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2-(Tricyclo[3.3.1.1^{3,7}]dec-2-ylamino)-ethanol hemihydrate

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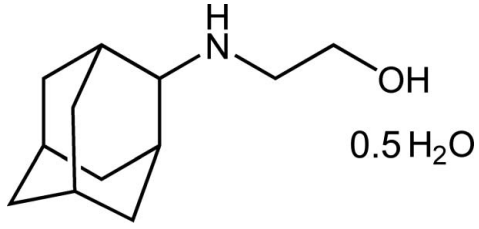
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.043; wR factor = 0.112; data-to-parameter ratio = 18.3.

The title adamantane derivative, $C_{12}H_{21}NO\cdot0.5H_2O$, was synthesized as part of an investigation into the biological activities of cage amino–alcohol compounds as potential anti-tuberculosis agents. The structure displays intermolecular O—H···N, N—H···O, O—H···O hydrogen bonding and a layered packing structure with distinct hydrophilic and hydrophobic regions. The water molecule lies on a twofold rotation axis.

Related literature

For related literature, see: Bogatcheva *et al.* (2006); du Pont de Nemours and Co. (1969); Lee *et al.* (2003); Tripathi *et al.* (2006).



Experimental

Crystal data

$C_{12}H_{21}NO\cdot0.5H_2O$
M_r = 204.31
Monoclinic, $C2/c$
a = 11.6739 (3) Å
b = 6.5043 (2) Å
c = 28.6241 (7) Å
β = 99.8620 (10)°
V = 2141.33 (10) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 173 (2) K
0.56 × 0.43 × 0.18 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: none
13147 measured reflections
2584 independent reflections
2352 reflections with $I > 2\sigma(I)$
R_{int} = 0.058

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.112
S = 1.07
2584 reflections
141 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1C···N1^{i}	0.84	1.86	2.7007 (12)	175
N1—H1B···O1W^{ii}	0.849 (16)	2.398 (16)	3.2241 (12)	164.6 (14)
O1W—H1W···O1^{iii}	0.863 (16)	1.963 (17)	2.8147 (12)	168.7 (16)

Symmetry codes: (i) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$; (ii) $x, y-1, z$; (iii) $-x+2, y, -z+\frac{3}{2}$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT-Plus* (Bruker, 1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *Mercury* (Macrae *et al.*, 2006) and *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

We thank Dr Manuel Fernandes of the Jan Boeyens Structural Chemistry Laboratory at the University of the Witwatersrand for his assistance in the acquisition of the crystallographic data. This work was supported by grants from the National Research Foundation (South Africa), GUN 2046819, the University of KwaZulu–Natal and Aspen Pharmacare.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2403).

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supporting information

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2-(Tricyclo[3.3.1.1^{3,7}]dec-2-ylamino)ethanol hemihydrate

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S1. Comment

The title compound, an adamantane derivative, was synthesized as part of an ongoing study to evaluate the biological activity of such compounds as potential anti-tuberculosis agents (Bogatcheva *et al.* (2006), Lee *et al.* (2003), Tripathi *et al.* (2006)). Although the compound is known (du Pont de Nemours and Co.; 1969), its crystal structure has not been reported.

The compound contains a polycyclic (lipophilic) hydrocarbon region, polar amine and hydroxyl units, and crystallizes with half a water molecule in the asymmetric unit (Fig.1)- the water molecule being situated on a crystallographic 2-fold axis at (1, *y*, 3/4). The title molecule exhibits several C–C bond lengths in the adamantane skeleton that deviate from the expected value of 1.54 Å. This has been observed previously and is typical for these types of compounds.

The structure exhibits intermolecular hydrogen bonding between O1 and N1 of adjacent molecules as well as between O1 and O1W of the water molecule (Fig. 2). There is also a complex network of short contacts between the molecules in structure. These intermolecular interactions result in a layered structure with distinct hydrophilic and hydrophobic regions (Fig. 3). The adamantane skeleton forms the hydrophobic layer while the polar hydroxyl and amino moeties constitute the hydrophilic region.

