

Acta Crystallographica Section C

Structural Chemistry

ISSN 2053-2296

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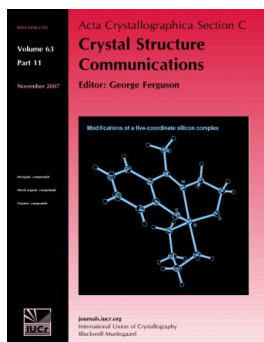
Cristian Paz Robles, Viviana Burgos, Sebastián Suarez and Ricardo Baggio

Acta Cryst. (2014). **C70**, 1007–1010

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Dendocarbin A: a sesquiterpene lactone from *Drimys winteri*

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Received 7 August 2014

Accepted 29 September 2014

The natural compound dendocarbin A, C₁₅H₂₂O₃, is a sesquiterpene lactone isolated for the first time from *Drimys winteri* for var *chilensis*. The compound crystallizes in the orthorhombic space group *P*2₁2₁2₁ and its X-ray crystal structure confirmed the *S/R* character of the chiral centres at C-5/C-10 and C-9/C-11, respectively. The α -OH group at C-11 was found to be involved in intermolecular hydrogen bonding, defining chains along the $\langle 100 \rangle$ 2₁ screw axis.

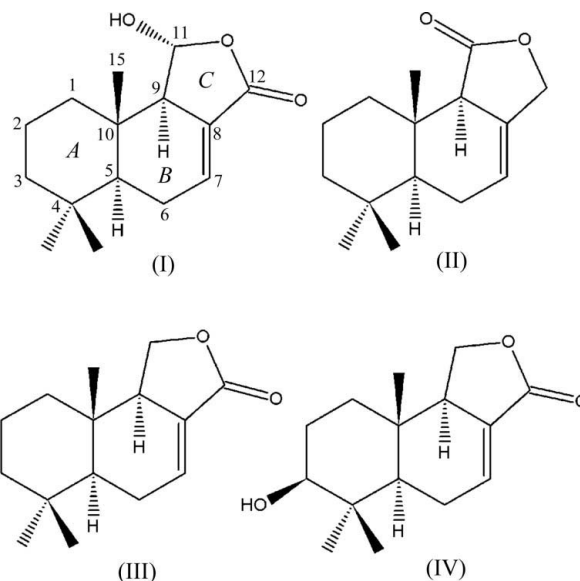
Keywords: crystal structure; natural sesquiterpene lactone; natural product; dendocarbin A; cytotoxic activity; *Drimys winteri*.

1. Introduction

Drimys winteri (Winteraceae) is a native tree of Chile, sacred for the native people (Araucanian) due to its medicinal properties, including bactericidal, antifungal and insecticidal activity (Kubo *et al.*, 2005; Jansen & de Groot, 2004). The main secondary metabolites in its barks are drimane sesquiterpenoids which have been described by Appel *et al.* (1963). On the other hand, the main scope of the present report, the natural compound dendocarbin A, (I), even if not novel, has been treated only tangentially in the literature. It was originally obtained from ethanol extracts of the Japanese udibranch *Dendrodoris carbunculosa* by Sakio *et al.* (2001), who found cytotoxic activity in its extracts. A few years later, Gaspar *et al.* (2005), reported the first chemical study of the porostome nudibranch *Doriopsilla pelseneeri* collected off the Portuguese coast, finding in his case the secondary metabolite. Finally, Xu *et al.* (2009) reported compound (I) as being isolated from the ethyl acetate extract of *Warburgia ugandensis* (Canellaceae) barks.

The present work is part of a series of structural characterizations of naturally occurring molecules isolated from

southern Andean flora (a seemingly inextinguishable source for extractive chemists). We describe herein the crystal structure of (I), isolated for the first time from *Drimys winteri* var *chilensis* (Winteraceae), in order to ascertain unambiguously the relative stereochemistry of the OH group at C-11 and the methyl group at C-15, as well as to confirm the relative configurations of the remaining asymmetric centres.



