IUCrData

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9α-Hydroxy-4,8-dimethyl-3′-phenyl-3,14-dioxatricyclo[9.3.0.0^{2,4}]tetradec-7-en-13-one-12-spiro-5′-isoxazole monohydrate

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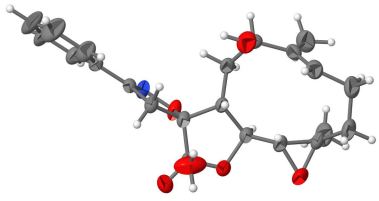
CCDC references: 1959478; 1959478

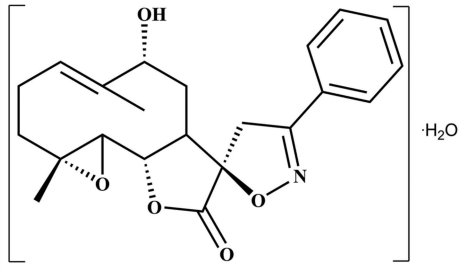
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{22}H_{25}NO_5\cdot H_2O$, the ten-membered ring displays an approximate chair–chair conformation, whereas the five-membered furan ring has an envelope conformation, with the C atom of the methine group adjacent to the spiro C atom as the flap. The isoxazole ring is almost planar and its plane is slightly inclined to the plane of the attached phenyl ring. The mean plane of the furan ring is nearly perpendicular to that of the isoxazole ring, as indicated by the dihedral angle between them of 89.39 (12)°. In the crystal, the organic molecules are linked into [010] chains by O—H···O hydrogen bonds. The water molecule forms O—H···O and O—H···N hydrogen bonds and a weak C—H···O interaction is also observed. Together, these lead to a three-dimensional network.

3D view



Chemical scheme



Structure description

The title compound was synthesized from 9α-hydroxyparthenolide (9α-hydroxy-4,8-dimethyl-12-methylene-3,14-dioxatricyclo[9.3.0.0^{2,4}]tetradec-7-en-13-one), which was isolated from the chloroform extract of the aerial parts of *Anvillea radiata* (Quezel & Santa, 1963; Ozenda, 1958). Our work lies within the framework of the evaluation of medicinal plants and, in particular, *Anvillea radiata*. The main constituent of the chloroform extract of the aerial parts of this plant is 9α-hydroxypartenolide (El Hassany *et al.*, 2004). The reactivities of this sesquiterpene lactone and its derivatives have been the subject of several studies (Castaneda-Acosta *et al.*, 1997; Neukirch *et al.*, 2003; Der-

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

D—H···A	D—H	H···A	D···A	D—H···A
C15—H15A···O2^{i}	0.97	2.56	3.251 (3)	128
O2—H2···O4^{ii}	0.82	2.03	2.822 (3)	163
O6—H6A···O1^{iii}	0.82	2.03	2.848 (3)	173
O6—H6B···N1^{ii}	0.82	2.08	2.863 (3)	159

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

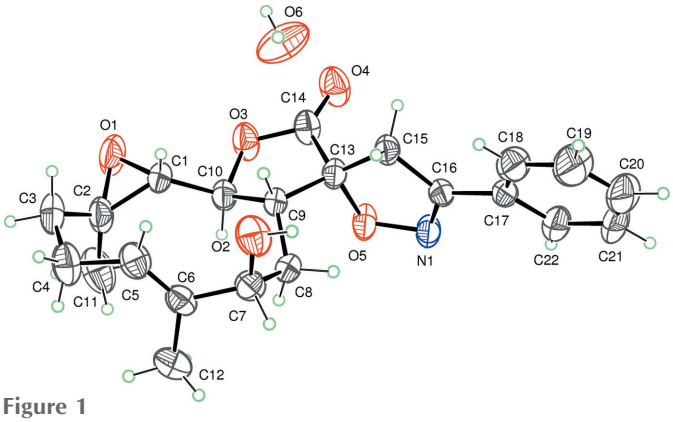


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

Ren *et al.*, 2006; Neelakantan *et al.*, 2009), in order to prepare products with potential use in industrial pharmacology. In this context, we have developed a synthesis of new spiro-isoxazolines by 1,3-dipolar cycloaddition reactions. We report here the crystal structure of the product arising from the treatment of 9α-hydroxyparthenolide with 1.5 equivalents of benzaldoxime in the presence of tetrahydrofuran (THF) and bleach (sodium hypochlorite) at room temperature, which crystallized as a monohydrate.