S2. Experimental

A mixture of 2-adamantanone (2 g, 13 mmol) and 2-aminoethanol (1 g, 16 mmol) in 20 ml of methanol was stirred under dinitrogen atmosphere at room temperature for 2 h. The mixture was cooled to zero degrees using an external ice bath after with $NaBH_4$ (1 g, 26 mmol) was added slowly over a 30 minutes. The mixture was stirred for overnight at room temperature after which it was concentrated *in vacuo* and excess $NaBH_4$ was quenched by adding 40 ml of 10% HCl and the product was also extracted as its HCl salt in the process. The aqueous solution was washed with 2x20ml of dichloromethane, after which the aqueous layer was basified (pH 12) with NH_4OH and the product was extracted from the mixture with dichloromethane (2x30ml), the solvent was dried over Na_2SO_4 and concentrated *in vacuo*. The product was recrystallized from dichloromethane, thereby affording pure 2-aminoethanol adamantane (2 g, 77% yield).

S3. Refinement

Non-hydrogen atoms were first refined isotropically followed by anisotropic refinement by full matrix least-squares calculations based on F^2 using *SHELXTL*. With the exception to H1B and H1W, all hydrogen atoms were first located in the difference map then positioned geometrically, and allowed to ride on their respective parent atoms, with bond lengths of 0.99 Å (CH_2), 1.00 Å (Methine CH) or 0.84 Å (OH). Isotropic displacement parameters for these atoms were set equal to 1.2 (CH_2 and CH), or 1.5 OH) times U_{eq} of the parent atom. Atoms H1B and H1W were located in the difference map and refined freely.

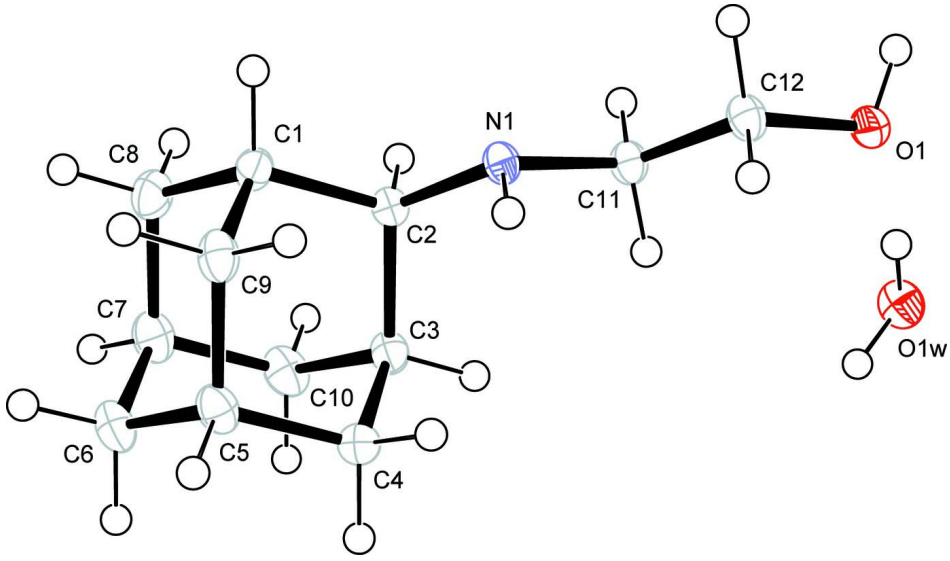

Figure 1

The *ORTEP* (Farrugia, 1997) diagram of the title compound showing the displacement ellipsoids for non-hydrogen atoms at the 50% probability level.