2. Experimental

2.1. Synthesis and crystallization

Compound (I) was isolated from the stem bark of *Drimys winteri* (Canelo) collected in Concepcion, VIII Region of Chile, in February 2012. The bark (1 kg) was powdered and extracted by maceration with ethanol for 3 d, giving a crude product (20 g) which was further purified by column chromatography. Compound (I) was obtained as a white solid from hexane/ethyl acetate (1:1 v/v) and was recrystallized from methanol producing colourless crystals suitable for X-ray diffraction analysis.

2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. All H atoms were identified in an intermediate difference map and were refined with different constraints according to their environment. H atoms on C atoms were idealized and allowed to ride both in coordinates and in displacement parameters, the latter taken as $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C})$, with C–H = 0.93 Å and $x = 1.2$ for aromatic, C–H = 0.97 Å and $x = 1.2$ for methylene and C–H = 0.96 Å and $x = 1.5$ for methyl groups. The hydroxy H atom was refined freely. The combined effect of weak diffractors and a medium quality data set precluded a trustworthy determination of the configuration of the chiral centres. The presently reported ‘handedness’, however, defined by C5(*S*), C9(*R*), C10(*S*), C11(*R*), was found to coincide with that reported in (IV), which was in turn assigned by similarity with related compounds.

Table 1

Experimental details.

Crystal data	
Chemical formula	C ₁₅ H ₂₂ O ₃
<i>M_r</i>	250.32
Crystal system, space group	Orthorhombic, <i>P</i> 2 ₁ 2 ₁ 2 ₁
Temperature (K)	295
<i>a</i> , <i>b</i> , <i>c</i> (Å)	6.335 (4), 13.399 (5), 16.613 (5)
<i>V</i> (Å ³)	1410.2 (11)
<i>Z</i>	4
Radiation type	Mo <i>K</i> α
<i>μ</i> (mm ⁻¹)	0.08
Crystal size (mm)	0.35 × 0.25 × 0.20
Data collection	
Diffractometer	Oxford Diffraction Gemini CCD S Ultra diffractometer
Absorption correction	Multi-scan (<i>CrysAlis PRO</i> ; Oxford Diffraction, 2009)
<i>T_{min}</i> , <i>T_{max}</i>	0.91, 0.94
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	12609, 3411, 2059
<i>R_{int}</i>	0.044
(sin θ/λ) _{max} (Å ⁻¹)	0.687
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.055, 0.133, 0.99
No. of reflections	3411
No. of parameters	170
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
Δρ _{max} , Δρ _{min} (e Å ⁻³)	0.13, -0.16
Absolute structure	Flack <i>x</i> determined using 627 quotients [(<i>I</i> ⁺) - (<i>I</i> ⁻)] / [(<i>I</i> ⁺) + (<i>I</i> ⁻)] (Parsons & Flack, 2004)
Absolute structure parameter	0.3 (8)

Computer programs: *CrysAlis PRO* (Oxford Diffraction, 2009), *SHELXS97* (Sheldrick, 2008), *XP* in *SHELXTL* (Sheldrick, 2008), *SHELXL2013* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

3. Results and discussion

The molecule of (I) (Fig. 1) is characterized by a rigid backbone made up of three fused rings (see Scheme for labelling), where lateral ring *A* (atoms C1–C5/C10) has a chair conformation [$\theta = 6.1$ (4)°; *cf.* $\theta = 0.00^\circ$ for an ideal chair conformation (Boeyens, 1978)], central ring *B* (atoms C5–C10) has a

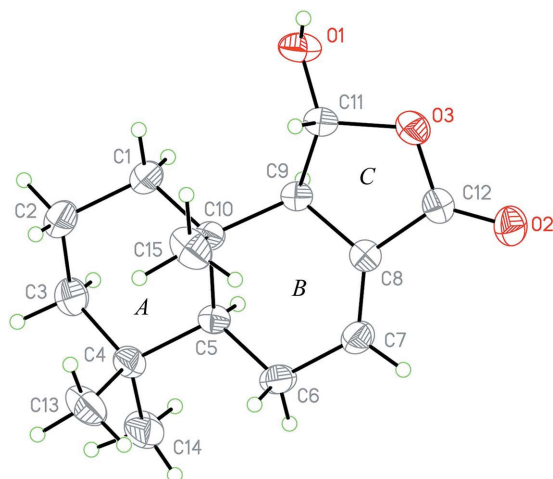


Figure 1

The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level.

