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(E)-3-Propoxymethylidene-2,3-dihydro-1H-pyrrolo[2,1-b]quinazolin-9-one monohydrate

Burkhon Zh Elmuradov,* Kambarali Turgunov, Bakhodir Tashkhodjaev and Khusniddin M. Shakhidoyatov

S. Yunusov Institute of the Chemistry of Plant Substances, Academy of Sciences of Uzbekistan, Mirzo Ulugbek Str. 77, Tashkent 100170, Uzbekistan

Correspondence e-mail: burkhon@rambler.ru

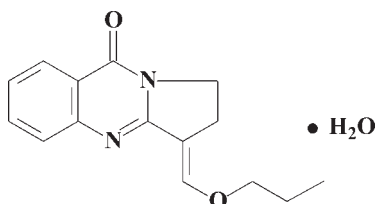
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.064; wR factor = 0.144; data-to-parameter ratio = 11.6.

The title compound, $\text{C}_{15}\text{H}_{16}\text{N}_2\text{O}_2 \cdot \text{H}_2\text{O}$, was synthesized *via* the alkylation of 3-hydroxymethylidene-2,3-dihydro-1H-pyrrolo[2,1-b]quinazolin-9-one with *n*-propyl iodide in the presence of sodium hydroxide. The organic molecule and the water molecule both lie on a crystallographic mirror plane. In the crystal structure, intermolecular $\text{O}-\text{H} \cdots \text{O}$ and $\text{O}-\text{H} \cdots \text{N}$ hydrogen bonds link the components into extended chains along [100].

Related literature

For the synthesis of the title compound and its derivatives, see: Späth & Platzer, (1935); Shakhidoyatov *et al.* (1976); Oripov *et al.* (1979); Elmuradov *et al.* (2006); Elmuradov & Shakhidoyatov (2004); Jahng *et al.* (2008). For the physiological activity of the title compound and its derivatives, see: Amin & Mehta (1959); Chatterjee & Ganguly (1968); Yakhontov *et al.* (1977); Yunusov *et al.* (1978); Johne (1981); Shakhidoyatov (1988). For standard bond distances, see: Allen *et al.* (1987).



Experimental

Crystal data

$\text{C}_{15}\text{H}_{16}\text{N}_2\text{O}_2 \cdot \text{H}_2\text{O}$
 $M_r = 274.31$
 Monoclinic, $P2_1/m$
 $a = 9.247$ (2) Å
 $b = 6.876$ (1) Å

$c = 10.950$ (2) Å
 $\beta = 97.90$ (3)°
 $V = 689.6$ (2) Å³
 $Z = 2$
 Mo $K\alpha$ radiation

$\mu = 0.09$ mm⁻¹
 $T = 293$ K

$0.75 \times 0.53 \times 0.20$ mm

Data collection

Stoe Stadi-4 four-circle diffractometer
 1483 measured reflections
 1479 independent reflections

1000 reflections with $I > 2\sigma(I)$
 3 standard reflections every 60 min
 intensity decay: 2%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.064$
 $wR(F^2) = 0.144$
 $S = 1.15$
 1479 reflections
 128 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.21$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.19$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{O1w}-\text{H1} \cdots \text{N1}$	0.85 (7)	2.14 (8)	2.968 (5)	165 (7)
$\text{O1w}-\text{H2} \cdots \text{O2}^i$	0.90 (7)	1.95 (7)	2.855 (5)	176 (6)

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

Data collection: *STADIA* (Stoe & Cie, 1997); cell refinement: *STADIA*; data reduction: *X-RED* (Stoe & Cie, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* (Bruker, 1998); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5028).