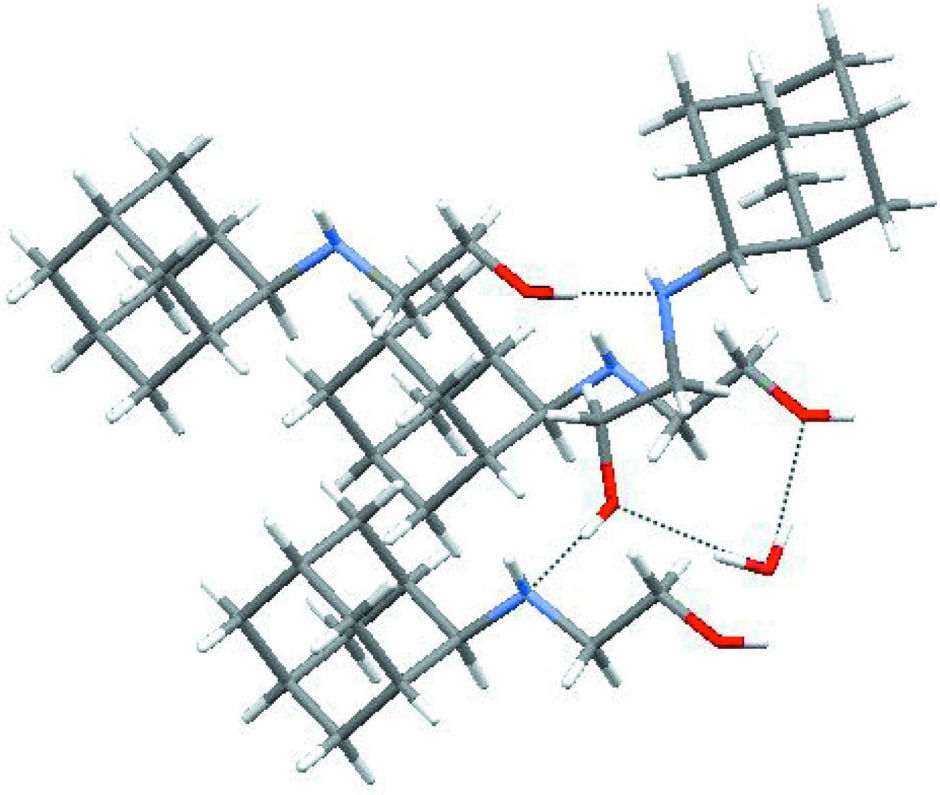

Figure 2

Figure depicting the intermolecular hydrogen bonding.

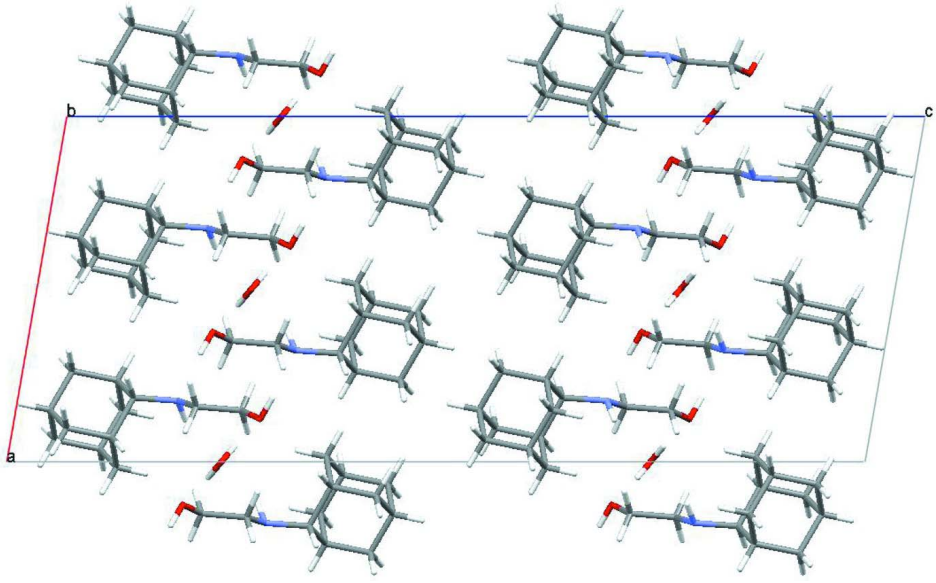

Figure 3

Packing diagram depicting layered structure as seen down the *b*-axis.