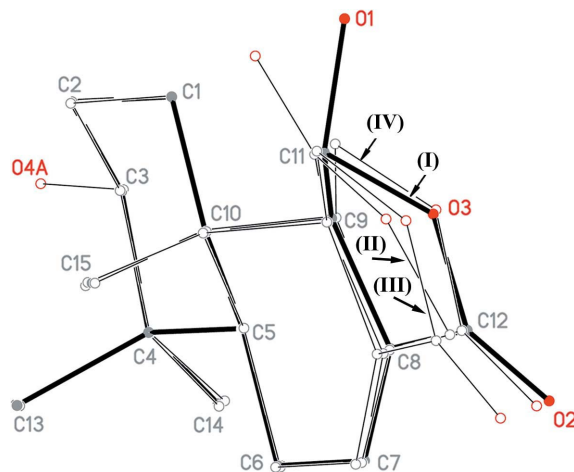


Figure 2

Superposition of the structures of (I), (II), (III) and (IV).

half-chair conformation [$\theta = 52.8$ (3)° and $\varphi = 321.5$ (5)° = $5 \times 60 + 21.5^\circ$; *cf.* $\theta = 50.8$ and $\varphi = k \times 60 + 30^\circ$ for an ideal half-chair conformation (Boeyens, 1978)] and five-membered lactone ring *C* (atoms C8/C9/C11/C12/O3) has an envelope conformation [$\varphi = 63.7$ (6)° = $2 \times 36 - 8.3^\circ$; *cf.* $\varphi = k \times 36 + 0^\circ$ for an ideal envelope conformation (Cremer & Pople, 1975)], with the carbonyl group at atom C12 conjugated with the C7=C8 double bond. It is worth mentioning that this envelope geometry for the lactone ring is favoured by the ‘outer’ position of the double bond; when the location is instead ‘inner’ (C8=C9), the group is strictly planar, with mean deviations from planarity smaller than 0.02 Å (see, for example, Nicotra *et al.*, 2006; Qian & Zhao, 2012; von Nussbaum *et al.*, 2012)

A search of the Cambridge Structural Database (CSD, Version 5.34; Allen, 2002) disclosed that the structure of (I) is closely related to three analogues, *viz.* the dimeric lactone drimenin [(II); CSD refcode DIWSEI; Brito, López-Rodríguez *et al.*, 2008], cinnamolide [(III); CSD refcode UTONUN; Brito, Cardenas *et al.* 2008] and 3-hydroxy-7-drimen-12,11-olide hemihydrate [(IV); CSD refcode UCOKUT; Zhang *et*

Table 2

Comparison of the corresponding parameters (Å, °) in (I), (II), (III) and (IV).

	(I)	(II)	(III)	(IV)
O3–C12	1.343 (4)	1.449 (4)	1.345 (5)	1.358 (3)
O3–C11	1.483 (4)	1.348 (4)	1.453 (5)	1.467 (2)
O2–C12	1.208 (4)		1.202 (5)	1.204 (2)
O1–C11	1.370 (4)	1.199 (3)		
O3–C11–C9	104.1 (2)	110.3 (2)	106.0 (2)	105.47 (14)
O3–C12–C8	108.3 (3)	104.1 (2)	108.9 (3)	108.44 (16)
O3–C11–O1	109.1 (2)	120.3 (3)		
O1–C11–C9	113.1 (3)	129.3 (3)		
C7–C8–C12–O3	-158.6 (3)	-150.1 (3)	-161.5 (3)	-164.7 (2)
C8–C9–C11–O3	27.9 (3)	15.4 (3)	22.2 (3)	23.8 (2)
C10–C9–C11–O3	152.2 (2)	140.3 (2)	145.3 (3)	147.30 (17)
C9–C8–C12–O3	14.3 (3)	23.0 (3)	10.0 (4)	9.3 (3)
C8–C9–C11–O1	146.0 (3)	-166.2 (3)		
C12–O3–C11–O1	-141.2 (3)	-179.9 (3)		

