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Two drimane lactones, Valdiviolide and 11-epivaldiviolide, in the form of a (1:1) co-crystal obtained from *Drimys winteri* extracts.

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1 **Two drimane lactones, *Valdiviolide* and *11-epivaldiviolide*, in the form of a (1:1)**
 2 **co-crystal obtained from *Drimys winteri* extracts.**

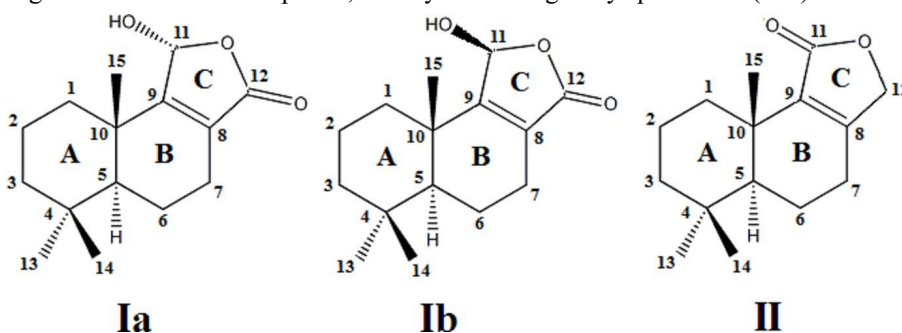
3 **Cristian Paz Robles,^{a*} Darío Mercado,^a Sebastián Suarez^b and Ricardo Baggio^c**

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9 **Abstract**

10 The species herein presented, formulated as C₁₅H₂₂O₃ (**I**) was obtained from *Drimys winteri*, and appears as a co-
 11 crystallization of two isomeric drimane sesquiterpene lactones, *valdiviolide* (**Ia**) and *11-epivaldiviolide* (**Ib**), none of
 12 which had been individually reported in crystal form. Both isomers present three quiral centres at sites 5, 10 and 11, with
 13 a (SSR)/(SSS) sequence in (**Ia/Ib**), respectively. O—H···O bonds bind molecules into chains running along [1̄20], in turn
 14 linked by π···π stacking interactions to define planar, weakly interacting arrays parallel to (001).



15 **1. Introduction**

16 The folk medicinal plant *Drimys winteri* (Winteraceae) is a slender tree native to the Magellanic and Valdivian temperate
 17 rain forests of Chile (where it is locally called "Canelo"). The tree's barks are rich in drimane sesquiterpenoids as
 18 secondary metabolites, some of which present intense pungent and potent antifeedant, antimicrobial, plant growth
 19 inhibitory, cytotoxic and piscicidal activities. A paradigmatic example of these multi-functional sesquiterpenoids is
 20 *polygodial* (Kubo *et al.*, 2005; Jansen & de Groot, 2004). It is perhaps worth mentioning that these properties of Canelo
 21 barks are not new, and they had been known for long by the native Araucanean people who used them in their ancient
 22 medicinal rituals.

23 Following a well established research line in our laboratory, focused on the study of natural products from the Southern
 24 Andean flora we succeeded in extracting from *Drimys winteri* barks a compound of general formulation C₁₅ H₂₂ O₃ (**I**). To
 25 our surprise, upon crystallization (followed by its structure resolution) the solid showed to consist of a single phase
 26 lodging two molecules of identical formula but diverse stereochemistry, *viz.*, *valdiviolide* and *11-epivaldiviolide*
 27 (**Scheme**).

28 *Valdiviolide* (**Ia**) is a drimane sesquiterpene mostly found in a variety of south American plants. It was originally
 29 extracted from *Drimys winteri* by Appel *et al.*, 1963, and subsequently the subject of a large amount of synthetic work
 30 (Ley & Mahon, 1983; Nakano *et al.*, 1998, *etc.*), from which its absolute structure could be envisaged.