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supporting information

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(*E*)-3-Propoxymethylidene-2,3-dihydro-1*H*-pyrrolo[2,1-*b*]quinazolin-9-one monohydrate

Burkhon Zh Elmuradov, Kambarali Turgunov, Bakhodir Tashkhodjaev and Khusniddin M. Shakhidoyatov

S1. Comment

Tricyclic quinazoline alkaloids are a large group of heterocyclic compounds (Späth & Platzer, 1935; Shakhidoyatov *et al.*, 1976; Oripov *et al.*, 1979; Elmuradov & Shakhidoyatov, 2006; Jahng *et al.*, 2008). These compounds and their derivatives possess different pharmacological activities (Amin & Mehta 1959; Chatterjee & Ganguly, 1968; Yakhontov *et al.*, 1977; Yunusov *et al.*, 1978; Johne, 1981; Shakhidoyatov, 1988).

Alkylation of 3-hydroxymethylidene-2,3-dihydro-1*H*-pyrrolo[2,1-*b*]quinazolin-9-one with C1—C3 alkyl halides leads to the formation of new *C*-alkyl (Elmuradov & Shakhidoyatov, 2004) or *O*-alkyl derivatives (Elmuradov *et al.*, 2006). Using the typical synthesis for *O*-alkyl derivatives the reaction of 3-hydroxymethylidene-2,3-dihydro-1*H*-pyrrolo[2,1-*b*]quinazolin-9-one with *n*-propyl iodide was carried out by boiling of the initial reagents (1:2 ratio) over 7 hours in ethanol in the presence of sodium hydroxide (Elmuradov *et al.*, 2006) (Figure 1). The compound (3-hydroxymethylidene-2,3-dihydro-1*H*-pyrrolo[2,1-*b*]quinazolin-9-one) is obtained by formylation of 2,3-dihydro-1*H*-pyrrolo[2,1-*b*]quinazolin-9-one (alkaloid Deoxyvasicinone, isolated from *Peganum Harmala*) (Chatterjee & Ganguly, 1968) with Vilsmeier-Haack reagent.

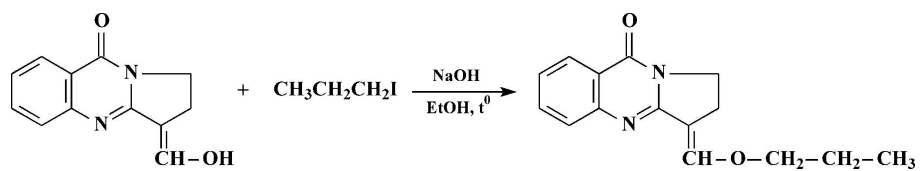
The asymmetric unit contains half molecule of 3-propoxymethylidene-2,3-dihydro-1*H*-pyrrolo[2,1-*b*]quinazolin-9-one and a half water molecule (Figure 2). Both molecules of the asymmetric unit lay on the crystallographic mirror plane. The water molecule links the N and Oⁱ atoms of title compound molecule by O—H[⋯]N and O—H[⋯]Oⁱ hydrogen bonds, which form a H-bond chain along [100] (Figure 3). The bond distances (Allen *et al.*, 1987) and angles in molecule are in normal ranges.