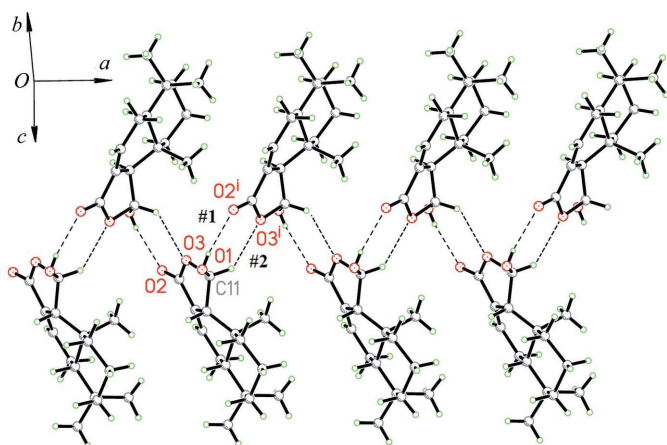


Figure 3
A view of the hydrogen-bonded chain in (I), with the hydrogen bonds drawn as broken lines. Hashes (#) indicate hydrogen bonds included in Table 3. [Symmetry code: (i) $x + \frac{1}{2}, -y + \frac{3}{2}, -z + 1$.]

al., 2006] (see Scheme). All four structures are, as expected, quite similar and Table 2 provides a comparison of corresponding parameters highlighting the most noticeable differences, while Fig. 2 presents, in turn, a superposition of all four molecules, where the almost identical rings *A*, unaffected by the differing locations of the carbonyl group, have been used for the least-squares fitting.

Table 3
Hydrogen-bond geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
O1—H1O···O2 ⁱ	0.94 (4)	1.89 (5)	2.832 (4)	176 (4)
C11—H11···O3 ⁱ	0.98	2.40	3.190 (4)	137
C6—H6B···O1 ⁱⁱ	0.97	2.67	3.630 (4)	172

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

The most relevant differences regarding bond distances or angles are to be found around the lactone O3 atom, and are related to the position of the carbonyl group [C12=O2 in (I), (III) and (IV), and C11=O1 in (II)]. In all cases, the C=O group presents a clear resonance with the neighbouring C12—O3 (C11—O3) group, which is significantly shorter than its C11—O3 (C12—O3) neighbour (see Table 2). On the other hand, the identical lactone rings in (III) and (IV) appear rather parallel to each other, even if slightly offset. The inclusion of an O atom at C11, either single bonded as in (I) or double bonded as in (II), tends to twist the group, as shown in Fig. 2, and can be assessed by the difference in the torsion angles presented in Table 2.

Regarding the supramolecular structure, there are two significant intermolecular interactions in (I) (entries 1 and 2 in Table 3). These hydrogen bonds generate $R_2^2(7)$ loops (for graph-set nomenclature, see Bernstein *et al.*, 1995) connecting neighbouring molecules along the rather short *a* direction,