The organic molecule is built from fused five- and ten-membered rings, with an additional epoxy ring system and a 4,5-dihydro-3-phenylisoxazole group as a substituent (Fig. 1). The ten-membered ring adopts an approximate chair–chair conformation, while the furan ring displays an envelope conformation, with atom C9 as the flap. The dihedral angles between the isoxazole ring, the mean plan of the furan ring and the phenyl ring are 89.39 (12) and 15.45 (13)°, respec-

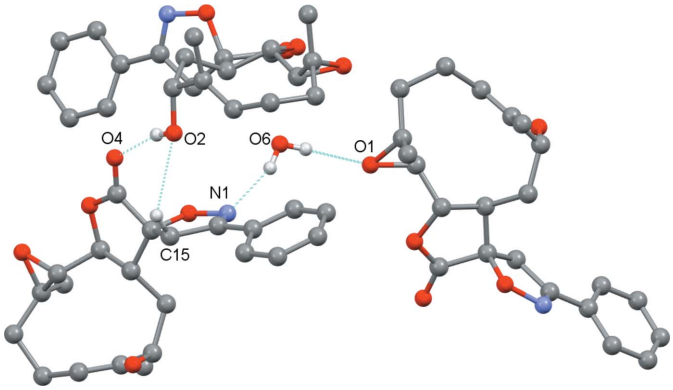


Figure 2
A fragment of the title structure, showing molecules connected by hydrogen bonds (dashed cyan lines).

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{22}H_{25}NO_5{\cdot}H_2O$
M_r	401.44
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	296
a, b, c (Å)	9.8947 (4), 10.6554 (4), 19.0286 (8)
V (Å^3)	2006.22 (14)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.10
Crystal size (mm)	0.35 × 0.30 × 0.22
Data collection	
Diffractometer	Bruker D8 VENTURE Super DUO
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.667, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	60442, 5184, 4262
R_{int}	0.049
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.676
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.042, 0.107, 1.04
No. of reflections	5184
No. of parameters	265
No. of restraints	3
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.24, −0.24
Absolute structure	Flack x determined using 1582 quotients $[(I^+) - (I^-)]/[(I^+) + (I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.2 (3)

Computer programs: *APEX3* (Bruker, 2016), *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2018* (Sheldrick, 2015*b*), *WinGX* and *ORTEP-3 for Windows* (Farrugia, 2012), *Mercury* (Macrae *et al.*, 2008) and *publCIF* (Westrip, 2010).

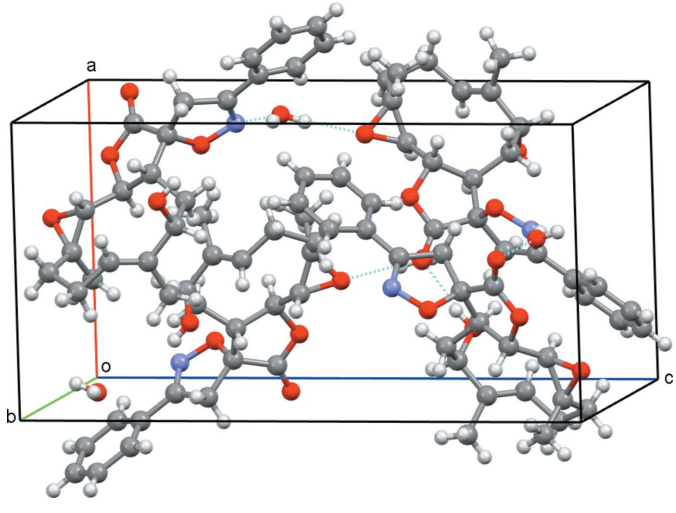


Figure 3
The crystal packing for the title compound, showing molecules linked by hydrogen bonds (dashed cyan lines).

tively. In the crystal, molecules are connected by four hydrogen bonds (Table 1), as shown in Fig. 2. The packing is shown in Fig. 3.