S2. Experimental

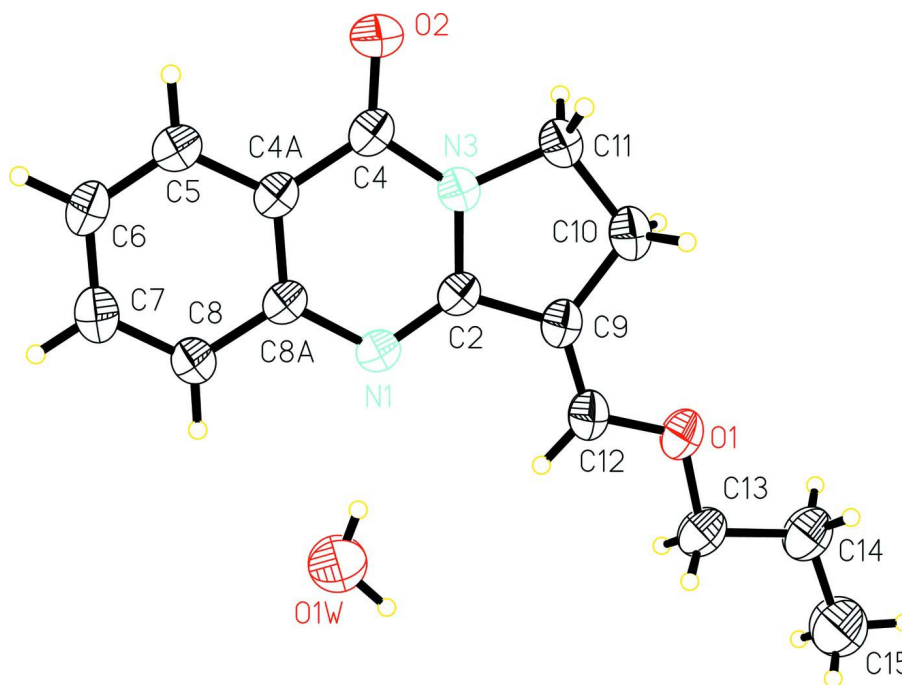
Sodium hydroxide (0.08 g, 2 mmol) was dissolved in ethanol (10 ml), and 3-hydroxymethylidene-2,3-dihydro-1*H*-pyrrolo[2,1-*b*]quinazolin-9-one (0.214 g, 1 mmol) and propyl iodide (0.34 g, 0.18 ml, *d*=1.747 g/ml, 2 mmol) were added. The mixture was heated to reflux on a water bath for 7 hours. The solvent was distilled off and the residue was recrystallized from hexane. The title compound was obtained in 70 % yield (0.18 g). Colorless crystals suitable for X-ray analysis were obtained from hexane by slow evaporation.

S3. Refinement

Carbon-bound H atoms were positioned geometrically and treated as riding on their C atoms, with C—H distances of 0.93 Å (aromatic) and 0.97 Å (CH₂) and 0.96 Å (CH₃) and were refined with U_{iso}(H) = 1.2U_{eq}(C)]. The H atoms of the water molecule involved in the intramolecular hydrogen bonds were located by difference Fourier synthesis and refined freely [O—H = 0.84 (7) and 0.90 (7) Å].

**Figure 1**

The reaction scheme.

**Figure 2**

The molecular structure of (I), with atom labels and 50% probability displacement ellipsoids for non-H atoms.

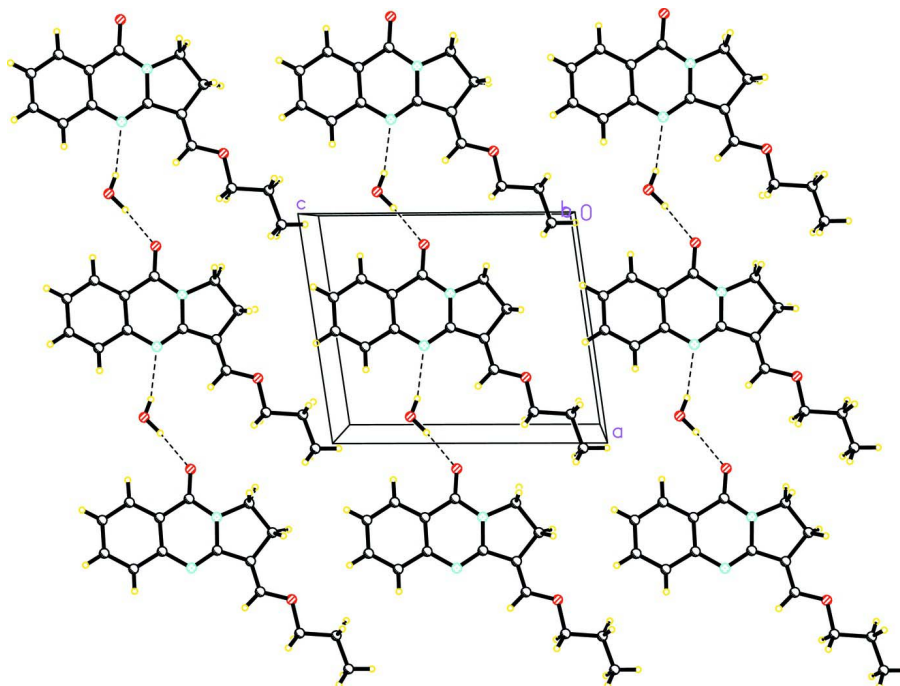


Figure 3

Part of the crystal structure of (I), showing the formation of hydrogen-bonded (dashed lines) chains along [100].