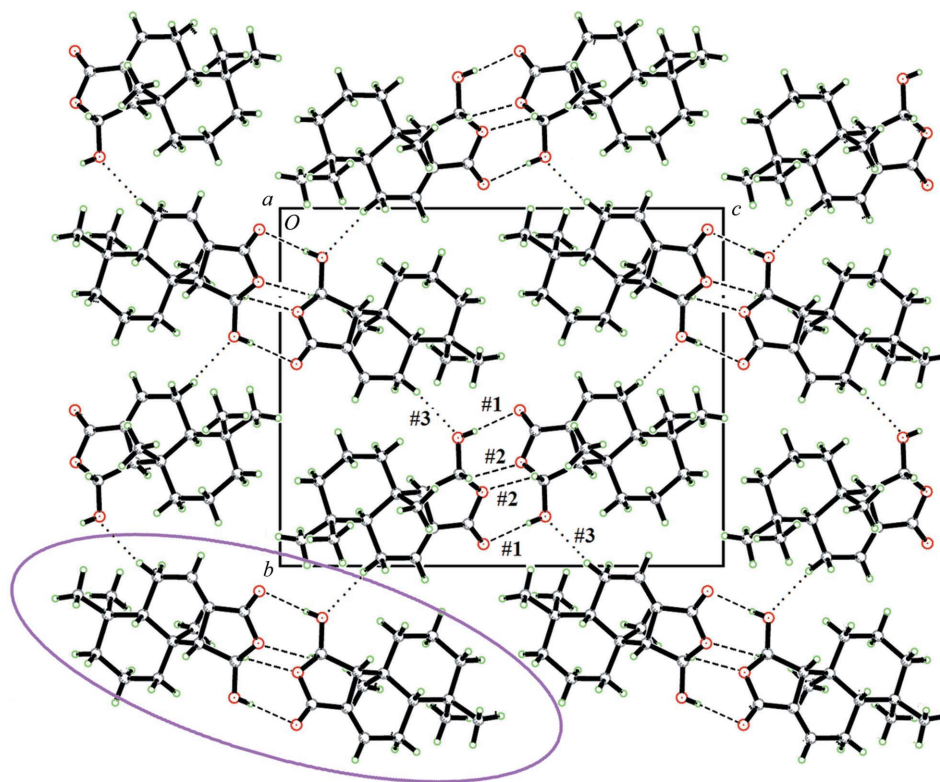


Figure 4
Packing view of (I) along *a*, the chain direction, showing the latter in projection and the intra/inter-chain hydrogen bonds with broken/dotted lines. Hashes (#) indicate hydrogen bonds included in Table 3.

riding on a twofold screw axis. This generates a one-dimensional substructure threaded by the symmetry axis (Fig. 3). Due to the particular disposition of the molecules in these chains, the graph-set codes describing the responsible synthons have extremely short repetition periods, *viz.* $C(6)$ for the $O-H \cdots O$ and $C(3)$ for the $C-H \cdots O$ hydrogen bonds.

These $\langle 100 \rangle$ chains are poorly interacting, the only link worth mentioning being an extremely weak $C-H \cdots O$ contact (presented as entry 3 in Table 3 and drawn as dotted lines in Fig. 4), by way of which the parallel chains form a weakly bound three-dimensional structure. By comparison, structures (II) and (III), which do not have any active hydrogen-bond donors present, display only normal van der Waals contacts. Compound (IV), instead, presents comparable intermolecular interactions, through the OH groups in the two independent moieties, as well as an active water solvent molecule, giving rise to tightly bound two-dimensional substructures of juxtaposed chains. In spite of the obvious differences due to the different OH position and the presence of the solvent water in (IV), the way in which chains are formed is similar, threaded along a 2_1 axis.

The authors are grateful for the financial support given by project Fondecyt 3130378 and the University of La Frontera. ANPCyT project PME 2006–01113 is acknowledged for the purchase of the Oxford Gemini CCD diffractometer.

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supporting information

Acta Cryst. (2014). **C70**, 1007-1010 [doi:10.1107/S205322961402155X]

Dendocarin A: a sesquiterpene lactone from *Drimys winteri*

Cristian Paz Robles, Viviana Burgos, Sebastián Suarez and Ricardo Baggio

Computing details

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO* (Oxford Diffraction, 2009); data reduction: *CrysAlis PRO* (Oxford Diffraction, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2008); molecular graphics: *XP in SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL2013* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

(1R,5aS,9aS,9bR)-5,5a,6,7,8,9,9a,9b-Octahydro-1-hydroxy-6,6,9a-trimethylnaphtho[1,2-c]furan-3(1H)-one