Synthesis and crystallization

9α-Hydroxyparthenolide (500 mg, 1.89 mmol) was treated with benzaldoxime (344 mg, 2.84 mmol) diluted in THF (10 ml) and 12% sodium hypochlorite solution (5 ml) was added dropwise. The reaction mixture was stirred at room temperature for 12 h. The mixture was diluted with water (10 ml) and extracted with CH_2Cl_2 (3 × 10 ml). The combined organic layers were dried with $MgSO_4$, filtered and concentrated under reduced pressure, providing a crude product. Chromatography of the residue obtained on a column of silica gel, eluting with hexane–ethyl acetate (70:30 *v*/*v*), allowed the isolation of the title compound in a yield of 67%. Crystallization of this product was carried out at room temperature from an ethyl acetate solution (m.p. 453–455 K) and the water molecule of crystallization was presumably incorporated from the surroundings.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The absolute structure could not be reliably established in the present experiment, but the relative configurations of the stereogenic centres are: C1 *R*, C2 *R*, C7 *R*, C9 *R*, C10 *S* and C13 *R*.

Acknowledgements

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full crystallographic data

IUCrData (2019). **4**, x191408 [https://doi.org/10.1107/S2414314619014081]

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Crystal data

$C_{22}H_{25}NO_5 \cdot H_2O$
M_r = 401.44
Orthorhombic, $P2_12_12_1$
a = 9.8947 (4) Å
b = 10.6554 (4) Å
c = 19.0286 (8) Å
V = 2006.22 (14) Å^3
Z = 4
$F(000)$ = 856
D_x = 1.329 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5184 reflections
θ = 2.3–28.7°
μ = 0.10 mm^{-1}
T = 296 K
Block, colourless
0.35 × 0.30 × 0.22 mm

Data collection

Bruker D8 VENTURE Super DUO diffractometer
Radiation source: INCOATEC IμS micro-focus source
HELIOS mirror optics monochromator
Detector resolution: 10.4167 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
T_{min} = 0.667, T_{max} = 0.746
60442 measured reflections
5184 independent reflections
4262 reflections with $I > 2\sigma(I)$
R_{int} = 0.049
θ_{max} = 28.7°, θ_{min} = 2.3°
h = −13→13
k = −14→14
l = −25→25

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.107
S = 1.03
5184 reflections
265 parameters
3 restraints
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0542P)^2 + 0.2739P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}
Extinction correction: SHELXL2018 (Sheldrick, 2015b), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.020 (3)
Absolute structure: Flack x determined using 1582 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons *et al.*, 2013)
Absolute structure parameter: 0.2 (3)

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.1760 (2)	0.7227 (2)	0.90463 (10)	0.0404 (5)
H1	0.224392	0.642647	0.907993	0.048*
C2	0.0347 (2)	0.7171 (2)	0.92900 (12)	0.0462 (5)
C3	−0.0159 (3)	0.5912 (3)	0.95379 (14)	0.0591 (7)
H3A	−0.088415	0.603557	0.987404	0.071*
H3B	0.056725	0.546968	0.977423	0.071*
C4	−0.0679 (3)	0.5109 (3)	0.89225 (16)	0.0586 (7)
H4A	−0.085000	0.426128	0.908619	0.070*
H4B	−0.152531	0.545344	0.875262	0.070*
C5	0.0324 (2)	0.5072 (2)	0.83321 (14)	0.0464 (5)
H5	0.108115	0.456949	0.840091	0.056*
C6	0.0255 (2)	0.5666 (2)	0.77299 (13)	0.0392 (5)
C7	0.1445 (2)	0.5737 (2)	0.72326 (12)	0.0401 (5)
H7	0.111355	0.555812	0.675786	0.048*
C8	0.2097 (2)	0.7044 (2)	0.72228 (11)	0.0395 (5)
H8A	0.266325	0.710666	0.680811	0.047*
H8B	0.138557	0.766483	0.717730	0.047*
C9	0.29549 (19)	0.7378 (2)	0.78669 (10)	0.0317 (4)
H9	0.333775	0.659763	0.805273	0.038*
C10	0.2273 (2)	0.8075 (2)	0.84807 (11)	0.0370 (5)
H10	0.154240	0.861266	0.830595	0.044*
C11	−0.0696 (3)	0.8083 (3)	0.90614 (19)	0.0681 (8)
H11A	−0.133670	0.820708	0.943398	0.102*
H11B	−0.115452	0.776345	0.865421	0.102*
H11C	−0.027341	0.886787	0.894806	0.102*
C12	−0.0961 (2)	0.6355 (3)	0.74590 (16)	0.0631 (8)
H12A	−0.174487	0.610443	0.772092	0.095*
H12B	−0.109088	0.615938	0.697116	0.095*
H12C	−0.082476	0.724223	0.751145	0.095*
C13	0.41259 (19)	0.82485 (19)	0.76828 (11)	0.0338 (4)
C14	0.4417 (2)	0.8933 (2)	0.83647 (12)	0.0399 (5)
C15	0.5313 (2)	0.76769 (19)	0.72907 (11)	0.0347 (4)
H15A	0.615524	0.780620	0.754171	0.042*
H15B	0.518251	0.678594	0.721140	0.042*
C16	0.5286 (2)	0.83963 (19)	0.66158 (11)	0.0325 (4)
C17	0.61864 (19)	0.8169 (2)	0.60106 (11)	0.0357 (4)
C18	0.6899 (3)	0.7063 (3)	0.59685 (14)	0.0546 (6)
H18	0.681597	0.646653	0.632272	0.065*
C19	0.7743 (3)	0.6833 (4)	0.53986 (17)	0.0759 (9)