(E)-3-Propoxymethylidene-2,3-dihydro-1H-pyrrolo[2,1-b]quinazolin-9-one monohydrate

Crystal data

$C_{15}H_{16}N_2O_2 \cdot H_2O$

$M_r = 274.31$

Monoclinic, $P2_1/m$

Hall symbol: $-P\ 2_1y$

$a = 9.247\ (2)\ \text{\AA}$

$b = 6.876\ (1)\ \text{\AA}$

$c = 10.950\ (2)\ \text{\AA}$

$\beta = 97.90\ (3)^\circ$

$V = 689.6\ (2)\ \text{\AA}^3$

$Z = 2$

$F(000) = 292$

$D_x = 1.321\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 15 reflections

$\theta = 5\text{--}10^\circ$

$\mu = 0.09\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Prism, colourless

$0.75 \times 0.53 \times 0.20\ \text{mm}$

Data collection

Stoe Stadi-4 four-circle
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Scan width (ω) = 1.56–1.68, scan ratio $2\theta:\omega =$

1.00 I(Net) and $\sigma(I)$ calculated according to

Blessing (1987)

[Blessing, R. H. (1987). *Cryst. Rev.* **1**, 3–58]

1483 measured reflections

1479 independent reflections

1000 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.000$

$\theta_{\text{max}} = 26.0^\circ$, $\theta_{\text{min}} = 1.9^\circ$

$h = -11 \rightarrow 11$

$k = 0 \rightarrow 8$

$l = 0 \rightarrow 13$

3 standard reflections every 60 min

intensity decay: 2%

*Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.064$ $wR(F^2) = 0.144$ $S = 1.15$

1479 reflections

128 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sitesH atoms treated by a mixture of independent
and constrained refinement $w = 1/[\sigma^2(F_o^2) + (0.034P)^2 + 0.5511P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.21 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.19 \text{ e } \text{\AA}^{-3}$ Extinction correction: *SHELXL*, $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.009 (2)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
O1	0.7289 (3)	0.2500	0.2635 (2)	0.0522 (8)	
O2	0.1375 (3)	0.2500	0.5696 (3)	0.0682 (10)	
N1	0.5853 (3)	0.2500	0.6240 (3)	0.0390 (8)	
C2	0.5055 (4)	0.2500	0.5163 (3)	0.0349 (9)	
N3	0.3551 (3)	0.2500	0.4988 (3)	0.0370 (8)	
C4	0.2711 (4)	0.2500	0.5924 (3)	0.0430 (10)	
C4A	0.3567 (4)	0.2500	0.7153 (3)	0.0384 (9)	
C5	0.2880 (4)	0.2500	0.8207 (4)	0.0493 (11)	
H5A	0.1865	0.2500	0.8133	0.059*	
C6	0.3684 (4)	0.2500	0.9343 (4)	0.0546 (12)	
H6A	0.3215	0.2500	1.0043	0.066*	
C7	0.5192 (5)	0.2500	0.9468 (4)	0.0531 (11)	
H7A	0.5731	0.2500	1.0251	0.064*	
C8	0.5901 (4)	0.2500	0.8445 (3)	0.0448 (10)	
H8A	0.6916	0.2500	0.8539	0.054*	
C8A	0.5102 (4)	0.2500	0.7258 (3)	0.0355 (9)	
C9	0.5544 (4)	0.2500	0.3964 (3)	0.0384 (9)	
C10	0.4244 (4)	0.2500	0.2975 (3)	0.0452 (10)	
H10A	0.4243	0.1352	0.2460	0.054*	0.50
H10B	0.4243	0.3648	0.2460	0.054*	0.50
C11	0.2918 (4)	0.2500	0.3684 (3)	0.0472 (10)	
H11A	0.2322	0.3648	0.3486	0.057*	0.50
H11B	0.2322	0.1352	0.3486	0.057*	0.50