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Crystal data

$C_{12}H_{21}NO{\cdot}0.5H_2O$
$M_r = 204.31$
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
$a = 11.6739\ (3)$ Å
$b = 6.5043\ (2)$ Å
$c = 28.6241\ (7)$ Å
$\beta = 99.862\ (1)°$
$V = 2141.33\ (10)$ Å^3
$Z = 8$
$F(000) = 904$
$D_x = 1.267$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7587 reflections
$\theta = 2.9–28.3°$
$\mu = 0.08$ mm^{-1}
$T = 173$ K
Plate, colourless
0.56 × 0.43 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
13147 measured reflections
2584 independent reflections
2352 reflections with $I > 2\sigma(I)$
$R_{int} = 0.058$
$\theta_{max} = 28.0°$, $\theta_{min} = 1.4°$
$h = -15\rightarrow15$
$k = -8\rightarrow8$
$l = -37\rightarrow37$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.112$
$S = 1.07$
2584 reflections
141 parameters
0 restraints
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

$w = 1/[\sigma^2(F_o^2) + (0.0507P)^2 + 1.5961P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.39$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.76341 (9)	−0.06132 (17)	0.61221 (4)	0.0189 (2)
H1	0.6899	−0.0946	0.6241	0.023*
C2	0.81420 (9)	0.13986 (16)	0.63482 (3)	0.0156 (2)
H2	0.7582	0.2520	0.6229	0.019*
C3	0.92859 (10)	0.18681 (17)	0.61700 (4)	0.0181 (2)
H3	0.9635	0.3162	0.6321	0.022*
C4	1.01487 (10)	0.00885 (18)	0.62888 (4)	0.0205 (2)
H4A	1.0880	0.0411	0.6173	0.025*
H4B	1.0334	−0.0096	0.6637	0.025*
C5	0.96251 (10)	−0.18982 (17)	0.60569 (4)	0.0208 (2)
H5	1.0189	−0.3054	0.6137	0.025*
C6	0.93562 (11)	−0.16124 (19)	0.55178 (4)	0.0243 (3)
H6A	1.0082	−0.1301	0.5396	0.029*
H6B	0.9023	−0.2896	0.5366	0.029*
C7	0.84905 (11)	0.01531 (19)	0.53962 (4)	0.0240 (3)
H7	0.8313	0.0338	0.5044	0.029*
C8	0.73704 (10)	−0.0346 (2)	0.55821 (4)	0.0250 (3)
H8A	0.7025	−0.1627	0.5433	0.030*
H8B	0.6802	0.0781	0.5499	0.030*
C9	0.85024 (10)	−0.23740 (17)	0.62443 (4)	0.0208 (2)
H9A	0.8159	−0.3668	0.6101	0.025*
H9B	0.8677	−0.2561	0.6593	0.025*
C10	0.90159 (11)	0.21388 (18)	0.56282 (4)	0.0241 (3)
H10A	0.8462	0.3288	0.5546	0.029*
H10B	0.9739	0.2475	0.5507	0.029*
C11	0.83975 (9)	0.33596 (16)	0.70953 (4)	0.0170 (2)
H11A	0.7728	0.4258	0.6973	0.020*
H11B	0.9109	0.4007	0.7018	0.020*
C12	0.85067 (10)	0.31373 (16)	0.76292 (4)	0.0186 (2)
H12A	0.7770	0.2587	0.7706	0.022*
H12B	0.9132	0.2141	0.7745	0.022*
N1	0.82297 (8)	0.13298 (14)	0.68680 (3)	0.0156 (2)