31 For its isomer *11-epivaldiviolide* (**Ib**), on the other hand, we could not trace reliable reports of its extraction from
32 botanical sources; it has, however, been found as a metabolite of marine organisms such as the Japanese *nudibranch*
33 *Dendrodoris carbunculosa*, (Sakio *et al.*, 2001)

34 In spite of both species being already known for some time they have not been ever reported in crystal form, either in
35 isolation or mixed up as in the present compound **I**, where they are found co-crystallizing in an orderly fashion, in the
36 triclinic space group P1.

37 Thus, in what follows we present the structure of the *valdiviolide: 11-epivaldiviolide* co-crystal (**I**).

38 2. Experimental

39 2.1. Extraction, purification and crystallization

40 Compound **I** was isolated from the stem bark of *Drimys winteri* (Canelo) collected in Concepcion, VIII Region of Chile
41 in February 2012. 1 kg of barks was powdered and extracted by maceration with ethanol for 3 days, giving 20 g of crude
42 which was further purified by column chromatography. It afforded as a yellow oil from hexane/ethyl acetate 4:1 and as a
43 white solid from hexane/ethyl acetate 1:1. This solid was recrystallized from methanol at 4°C producing colourless
44 crystals, suitable for X-ray diffraction analysis.

45 2.2. Refinement

46 Crystal data, data collection and structure refinement details are summarized in Table 1. All H atoms were identified in
47 an intermediate difference map, and treated differently in refinement: C—H's were idealized and allowed to ride both in
48 coordinates as in displacement factors, the latter taken as $U(H) = x U_{equiv}(C)$, with C—H = 0.93 Å, $x=1.2$ for aromatic, C
49 —H = 0.97 Å, $x=1.2$ for methylene and C—H = 0.96 Å, $x=1.5$ for methyl groups. The H atoms attached to O were refined
50 freely.

51 The use of Mo Ka radiation for data collection precluded a trustable determination of the absolute structure from
52 diffraction data alone (Flack parameters: 0.2 (6)/0.8 (6) for the reported/inverted configurations, respectively). The
53 present "handedness", however, defined by C5(S/S), C10(S/S), C11(R/S) for **Ia/Ib**, respectively, was found to coincide with
54 the ones reported in the literature.

55 3. Results and discussion

56 Fig. 1 shows an ellipsoid plot of the asymmetric unit of **I**, where the two molecules in the co-crystal (*valdiviolide*, **Ia**, and
57 *11-epivaldiviolide*, **Ib**) are identified by their trailing labels A and B. They are almost identical except by the different
58 configuration at site 11 (*R* in **Ia**, *S* in **Ib**), and the similarities can be disclosed in Fig 2, where a superposition of both
59 moieties is presented.

60 The characteristic rigid backbone of this family of compounds is made out of three fused rings (See Scheme labeling),
61 where lateral rings **A** (C1—C5,C10) have a chair conformation (Cremer & Pople, 1975) where $\theta = 6.4$ (5)/3.2 (4)° for
62 **Ia/Ib**, respectively; ideal chair: $\theta = 0.00^\circ$, (Boeyens, 1978); the central **B** ring (C5—C10) presents a quasi envelope
63 conformation [$\theta = 49.1$ (4)/50.6 (3)°, $\varphi = 9.6$ (4)/8.5 (5)°; ideal envelope: $\theta = 54.7^\circ$; $\varphi = 0^\circ$, (Boeyens, 1978)] and the
64 five membered lactone ring **C** (C8,C9,C11,C12,O3) a nearly planar conformation [mean torsion: 0.9 (3)/1.9 (4)°] a direct
65 consequence of the "inner" location of the C8=C9 double bond; when the double bond lays outside the lactone ring, *viz.*,
66 C7=C8 (as in *Dendrocarbin A*, Paz *et al.*, 2014, and related structures discussed therein) the lactone ring is no longer
67 aromatic and adopts an envelope conformation. Another common feature commented by Paz *et al.* consists in the
68 concentration of electronic density (with the concomitant bond contraction) in the C11/12-O3 bonds neighbour to the
69 carbonyl group, irrespective of its position (either 11 or 12) in the lactone ring. This peculiarity is also found in the two