C12	0.6934 (4)	0.2500	0.3777 (3)	0.0447 (10)	
H12A	0.7667	0.2500	0.4449	0.054*	
C13	0.8823 (4)	0.2500	0.2525 (4)	0.0586 (13)	
H13A	0.9291	0.1354	0.2916	0.070*	0.50
H13B	0.9291	0.3646	0.2916	0.070*	0.50
C14	0.8937 (5)	0.2500	0.1161 (4)	0.0673 (14)	
H14A	0.8438	0.1361	0.0788	0.081*	0.50
H14B	0.8438	0.3639	0.0788	0.081*	0.50
C15	1.0456 (5)	0.2500	0.0877 (5)	0.096 (2)	
H15A	1.0448	0.2500	-0.0001	0.144*	
H15B	1.0953	0.1360	0.1224	0.144*	0.50
H15C	1.0953	0.3640	0.1224	0.144*	0.50
O1W	0.9030 (4)	0.2500	0.7141 (4)	0.1047 (17)	
H2	0.975 (8)	0.2500	0.665 (6)	0.15 (3)*	
H1	0.816 (8)	0.2500	0.676 (7)	0.17 (3)*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0461 (16)	0.078 (2)	0.0357 (15)	0.000	0.0161 (12)	0.000
O2	0.0307 (15)	0.126 (3)	0.0479 (17)	0.000	0.0058 (12)	0.000
N1	0.0308 (16)	0.050 (2)	0.0367 (17)	0.000	0.0074 (13)	0.000
C2	0.0345 (18)	0.036 (2)	0.035 (2)	0.000	0.0094 (15)	0.000
N3	0.0347 (16)	0.045 (2)	0.0310 (16)	0.000	0.0030 (13)	0.000
C4	0.039 (2)	0.055 (3)	0.037 (2)	0.000	0.0115 (16)	0.000
C4A	0.0354 (19)	0.045 (2)	0.0363 (19)	0.000	0.0099 (15)	0.000
C5	0.037 (2)	0.070 (3)	0.043 (2)	0.000	0.0093 (17)	0.000
C6	0.049 (2)	0.082 (3)	0.037 (2)	0.000	0.0172 (18)	0.000
C7	0.055 (3)	0.070 (3)	0.034 (2)	0.000	0.0061 (18)	0.000
C8	0.038 (2)	0.061 (3)	0.036 (2)	0.000	0.0033 (16)	0.000
C8A	0.0328 (19)	0.039 (2)	0.036 (2)	0.000	0.0093 (15)	0.000
C9	0.043 (2)	0.041 (2)	0.0321 (19)	0.000	0.0089 (16)	0.000
C10	0.055 (2)	0.048 (3)	0.032 (2)	0.000	0.0055 (17)	0.000
C11	0.043 (2)	0.065 (3)	0.033 (2)	0.000	0.0015 (16)	0.000
C12	0.048 (2)	0.055 (3)	0.031 (2)	0.000	0.0086 (17)	0.000
C13	0.044 (2)	0.087 (4)	0.049 (2)	0.000	0.0206 (19)	0.000
C14	0.054 (3)	0.104 (4)	0.049 (3)	0.000	0.022 (2)	0.000
C15	0.063 (3)	0.164 (7)	0.063 (3)	0.000	0.020 (3)	0.000
O1W	0.046 (2)	0.204 (5)	0.065 (2)	0.000	0.0116 (19)	0.000

Geometric parameters (Å, °)

O1—C12	1.336 (4)	C9—C12	1.329 (5)
O1—C13	1.439 (4)	C9—C10	1.502 (5)
O2—C4	1.226 (4)	C10—C11	1.539 (5)
N1—C2	1.302 (4)	C10—H10A	0.9700
N1—C8A	1.391 (4)	C10—H10B	0.9700
C2—N3	1.377 (4)	C11—H11A	0.9700