O1	0.87618 (7)	0.50454 (12)	0.78653 (3)	0.01947 (19)
H1C	0.8167	0.5477	0.7963	0.029*
O1W	1.0000	0.82045 (19)	0.7500	0.0240 (3)
H1B	0.8774 (13)	0.053 (2)	0.6990 (5)	0.024 (4)*
H1W	1.0445 (14)	0.737 (3)	0.7380 (6)	0.037 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0192 (5)	0.0197 (5)	0.0178 (5)	−0.0041 (4)	0.0030 (4)	−0.0029 (4)
C2	0.0175 (5)	0.0146 (5)	0.0146 (5)	0.0007 (4)	0.0027 (4)	0.0003 (4)
C3	0.0224 (5)	0.0155 (5)	0.0172 (5)	−0.0032 (4)	0.0059 (4)	−0.0004 (4)
C4	0.0178 (5)	0.0236 (6)	0.0200 (5)	0.0000 (4)	0.0033 (4)	−0.0024 (4)
C5	0.0247 (6)	0.0180 (5)	0.0198 (5)	0.0045 (4)	0.0038 (4)	−0.0010 (4)
C6	0.0310 (6)	0.0236 (6)	0.0193 (5)	0.0008 (5)	0.0072 (4)	−0.0046 (4)
C7	0.0313 (6)	0.0265 (6)	0.0138 (5)	0.0015 (5)	0.0030 (4)	0.0010 (4)
C8	0.0245 (6)	0.0296 (6)	0.0188 (5)	−0.0003 (5)	−0.0023 (4)	−0.0036 (4)
C9	0.0300 (6)	0.0137 (5)	0.0189 (5)	−0.0029 (4)	0.0048 (4)	−0.0006 (4)
C10	0.0339 (6)	0.0206 (6)	0.0195 (5)	0.0004 (5)	0.0092 (4)	0.0040 (4)
C11	0.0207 (5)	0.0133 (5)	0.0170 (5)	0.0003 (4)	0.0033 (4)	−0.0009 (4)
C12	0.0239 (5)	0.0151 (5)	0.0172 (5)	−0.0006 (4)	0.0049 (4)	−0.0010 (4)
N1	0.0189 (4)	0.0131 (4)	0.0149 (4)	0.0008 (3)	0.0033 (3)	−0.0003 (3)
O1	0.0195 (4)	0.0188 (4)	0.0208 (4)	−0.0012 (3)	0.0054 (3)	−0.0058 (3)
O1W	0.0293 (6)	0.0175 (6)	0.0259 (6)	0.000	0.0064 (5)	0.000

Geometric parameters (Å, º)

C1—C9	1.5297 (16)	C7—C8	1.5287 (17)
C1—C8	1.5334 (15)	C7—C10	1.5316 (17)
C1—C2	1.5334 (14)	C7—H7	1.0000
C1—H1	1.0000	C8—H8A	0.9900
C2—N1	1.4745 (12)	C8—H8B	0.9900
C2—C3	1.5395 (15)	C9—H9A	0.9900
C2—H2	1.0000	C9—H9B	0.9900
C3—C4	1.5339 (15)	C10—H10A	0.9900
C3—C10	1.5388 (15)	C10—H10B	0.9900
C3—H3	1.0000	C11—N1	1.4700 (13)
C4—C5	1.5314 (16)	C11—C12	1.5187 (14)
C4—H4A	0.9900	C11—H11A	0.9900
C4—H4B	0.9900	C11—H11B	0.9900
C5—C9	1.5305 (16)	C12—O1	1.4200 (13)
C5—C6	1.5324 (15)	C12—H12A	0.9900
C5—H5	1.0000	C12—H12B	0.9900
C6—C7	1.5299 (17)	N1—H1B	0.849 (16)
C6—H6A	0.9900	O1—H1C	0.8400
C6—H6B	0.9900	O1W—H1W	0.863 (16)
C9—C1—C8	109.03 (9)	C6—C7—C10	109.54 (10)