H19	0.821728	0.608173	0.537020	0.091*
C20	0.7875 (3)	0.7709 (4)	0.48818 (15)	0.0757 (10)
H20	0.845054	0.755849	0.450492	0.091*
C21	0.7160 (3)	0.8817 (3)	0.49142 (14)	0.0655 (8)
H21	0.725273	0.940825	0.455801	0.079*
C22	0.6309 (3)	0.9053 (3)	0.54724 (12)	0.0490 (6)
H22	0.581824	0.979675	0.549041	0.059*
N1	0.43860 (18)	0.92543 (17)	0.65917 (10)	0.0391 (4)
O1	0.1401 (2)	0.7760 (2)	0.97130 (8)	0.0669 (6)
O2	0.23970 (18)	0.47987 (17)	0.74102 (10)	0.0558 (5)
H2	0.299560	0.487847	0.711560	0.084*
O3	0.33524 (17)	0.88429 (16)	0.87917 (9)	0.0476 (4)
O4	0.53835 (19)	0.95705 (17)	0.85124 (10)	0.0552 (5)
O5	0.36483 (15)	0.92729 (14)	0.72264 (9)	0.0430 (4)
O6	0.5694 (3)	0.6818 (2)	0.88559 (11)	0.0820 (7)
H6A	0.594778	0.698718	0.925509	0.123*
H6B	0.580171	0.605807	0.881833	0.123*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0382 (10)	0.0553 (13)	0.0276 (10)	−0.0055 (10)	0.0049 (8)	0.0042 (10)
C2	0.0436 (11)	0.0602 (14)	0.0349 (11)	−0.0079 (11)	0.0160 (9)	−0.0065 (10)
C3	0.0559 (14)	0.0747 (18)	0.0466 (14)	−0.0101 (14)	0.0208 (12)	0.0105 (13)
C4	0.0570 (15)	0.0557 (15)	0.0631 (17)	−0.0149 (12)	0.0187 (13)	0.0042 (13)
C5	0.0442 (12)	0.0403 (12)	0.0547 (14)	−0.0023 (10)	0.0068 (11)	−0.0014 (10)
C6	0.0313 (9)	0.0413 (11)	0.0450 (12)	−0.0039 (9)	0.0000 (9)	−0.0066 (10)
C7	0.0388 (10)	0.0470 (12)	0.0347 (11)	−0.0010 (9)	−0.0001 (9)	−0.0078 (9)
C8	0.0383 (10)	0.0523 (13)	0.0279 (10)	−0.0054 (9)	0.0013 (8)	0.0040 (9)
C9	0.0310 (9)	0.0375 (10)	0.0267 (9)	−0.0025 (8)	0.0037 (7)	0.0034 (8)
C10	0.0347 (10)	0.0447 (12)	0.0316 (10)	−0.0044 (9)	0.0064 (8)	0.0003 (9)
C11	0.0536 (15)	0.0609 (16)	0.090 (2)	0.0054 (13)	0.0273 (15)	−0.0072 (16)
C12	0.0371 (12)	0.084 (2)	0.0683 (19)	0.0054 (13)	−0.0057 (12)	0.0081 (15)
C13	0.0339 (9)	0.0350 (10)	0.0324 (10)	0.0010 (8)	0.0068 (8)	0.0048 (8)
C14	0.0420 (11)	0.0363 (10)	0.0413 (12)	−0.0062 (9)	0.0103 (9)	−0.0013 (9)
C15	0.0323 (9)	0.0378 (10)	0.0341 (10)	0.0025 (8)	0.0071 (8)	0.0037 (8)
C16	0.0292 (8)	0.0382 (10)	0.0303 (10)	−0.0047 (8)	0.0016 (8)	0.0000 (8)
C17	0.0305 (9)	0.0498 (12)	0.0269 (9)	−0.0024 (9)	0.0011 (8)	−0.0039 (9)
C18	0.0500 (13)	0.0685 (17)	0.0452 (13)	0.0169 (12)	0.0063 (11)	0.0025 (12)
C19	0.0748 (19)	0.095 (2)	0.0578 (18)	0.0356 (18)	0.0136 (15)	−0.0078 (17)
C20	0.0653 (17)	0.124 (3)	0.0379 (14)	0.0260 (19)	0.0184 (13)	−0.0058 (17)
C21	0.0696 (17)	0.094 (2)	0.0328 (13)	0.0055 (16)	0.0166 (12)	0.0089 (14)
C22	0.0523 (13)	0.0594 (14)	0.0354 (12)	0.0009 (12)	0.0082 (10)	0.0022 (11)
N1	0.0381 (9)	0.0437 (10)	0.0354 (9)	0.0024 (8)	0.0100 (8)	0.0089 (8)
O1	0.0700 (12)	0.1009 (16)	0.0300 (8)	−0.0317 (12)	0.0150 (8)	−0.0098 (9)
O2	0.0506 (9)	0.0519 (10)	0.0648 (12)	0.0128 (8)	0.0138 (9)	−0.0071 (9)
O3	0.0479 (9)	0.0534 (9)	0.0415 (8)	−0.0148 (8)	0.0152 (7)	−0.0135 (7)
O4	0.0526 (10)	0.0539 (10)	0.0590 (11)	−0.0206 (8)	0.0117 (9)	−0.0098 (8)