C2—C9	1.447 (5)	C11—H11B	0.9700
N3—C4	1.369 (4)	C12—H12A	0.9300
N3—C11	1.467 (4)	C13—C14	1.512 (5)
C4—C4A	1.464 (5)	C13—H13A	0.9700
C4A—C5	1.392 (5)	C13—H13B	0.9700
C4A—C8A	1.408 (5)	C14—C15	1.479 (6)
C5—C6	1.359 (5)	C14—H14A	0.9700
C5—H5A	0.9300	C14—H14B	0.9700
C6—C7	1.383 (5)	C15—H15A	0.9600
C6—H6A	0.9300	C15—H15B	0.9600
C7—C8	1.374 (5)	C15—H15C	0.9600
C7—H7A	0.9300	O1W—H2	0.91 (7)
C8—C8A	1.404 (5)	O1W—H1	0.85 (8)
C8—H8A	0.9300		
C12—O1—C13	116.8 (3)	C9—C10—H10A	110.8
C2—N1—C8A	116.3 (3)	C11—C10—H10A	110.8
N1—C2—N3	124.2 (3)	C9—C10—H10B	110.8
N1—C2—C9	127.8 (3)	C11—C10—H10B	110.8
N3—C2—C9	108.0 (3)	H10A—C10—H10B	108.9
C4—N3—C2	124.2 (3)	N3—C11—C10	104.6 (3)
C4—N3—C11	122.5 (3)	N3—C11—H11A	110.8
C2—N3—C11	113.3 (3)	C10—C11—H11A	110.8
O2—C4—N3	120.4 (4)	N3—C11—H11B	110.8
O2—C4—C4A	126.2 (3)	C10—C11—H11B	110.8
N3—C4—C4A	113.4 (3)	H11A—C11—H11B	108.9
C5—C4A—C8A	120.2 (3)	C9—C12—O1	120.8 (3)
C5—C4A—C4	120.7 (3)	C9—C12—H12A	119.6
C8A—C4A—C4	119.1 (3)	O1—C12—H12A	119.6
C6—C5—C4A	120.3 (4)	O1—C13—C14	106.6 (3)
C6—C5—H5A	119.9	O1—C13—H13A	110.4
C4A—C5—H5A	119.9	C14—C13—H13A	110.4
C5—C6—C7	120.6 (4)	O1—C13—H13B	110.4
C5—C6—H6A	119.7	C14—C13—H13B	110.4
C7—C6—H6A	119.7	H13A—C13—H13B	108.6
C8—C7—C6	120.5 (4)	C15—C14—C13	113.9 (4)
C8—C7—H7A	119.8	C15—C14—H14A	108.8
C6—C7—H7A	119.8	C13—C14—H14A	108.8
C7—C8—C8A	120.4 (4)	C15—C14—H14B	108.8
C7—C8—H8A	119.8	C13—C14—H14B	108.8
C8A—C8—H8A	119.8	H14A—C14—H14B	107.7
N1—C8A—C8	119.0 (3)	C14—C15—H15A	109.5
N1—C8A—C4A	122.9 (3)	C14—C15—H15B	109.5
C8—C8A—C4A	118.2 (3)	H15A—C15—H15B	109.5
C12—C9—C2	124.7 (3)	C14—C15—H15C	109.5
C12—C9—C10	125.7 (3)	H15A—C15—H15C	109.5
C2—C9—C10	109.6 (3)	H15B—C15—H15C	109.5
C9—C10—C11	104.5 (3)	H2—O1W—H1	115 (6)

Hydrogen-bond geometry (Å, °)

<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>
O1w—H1 \cdots N1	0.85 (7)	2.14 (8)	2.968 (5)	165 (7)
O1w—H2 \cdots O2 ⁱ	0.90 (7)	1.95 (7)	2.855 (5)	176 (6)

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