C9—C1—C2	110.44 (9)	C8—C7—H7	109.5
C8—C1—C2	108.97 (9)	C6—C7—H7	109.5
C9—C1—H1	109.5	C10—C7—H7	109.5
C8—C1—H1	109.5	C7—C8—C1	109.82 (9)
C2—C1—H1	109.5	C7—C8—H8A	109.7
N1—C2—C1	110.79 (8)	C1—C8—H8A	109.7
N1—C2—C3	115.22 (8)	C7—C8—H8B	109.7
C1—C2—C3	108.91 (8)	C1—C8—H8B	109.7
N1—C2—H2	107.2	H8A—C8—H8B	108.2
C1—C2—H2	107.2	C1—C9—C5	110.01 (9)
C3—C2—H2	107.2	C1—C9—H9A	109.7
C4—C3—C10	108.86 (9)	C5—C9—H9A	109.7
C4—C3—C2	110.53 (9)	C1—C9—H9B	109.7
C10—C3—C2	108.48 (9)	C5—C9—H9B	109.7
C4—C3—H3	109.6	H9A—C9—H9B	108.2
C10—C3—H3	109.6	C7—C10—C3	109.77 (9)
C2—C3—H3	109.6	C7—C10—H10A	109.7
C5—C4—C3	110.03 (9)	C3—C10—H10A	109.7
C5—C4—H4A	109.7	C7—C10—H10B	109.7
C3—C4—H4A	109.7	C3—C10—H10B	109.7
C5—C4—H4B	109.7	H10A—C10—H10B	108.2
C3—C4—H4B	109.7	N1—C11—C12	109.98 (8)
H4A—C4—H4B	108.2	N1—C11—H11A	109.7
C9—C5—C4	108.72 (9)	C12—C11—H11A	109.7
C9—C5—C6	109.72 (9)	N1—C11—H11B	109.7
C4—C5—C6	109.38 (9)	C12—C11—H11B	109.7
C9—C5—H5	109.7	H11A—C11—H11B	108.2
C4—C5—H5	109.7	O1—C12—C11	111.73 (8)
C6—C5—H5	109.7	O1—C12—H12A	109.3
C7—C6—C5	109.49 (9)	C11—C12—H12A	109.3
C7—C6—H6A	109.8	O1—C12—H12B	109.3
C5—C6—H6A	109.8	C11—C12—H12B	109.3
C7—C6—H6B	109.8	H12A—C12—H12B	107.9
C5—C6—H6B	109.8	C11—N1—C2	113.60 (8)
H6A—C6—H6B	108.2	C11—N1—H1B	109.6 (10)
C8—C7—C6	109.42 (10)	C2—N1—H1B	110.6 (10)
C8—C7—C10	109.34 (10)	C12—O1—H1C	109.5

C9—C1—C2—N1	−69.54 (11)	C6—C7—C8—C1	60.43 (12)
C8—C1—C2—N1	170.72 (9)	C10—C7—C8—C1	−59.53 (12)
C9—C1—C2—C3	58.18 (11)	C9—C1—C8—C7	−60.00 (12)
C8—C1—C2—C3	−61.56 (11)	C2—C1—C8—C7	60.61 (12)
N1—C2—C3—C4	67.40 (11)	C8—C1—C9—C5	59.43 (11)
C1—C2—C3—C4	−57.78 (11)	C2—C1—C9—C5	−60.27 (11)
N1—C2—C3—C10	−173.32 (9)	C4—C5—C9—C1	60.16 (11)
C1—C2—C3—C10	61.50 (11)	C6—C5—C9—C1	−59.42 (11)
C10—C3—C4—C5	−59.70 (12)	C8—C7—C10—C3	59.75 (12)
C2—C3—C4—C5	59.36 (11)	C6—C7—C10—C3	−60.14 (12)

C3—C4—C5—C9	−59.71 (11)	C4—C3—C10—C7	59.61 (12)
C3—C4—C5—C6	60.09 (12)	C2—C3—C10—C7	−60.72 (12)
C9—C5—C6—C7	59.30 (12)	N1—C11—C12—O1	175.36 (9)
C4—C5—C6—C7	−59.87 (12)	C12—C11—N1—C2	−178.67 (8)
C5—C6—C7—C8	−59.80 (12)	C1—C2—N1—C11	−164.04 (9)
C5—C6—C7—C10	60.05 (12)	C3—C2—N1—C11	71.76 (11)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*C*···N1^{i}	0.84	1.86	2.7007 (12)	175
N1—H1*B*···O1*W*^{ii}	0.849 (16)	2.398 (16)	3.2241 (12)	164.6 (14)
O1*W*—H1*W*···O1^{iii}	0.863 (16)	1.963 (17)	2.8147 (12)	168.7 (16)

Symmetry codes: (i) −*x*+3/2, *y*+1/2, −*z*+3/2; (ii) *x*, *y*−1, *z*; (iii) −*x*+2, *y*, −*z*+3/2.