Crystal data

$C_{15}H_{22}O_3$

$M_r = 250.32$

Orthorhombic, $P2_12_12_1$

$a = 6.335$ (4) Å

$b = 13.399$ (5) Å

$c = 16.613$ (5) Å

$V = 1410.2$ (11) Å³

$Z = 4$

$F(000) = 544$

$D_x = 1.179$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å

Cell parameters from 2073 reflections

$\theta = 3.9$ – 21.5°

$\mu = 0.08$ mm⁻¹

$T = 295$ K

Block, colourless

$0.35 \times 0.25 \times 0.20$ mm

Data collection

Oxford Diffraction Gemini CCD S Ultra diffractometer

ω scans, thick slices

Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2009)

$T_{\min} = 0.91$, $T_{\max} = 0.94$

12609 measured reflections

3411 independent reflections

2059 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.044$

$\theta_{\max} = 29.2^\circ$, $\theta_{\min} = 3.8^\circ$

$h = -8 \rightarrow 8$

$k = -18 \rightarrow 17$

$l = -22 \rightarrow 18$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.055$

$wR(F^2) = 0.133$

$S = 0.99$

3411 reflections

170 parameters

0 restraints

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0532P)^2 + 0.1546P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.13$ e Å⁻³

$\Delta\rho_{\min} = -0.16$ e Å⁻³

Absolute structure: Flack x determined using

627 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons & Flack, 2004)

Absolute structure parameter: 0.3 (8)

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.0934 (4)	0.64026 (17)	0.40375 (15)	0.0712 (7)
H1O	0.158 (7)	0.616 (3)	0.451 (3)	0.110 (15)*
O2	-0.1961 (4)	0.93607 (18)	0.45904 (13)	0.0734 (7)
O3	-0.0232 (4)	0.79121 (17)	0.45638 (11)	0.0655 (6)
C1	0.3357 (7)	0.6921 (3)	0.2291 (2)	0.0815 (11)
H1A	0.2005	0.6637	0.2141	0.098*
H1B	0.3970	0.6496	0.2701	0.098*
C2	0.4797 (8)	0.6918 (3)	0.1556 (2)	0.1016 (15)
H2A	0.6198	0.7141	0.1708	0.122*
H2B	0.4911	0.6245	0.1344	0.122*
C3	0.3901 (7)	0.7605 (3)	0.0918 (2)	0.0838 (12)
H3A	0.2540	0.7348	0.0749	0.101*
H3B	0.4829	0.7592	0.0453	0.101*
C4	0.3626 (5)	0.8683 (3)	0.11857 (18)	0.0576 (8)
C5	0.2292 (4)	0.8690 (2)	0.19758 (16)	0.0466 (7)
H5	0.0893	0.8454	0.1811	0.056*
C6	0.1905 (6)	0.9740 (2)	0.23063 (19)	0.0666 (9)
H6A	0.3254	1.0064	0.2397	0.080*
H6B	0.1142	1.0127	0.1908	0.080*
C7	0.0686 (6)	0.9739 (3)	0.3072 (2)	0.0656 (9)
H7	0.0195	1.0340	0.3280	0.079*
C8	0.0286 (4)	0.8903 (2)	0.34624 (17)	0.0487 (7)
C9	0.1088 (4)	0.7899 (2)	0.32286 (16)	0.0443 (7)
H9	-0.0056	0.7551	0.2947	0.053*
C10	0.2986 (4)	0.7955 (2)	0.26493 (17)	0.0467 (7)
C11	0.1352 (5)	0.7406 (2)	0.40490 (17)	0.0531 (8)
H11	0.2777	0.7526	0.4257	0.064*
C12	-0.0772 (5)	0.8798 (3)	0.42476 (18)	0.0557 (8)
C13	0.5800 (6)	0.9193 (4)	0.1250 (3)	0.0924 (14)
H13A	0.6452	0.9213	0.0729	0.139*
H13B	0.6679	0.8823	0.1615	0.139*
H13C	0.5624	0.9861	0.1448	0.139*
C14	0.2401 (7)	0.9234 (3)	0.0522 (2)	0.0823 (12)
H14A	0.3099	0.9140	0.0015	0.124*
H14B	0.2347	0.9934	0.0645	0.124*
H14C	0.0991	0.8974	0.0491	0.124*
C15	0.4940 (5)	0.8312 (3)	0.3109 (2)	0.0809 (12)
H15A	0.5342	0.7817	0.3497	0.121*
H15B	0.4624	0.8927	0.3379	0.121*