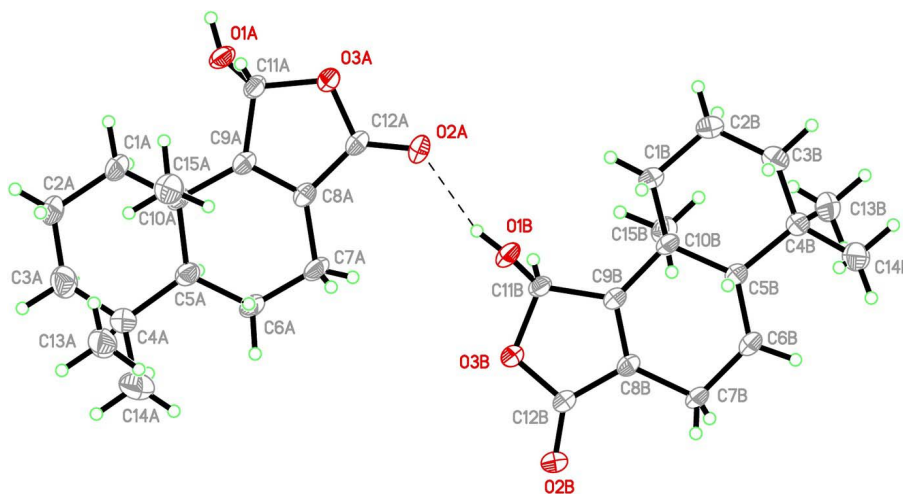
70 structures herein reported, as well as in *Isodrimenin* (Escobar & Wittke, 1988), CSD code FUXPOL, (**II**), Scheme 2), the
 71 only single structure found in the CSD (Version 5.34; Allen, 2002) sharing the same nucleus and double bond disposition,
 72 where the difference resides in the carbonyl C12=O2 group replaced by a methylene C12—H₂, and the hydroxy C11—
 73 OH by a carboxy C11=O3 one (Scheme). Table 2 provides a comparison of corresponding parameters displaying the
 74 differences in bond length between all three structures. As mentioned, the most relevant ones are found around the
 75 lactone oxygen O3, and have to do precisely with the position of the carbonyl group, C12=O2 in **Ia**, **Ib** and C11=O1 in
 76 **II**. In all cases the C=O presents a clear resonance with the neighbouring C12—O3 (C11—O3), which is sensibly shorter
 77 than its C11—O3 (C12—O3) neighbour (See Table 2).

78 Regarding the supramolecular structure, there are two significant intermolecular H-bonds in **I**, involving the hydroxyl
 79 groups as donors and the carbonyl ones as acceptors (Table 3). These H-bonds connect neighbouring molecules of
 80 different kind, in a **Ia**⋯**Ib**⋯**Ia**⋯**Ib** sequence, forming C(6) chains (see Bernstein *et al.*, 1995, for Graph set notation) along
 81 the $[\bar{1}20]$ direction, in patterns much resembling a "frustrated" 2₁ axis (Fig 3a) Incidentally, an eventually exact 2₁
 82 sequence would be impossible due to the different configuration of both molecules. Such a 2₁ pattern, however, is usual
 83 in related compounds crystallizing in chiral space groups having 2₁ axis (*P*2₁, *P*2₁2₁2₁), where the chain appears truly
 84 threaded along the real screw (E.g., the already mentioned *Dendocarbin A*).

85 These $[\bar{1}20]$ chains are in turn linked by π ⋯ π bonds connecting lactone rings of opposite types (Table 4, Fig 3 b) nearly
 86 along $[100]$. The result is the formation of planar arrays parallel to (001). Fig 4 presents two packing views of **I**: Fig 4a, a
 87 projection along $[100]$, showing the way in which chains (running from top to bottom in the Figure) overlap, bound by
 88 the lactone⋯lactone stacking interaction. Note the way in which molecules type A and B alternate along the chain. They
 89 also alternate along the direction of the stacking interaction, even if adjacent chains are related by full cell translations;
 90 the explanation is given by the slanted direction of the $[\bar{1}20]$ chains with reference to the unit cell axis.

