—C6—C1	120.73 (11)	C13—C12—H12	120.3
C5—C6—C7	126.05 (11)	C14—C13—C12	120.27 (11)
C1—C6—C7	113.11 (10)	C14—C13—H13	119.9
C16—C7—C6	108.00 (9)	C12—C13—H13	119.9
C16—C7—C8	107.41 (10)	C13—C14—C15	120.25 (11)
C6—C7—C8	105.01 (10)	C13—C14—H14	119.9
C16—C7—H7	112.0	C15—C14—H14	119.9
C6—C7—H7	112.0	C16—C15—C14	119.70 (11)
C8—C7—H7	112.0	C16—C15—H15	120.2
O1—C8—C7	110.26 (10)	C14—C15—H15	120.2
O1—C8—C9	110.68 (11)	C15—C16—C11	120.02 (11)
C7—C8—C9	109.82 (10)	C15—C16—C7	126.54 (11)
O1—C8—H8	108.7	C11—C16—C7	113.44 (10)
C6—C1—C2—C3	1.06 (17)	C2—C1—C10—C9	-113.84 (13)
C10—C1—C2—C3	176.01 (12)	C6—C1—C10—C9	61.39 (12)
C1—C2—C3—C4	0.59 (18)	N1—C9—C10—C1	-179.42 (10)

C2—C3—C4—C5	-1.72 (19)	C8—C9—C10—C1	-57.17 (11)
C3—C4—C5—C6	1.17 (18)	N1—C9—C10—C11	-64.31 (13)
C4—C5—C6—C1	0.48 (17)	C8—C9—C10—C11	57.94 (12)
C4—C5—C6—C7	-175.42 (11)	C1—C10—C11—C12	-128.68 (13)
C2—C1—C6—C5	-1.61 (17)	C9—C10—C11—C12	118.16 (13)
C10—C1—C6—C5	-177.17 (11)	C1—C10—C11—C16	52.62 (13)
C2—C1—C6—C7	174.79 (11)	C9—C10—C11—C16	-60.54 (13)
C10—C1—C6—C7	-0.77 (14)	C16—C11—C12—C13	-1.70 (18)
C5—C6—C7—C16	-129.68 (13)	C10—C11—C12—C13	179.68 (12)
C1—C6—C7—C16	54.15 (13)	C11—C12—C13—C14	0.43 (18)
C5—C6—C7—C8	115.94 (13)	C12—C13—C14—C15	0.78 (18)
C1—C6—C7—C8	-60.23 (12)	C13—C14—C15—C16	-0.72 (18)
C16—C7—C8—O1	66.32 (12)	C14—C15—C16—C11	-0.55 (17)
C6—C7—C8—O1	-178.88 (10)	C14—C15—C16—C7	179.28 (12)
C16—C7—C8—C9	-55.90 (12)	C12—C11—C16—C15	1.77 (17)
C6—C7—C8—C9	58.89 (12)	C10—C11—C16—C15	-179.44 (11)
O1—C8—C9—N1	1.54 (14)	C12—C11—C16—C7	-178.08 (11)
C7—C8—C9—N1	123.51 (11)	C10—C11—C16—C7	0.71 (14)
O1—C8—C9—C10	-123.22 (11)	C6—C7—C16—C15	126.02 (13)
C7—C8—C9—C10	-1.25 (13)	C8—C7—C16—C15	-121.19 (13)
C2—C1—C10—C11	132.18 (12)	C6—C7—C16—C11	-54.14 (14)
C6—C1—C10—C11	-52.59 (13)	C8—C7—C16—C11	58.65 (12)

Hydrogen-bond geometry (\AA , $^\circ$)

Cg1 and Cg2 are the centroids of the C1—C6 and C11—C16 benzene rings, respectively.

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O1—H1 \cdots N1	0.96 (3)	1.82 (2)	2.577 (2)	133 (2)
C5—H5 \cdots O1 ⁱ	0.95	2.56	3.3506 (16)	141
C4—H4 \cdots Cg1 ⁱⁱ	0.95	2.61	3.5064 (14)	158
C10—H10 \cdots Cg2 ⁱⁱⁱ	1.00	2.95	3.9212 (14)	164
C12—H12 \cdots Cg1 ⁱⁱⁱ	0.95	2.67	3.5159 (14)	149

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