H15C 0.6080 0.8417 0.2737 0.121*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0958 (18)	0.0539 (15)	0.0640 (15)	-0.0100 (13)	-0.0033 (13)	0.0134 (11)
O2	0.0826 (16)	0.0779 (17)	0.0596 (14)	0.0063 (14)	0.0100 (13)	-0.0122 (12)
O3	0.0768 (15)	0.0693 (15)	0.0505 (11)	0.0004 (13)	0.0082 (12)	0.0064 (11)
C1	0.109 (3)	0.057 (2)	0.078 (2)	0.025 (2)	0.027 (2)	0.0098 (19)
C2	0.138 (4)	0.076 (3)	0.090 (3)	0.042 (3)	0.043 (3)	0.011 (2)
C3	0.109 (3)	0.079 (3)	0.063 (2)	0.014 (2)	0.026 (2)	-0.001 (2)
C4	0.0609 (19)	0.062 (2)	0.0501 (18)	-0.0001 (17)	0.0052 (15)	0.0069 (15)
C5	0.0444 (15)	0.0480 (17)	0.0474 (15)	-0.0025 (13)	-0.0077 (13)	0.0048 (13)
C6	0.092 (2)	0.0468 (19)	0.0613 (19)	0.0037 (18)	0.006 (2)	0.0084 (16)
C7	0.085 (2)	0.0519 (19)	0.0599 (19)	0.0117 (18)	0.0048 (19)	-0.0014 (16)
C8	0.0470 (15)	0.0538 (19)	0.0454 (16)	-0.0022 (14)	-0.0067 (13)	-0.0007 (13)
C9	0.0423 (14)	0.0458 (17)	0.0447 (15)	-0.0053 (13)	-0.0080 (13)	0.0007 (12)
C10	0.0395 (14)	0.0495 (17)	0.0509 (15)	0.0020 (14)	-0.0041 (13)	0.0084 (13)
C11	0.0567 (18)	0.0507 (19)	0.0520 (17)	-0.0047 (15)	-0.0037 (14)	0.0071 (14)
C12	0.0528 (18)	0.063 (2)	0.0508 (18)	-0.0056 (16)	-0.0020 (15)	-0.0073 (16)
C13	0.072 (2)	0.120 (4)	0.086 (3)	-0.017 (2)	0.015 (2)	0.026 (2)
C14	0.097 (3)	0.096 (3)	0.053 (2)	0.008 (2)	0.003 (2)	0.0171 (19)
C15	0.0415 (17)	0.126 (3)	0.075 (2)	-0.010 (2)	-0.0164 (17)	0.026 (2)

Geometric parameters (Å, °)

O1—C11	1.370 (4)	C6—C7	1.489 (5)
O1—H1O	0.94 (4)	C6—H6A	0.9700
O2—C12	1.208 (4)	C6—H6B	0.9700
O3—C12	1.343 (4)	C7—C8	1.319 (4)
O3—C11	1.483 (4)	C7—H7	0.9300
C1—C10	1.526 (4)	C8—C12	1.473 (4)
C1—C2	1.524 (5)	C8—C9	1.489 (4)
C1—H1A	0.9700	C9—C11	1.524 (4)
C1—H1B	0.9700	C9—C10	1.542 (4)
C2—C3	1.515 (5)	C9—H9	0.9800
C2—H2A	0.9700	C10—C15	1.531 (4)
C2—H2B	0.9700	C11—H11	0.9800
C3—C4	1.521 (5)	C13—H13A	0.9600
C3—H3A	0.9700	C13—H13B	0.9600
C3—H3B	0.9700	C13—H13C	0.9600
C4—C14	1.538 (5)	C14—H14A	0.9600
C4—C13	1.542 (5)	C14—H14B	0.9600
C4—C5	1.561 (4)	C14—H14C	0.9600
C5—C6	1.530 (4)	C15—H15A	0.9600
C5—C10	1.554 (4)	C15—H15B	0.9600
C5—H5	0.9800	C15—H15C	0.9600