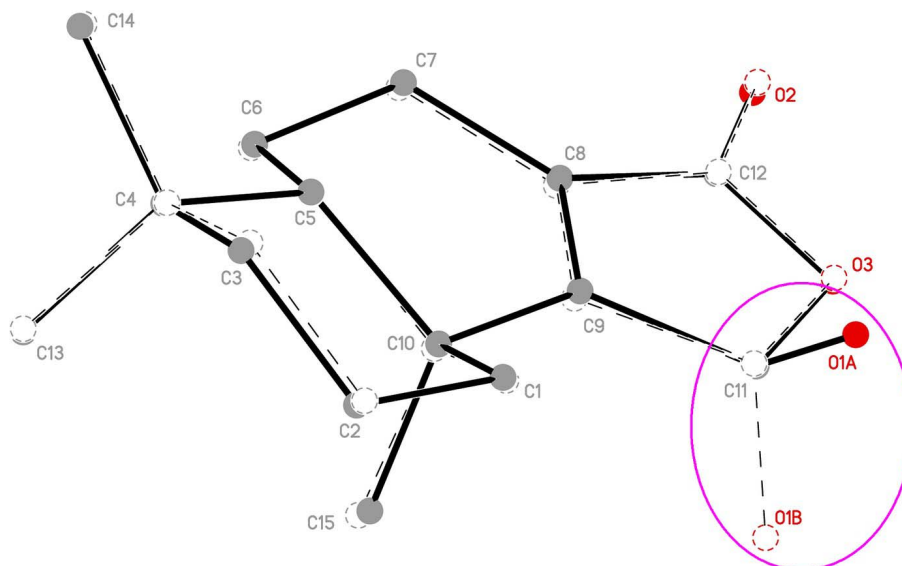
91 Fig 4 b, in turn, shows a view of the planes along $[001]$, where the $[\bar{1}20]$ direction of the H-bonded chains is clearly
 92 perceived. From inspection of Fig 4a it is also apparent that the interactive (hydrophilic) part of the molecules concentrate
 93 at $c \sim 0.50$; the cell edges ($c \sim 0.00, 1.00$) lodge instead the barely interactive hydrophobic parts, which face each other in
 94 the crystal packing, with what there are almost no interactions between adjacent planes.

fig1.tif

95 **Figure 1**

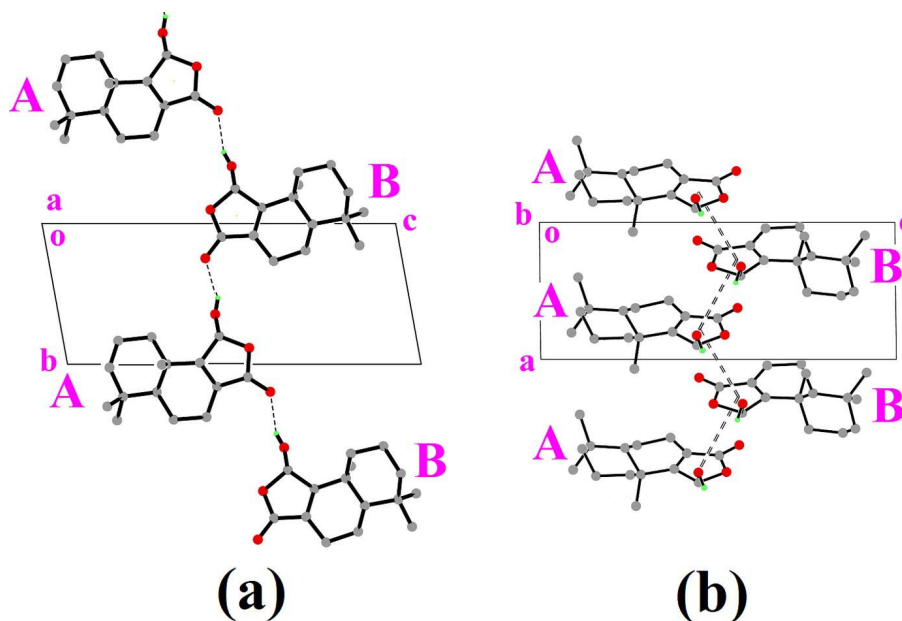
96 Ellipsoid plots of both molecules (**Ia**, **Ib**) in the asymmetric unit, drawn at a 30% level. In broken lines one of the
 97 intrachain H-bonds.