O5	0.0450 (8)	0.0406 (8)	0.0433 (9)	0.0102 (7)	0.0181 (7)	0.0135 (7)
O6	0.133 (2)	0.0567 (11)	0.0562 (12)	0.0122 (13)	−0.0280 (14)	−0.0094 (10)

Geometric parameters (Å, º)

C1—O1	1.435 (3)	C11—H11C	0.9600
C1—C2	1.474 (3)	C12—H12A	0.9600
C1—C10	1.494 (3)	C12—H12B	0.9600
C1—H1	0.9800	C12—H12C	0.9600
C2—O1	1.459 (3)	C13—O5	1.473 (2)
C2—C11	1.483 (4)	C13—C14	1.516 (3)
C2—C3	1.508 (4)	C13—C15	1.519 (3)
C3—C4	1.539 (4)	C14—O4	1.206 (3)
C3—H3A	0.9700	C14—O3	1.334 (3)
C3—H3B	0.9700	C15—C16	1.496 (3)
C4—C5	1.499 (4)	C15—H15A	0.9700
C4—H4A	0.9700	C15—H15B	0.9700
C4—H4B	0.9700	C16—N1	1.277 (3)
C5—C6	1.311 (3)	C16—C17	1.476 (3)
C5—H5	0.9300	C17—C18	1.376 (3)
C6—C12	1.501 (3)	C17—C22	1.396 (3)
C6—C7	1.513 (3)	C18—C19	1.390 (4)
C7—O2	1.414 (3)	C18—H18	0.9300
C7—C8	1.535 (3)	C19—C20	1.362 (5)
C7—H7	0.9800	C19—H19	0.9300
C8—C9	1.533 (3)	C20—C21	1.377 (5)
C8—H8A	0.9700	C20—H20	0.9300
C8—H8B	0.9700	C21—C22	1.379 (3)
C9—C13	1.525 (3)	C21—H21	0.9300
C9—C10	1.540 (3)	C22—H22	0.9300
C9—H9	0.9800	N1—O5	1.411 (2)
C10—O3	1.470 (3)	O2—H2	0.8199
C10—H10	0.9800	O6—H6A	0.8202
C11—H11A	0.9600	O6—H6B	0.8203
C11—H11B	0.9600		