C11—O1—H1O	104 (3)	C7—C8—C9	125.0 (3)
C12—O3—C11	110.6 (2)	C12—C8—C9	107.5 (3)
C10—C1—C2	114.1 (3)	C8—C9—C11	101.3 (2)
C10—C1—H1A	108.7	C8—C9—C10	112.6 (2)
C2—C1—H1A	108.7	C11—C9—C10	119.6 (2)
C10—C1—H1B	108.7	C8—C9—H9	107.6
C2—C1—H1B	108.7	C11—C9—H9	107.6
H1A—C1—H1B	107.6	C10—C9—H9	107.6
C3—C2—C1	109.6 (3)	C1—C10—C15	110.7 (3)
C3—C2—H2A	109.8	C1—C10—C9	108.6 (2)
C1—C2—H2A	109.8	C15—C10—C9	109.6 (2)
C3—C2—H2B	109.8	C1—C10—C5	109.8 (2)
C1—C2—H2B	109.8	C15—C10—C5	112.9 (3)
H2A—C2—H2B	108.2	C9—C10—C5	105.0 (2)
C2—C3—C4	114.5 (3)	O1—C11—O3	109.1 (2)
C2—C3—H3A	108.6	O1—C11—C9	113.1 (3)
C4—C3—H3A	108.6	O3—C11—C9	104.1 (2)
C2—C3—H3B	108.6	O1—C11—H11	110.2
C4—C3—H3B	108.6	O3—C11—H11	110.2
H3A—C3—H3B	107.6	C9—C11—H11	110.2
C3—C4—C14	107.7 (3)	O2—C12—O3	121.7 (3)
C3—C4—C13	109.8 (3)	O2—C12—C8	129.9 (3)
C14—C4—C13	106.7 (3)	O3—C12—C8	108.3 (3)
C3—C4—C5	108.3 (3)	C4—C13—H13A	109.5
C14—C4—C5	109.1 (3)	C4—C13—H13B	109.5
C13—C4—C5	115.0 (3)	H13A—C13—H13B	109.5
C6—C5—C10	111.7 (2)	C4—C13—H13C	109.5
C6—C5—C4	113.2 (2)	H13A—C13—H13C	109.5
C10—C5—C4	116.6 (2)	H13B—C13—H13C	109.5
C6—C5—H5	104.6	C4—C14—H14A	109.5
C10—C5—H5	104.6	C4—C14—H14B	109.5
C4—C5—H5	104.6	H14A—C14—H14B	109.5
C7—C6—C5	112.9 (3)	C4—C14—H14C	109.5
C7—C6—H6A	109.0	H14A—C14—H14C	109.5
C5—C6—H6A	109.0	H14B—C14—H14C	109.5
C7—C6—H6B	109.0	C10—C15—H15A	109.5
C5—C6—H6B	109.0	C10—C15—H15B	109.5
H6A—C6—H6B	107.8	H15A—C15—H15B	109.5
C8—C7—C6	121.4 (3)	C10—C15—H15C	109.5
C8—C7—H7	119.3	H15A—C15—H15C	109.5
C6—C7—H7	119.3	H15B—C15—H15C	109.5
C7—C8—C12	127.1 (3)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
O1—H1O···O2 ⁱ	0.94 (4)	1.89 (5)	2.832 (4)	176 (4)

C11—H11 \cdots O3 ⁱ	0.98	2.40	3.190 (4)	137
C6—H6B \cdots O1 ⁱⁱ	0.97	2.67	3.630 (4)	172

Symmetry codes: (i) $x+1/2, -y+3/2, -z+1$; (ii) $-x, y+1/2, -z+1/2$.