fig2.tif



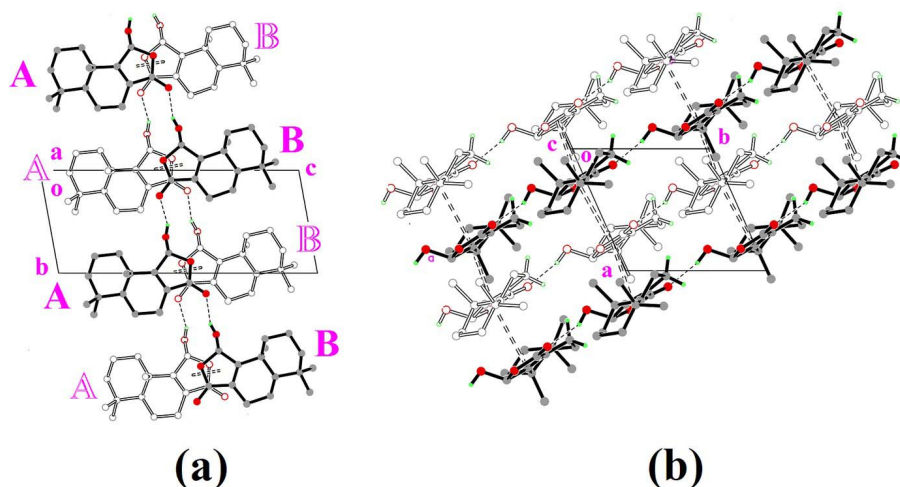
98 **Figure 2**
 99 A schematic view of the L-S. fit of **Ia** (in full lines) and **Ib** (in broken lines). Highlighted, the conformational difference
 100 between both molecules.

fig3.tif



101 **Figure 3**
 102 Intermolecular interactions described in Tables 3 and 4. (a) A single chain built up by the O—H...O bonds. (Table 3) (b)
 103 The stacking interaction linking lactone rings (Table 4).

fig4.tif



104 **Figure 4**
 105 Different views of the (001) planes (a) Projected down [100]. (b) Projected down [001]. Chains are differentiated by their
 106 line blackening.

107 **Table 1**

108 Experimental details

109 Crystal data

110	Chemical formula	$C_{30}H_{44}O_6$
111	M_r	500.65
112	Crystal system, space group	Triclinic, $P1$
113	Temperature (K)	295
114	a, b, c (Å)	6.6766 (4), 7.1292 (4), 16.0995 (10)
115	α, β, γ (°)	78.423 (5), 84.663 (5), 65.460 (5)
116	V (Å ³)	682.88 (7)
117	Z	1
118	Radiation type	Mo $K\alpha$
119	μ (mm ⁻¹)	0.08
120	Crystal size (mm)	0.30 × 0.22 × 0.22
121		
122	Data collection	
123	Diffractometer	Oxford Diffraction Gemini CCD S Ultra diffractometer
124	Absorption correction	Multi-scan <i>CrysAlis PRO</i> (Oxford Diffraction, 2009)
125	T_{\min}, T_{\max}	0.96, 0.97
126	No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	21131, 6463, 4929
127	R_{int}	0.053
128	$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)	0.688
129		
130	Refinement	
131	$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.049, 0.130, 1.01

132	No. of reflections	6463
133	No. of parameters	339
134	No. of restraints	7
135	H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
136	$\Delta\rho_{\max}, \Delta\rho_{\min}$ (e Å ⁻³)	0.24, -0.17

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

139 **Table 2**

140 Comparative Table for corresponding parameters in Ia, Ib and II (Å).

141	Bond	Ia*	Ib*	II**
142	C8-C9	1.342 (4)	1.333 (4)	1.318 (5)
143	C8-C7	1.469 (4)	1.489 (4)	1.487 (5)
144	C11-O1	1.380 (4)	1.380 (4)	1.203 (4)
145	C11-O3	1.460 (4)	1.470 (4)	1.367 (5)
146	C11-C9	1.503 (4)	1.499 (4)	1.475 (5)
147	C12-O2	1.221 (4)	1.217 (4)	—
148	C12-O3	1.356 (3)	1.353 (3)	1.446 (5)
149	C12-C8	1.453 (4)	1.459 (4)	1.488 (5)

150 (*) This work; (**) Escobar and Wittke, 1988.