O1—C1—C2	60.19 (14)	C2—C11—H11A	109.5
O1—C1—C10	118.8 (2)	C2—C11—H11B	109.5
C2—C1—C10	124.9 (2)	H11A—C11—H11B	109.5
O1—C1—H1	114.1	C2—C11—H11C	109.5
C2—C1—H1	114.1	H11A—C11—H11C	109.5
C10—C1—H1	114.1	H11B—C11—H11C	109.5
O1—C2—C1	58.56 (14)	C6—C12—H12A	109.5
O1—C2—C11	112.2 (2)	C6—C12—H12B	109.5
C1—C2—C11	122.8 (2)	H12A—C12—H12B	109.5
O1—C2—C3	116.6 (2)	C6—C12—H12C	109.5
C1—C2—C3	116.7 (2)	H12A—C12—H12C	109.5
C11—C2—C3	116.3 (2)	H12B—C12—H12C	109.5

C2—C3—C4	111.6 (2)	O5—C13—C14	102.06 (16)
C2—C3—H3A	109.3	O5—C13—C15	104.81 (15)
C4—C3—H3A	109.3	C14—C13—C15	117.79 (18)
C2—C3—H3B	109.3	O5—C13—C9	110.02 (16)
C4—C3—H3B	109.3	C14—C13—C9	103.90 (16)
H3A—C3—H3B	108.0	C15—C13—C9	117.18 (17)
C5—C4—C3	111.3 (2)	O4—C14—O3	121.7 (2)
C5—C4—H4A	109.4	O4—C14—C13	128.4 (2)
C3—C4—H4A	109.4	O3—C14—C13	109.67 (17)
C5—C4—H4B	109.4	C16—C15—C13	101.66 (16)
C3—C4—H4B	109.4	C16—C15—H15A	111.4
H4A—C4—H4B	108.0	C13—C15—H15A	111.4
C6—C5—C4	127.4 (2)	C16—C15—H15B	111.4
C6—C5—H5	116.3	C13—C15—H15B	111.4
C4—C5—H5	116.3	H15A—C15—H15B	109.3
C5—C6—C12	125.3 (2)	N1—C16—C17	120.69 (18)
C5—C6—C7	122.0 (2)	N1—C16—C15	114.23 (17)
C12—C6—C7	112.6 (2)	C17—C16—C15	125.09 (18)
O2—C7—C6	109.50 (19)	C18—C17—C22	119.4 (2)
O2—C7—C8	111.38 (18)	C18—C17—C16	119.7 (2)
C6—C7—C8	112.32 (18)	C22—C17—C16	120.9 (2)
O2—C7—H7	107.8	C17—C18—C19	120.3 (3)
C6—C7—H7	107.8	C17—C18—H18	119.9
C8—C7—H7	107.8	C19—C18—H18	119.9
C9—C8—C7	115.70 (18)	C20—C19—C18	120.0 (3)
C9—C8—H8A	108.4	C20—C19—H19	120.0
C7—C8—H8A	108.4	C18—C19—H19	120.0
C9—C8—H8B	108.4	C19—C20—C21	120.4 (2)
C7—C8—H8B	108.4	C19—C20—H20	119.8
H8A—C8—H8B	107.4	C21—C20—H20	119.8
C13—C9—C8	112.22 (16)	C20—C21—C22	120.3 (3)
C13—C9—C10	102.36 (16)	C20—C21—H21	119.9
C8—C9—C10	118.42 (16)	C22—C21—H21	119.9
C13—C9—H9	107.8	C21—C22—C17	119.6 (3)
C8—C9—H9	107.8	C21—C22—H22	120.2
C10—C9—H9	107.8	C17—C22—H22	120.2
O3—C10—C1	107.07 (17)	C16—N1—O5	109.87 (16)
O3—C10—C9	104.78 (15)	C1—O1—C2	61.26 (13)
C1—C10—C9	113.77 (18)	C7—O2—H2	104.1
O3—C10—H10	110.3	C14—O3—C10	111.65 (16)
C1—C10—H10	110.3	N1—O5—C13	109.17 (14)
C9—C10—H10	110.3	H6A—O6—H6B	104.9

C10—C1—C2—O1	−106.1 (3)	C15—C13—C14—O4	35.8 (3)
O1—C1—C2—C11	97.6 (3)	C9—C13—C14—O4	167.2 (2)
C10—C1—C2—C11	−8.4 (4)	O5—C13—C14—O3	95.55 (19)
O1—C1—C2—C3	−106.2 (2)	C15—C13—C14—O3	−150.37 (19)
C10—C1—C2—C3	147.7 (2)	C9—C13—C14—O3	−18.9 (2)

O1—C2—C3—C4	−151.8 (2)	O5—C13—C15—C16	−4.7 (2)
C1—C2—C3—C4	−85.5 (3)	C14—C13—C15—C16	−117.29 (19)
C11—C2—C3—C4	72.2 (3)	C9—C13—C15—C16	117.54 (19)
C2—C3—C4—C5	49.3 (3)	C13—C15—C16—N1	3.4 (2)
C3—C4—C5—C6	−105.1 (3)	C13—C15—C16—C17	−176.11 (18)
C4—C5—C6—C12	−10.3 (4)	N1—C16—C17—C18	−163.4 (2)
C4—C5—C6—C7	168.7 (2)	C15—C16—C17—C18	16.1 (3)
C5—C6—C7—O2	17.6 (3)	N1—C16—C17—C22	15.5 (3)
C12—C6—C7—O2	−163.2 (2)	C15—C16—C17—C22	−165.0 (2)
C5—C6—C7—C8	−106.7 (2)	C22—C17—C18—C19	0.6 (4)
C12—C6—C7—C8	72.5 (3)	C16—C17—C18—C19	179.5 (2)
O2—C7—C8—C9	−48.7 (3)	C17—C18—C19—C20	0.5 (5)
C6—C7—C8—C9	74.5 (2)	C18—C19—C20—C21	−1.0 (5)
C7—C8—C9—C13	148.31 (18)	C19—C20—C21—C22	0.3 (5)
C7—C8—C9—C10	−92.7 (2)	C20—C21—C22—C17	0.8 (4)
O1—C1—C10—O3	50.1 (3)	C18—C17—C22—C21	−1.3 (4)
C2—C1—C10—O3	122.2 (2)	C16—C17—C22—C21	179.8 (2)
O1—C1—C10—C9	165.37 (18)	C17—C16—N1—O5	179.14 (17)
C2—C1—C10—C9	−122.6 (2)	C15—C16—N1—O5	−0.3 (2)
C13—C9—C10—O3	−25.46 (19)	C10—C1—O1—C2	116.0 (2)
C8—C9—C10—O3	−149.44 (18)	C11—C2—O1—C1	−115.9 (2)
C13—C9—C10—C1	−142.07 (18)	C3—C2—O1—C1	106.4 (2)
C8—C9—C10—C1	94.0 (2)	O4—C14—O3—C10	176.6 (2)
C8—C9—C13—O5	45.8 (2)	C13—C14—O3—C10	2.3 (2)
C10—C9—C13—O5	−82.22 (18)	C1—C10—O3—C14	136.3 (2)
C8—C9—C13—C14	154.41 (17)	C9—C10—O3—C14	15.1 (2)
C10—C9—C13—C14	26.4 (2)	C16—N1—O5—C13	−3.0 (2)
C8—C9—C13—C15	−73.8 (2)	C14—C13—O5—N1	128.28 (17)
C10—C9—C13—C15	158.24 (17)	C15—C13—O5—N1	4.9 (2)
O5—C13—C14—O4	−78.3 (3)	C9—C13—O5—N1	−121.88 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C15—H15*A*···O2^{i}	0.97	2.56	3.251 (3)	128
O2—H2···O4^{ii}	0.82	2.03	2.822 (3)	163
O6—H6*A*···O1^{iii}	0.82	2.03	2.848 (3)	173
O6—H6*B*···N1^{ii}	0.82	2.08	2.863 (3)	159

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