151 **Table 3**

152 Hydrogen-bond geometry (Å, °)

153	<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>
154	O1B—H1OB \cdots O2A	0.865 (14)	1.90 (2)	2.730 (3)	159 (5)
155	O1A—H1OA \cdots O2B ⁱ	0.849 (14)	1.92 (2)	2.729 (3)	159 (5)

156 Symmetry code: (i) $x+1, y-2, z$.

157 **Table 4**

158 Table 4. $\pi\cdots\pi$ contacts in (I) (Å, °)

159	Group 1 \cdots Group 2	ccd(Å)	da(°)	ipd(Å)
160	<i>Cg</i> 1— <i>Cg</i> 2 ⁱⁱ	3.763 (2)	2.46 (19)	3.28 (5)
161	<i>Cg</i> 1— <i>Cg</i> 2 ⁱⁱⁱ	3.722 (2)	2.46 (19)	3.28 (5)

162 Symmetry Codes. (ii) $x, -1 + y, z$ (iii) $1 + x, -1 + y, z$ Ring Codes. *Cg*1: O3A, C8A, C9A, C11A, C12A; *Cg*2: O3B, C8B, C9B, C11B, C12B ccd: center-to-center distance; da: dihedral angle between rings; ipd: interplanar distance, or (mean) distance from one plane to the neighbouring centroid. For details, see Janiak (2000).

163 **Acknowledgements**

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

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Ellipsoid plot

checkCIF/PLATON results

No syntax errors found. CIF dictionary Interpreting this report

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Bond precision: C-C = 0.0047 A Wavelength=0.71069
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 alpha=78.423(5) beta=84.663(5) gamma=65.460(5)
Temperature: 295 K

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Hall group	P 1	P 1
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Sum formula	C15 H22 O3	C30 H44 O6
Mr	250.33	500.65
Dx, g cm-3	1.217	1.217
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Mu (mm-1)	0.083	0.083
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Theta(max)= 29.258

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S = 1.009 Npar= 339



Alert level C

PLAT340_ALERT_3_C Low Bond Precision on C-C Bonds	0.0047 Ang.
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PLAT915_ALERT_3_C Low Friedel Pair Coverage	83 %



Alert level G

PLAT002_ALERT_2_G Number of Distance or Angle Restraints on AtSite	6 Note
PLAT042_ALERT_1_G Calc. and Reported MoietyFormula Strings Differ	Please Check
PLAT045_ALERT_1_G Calculated and Reported Z Differ by	2.00 Ratio
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PLAT720_ALERT_4_G Number of Unusual/Non-Standard Labels	24 Note

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PLAT791_ALERT_4_G	The Model has Chirality at C10B	S Verify
PLAT791_ALERT_4_G	The Model has Chirality at C11A	R Verify
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PLAT860_ALERT_3_G	Number of Least-Squares Restraints	7 Note
PLAT912_ALERT_4_G	Missing # of FCF Reflections Above STh/L= 0.600		305 Note

0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
3 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
14 **ALERT level G** = General information/check it is not something unexpected

3 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
1 ALERT type 2 Indicator that the structure model may be wrong or deficient
4 ALERT type 3 Indicator that the structure quality may be low
9 ALERT type 4 Improvement, methodology, query or suggestion
0 ALERT type 5 Informative message, check

database duplication summary

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- *R* factor =
- Space group =
- Formula =
- a= b= c=
- alpha= beta= gamma=

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reference checking results

The following reference was not checked in detail as it was not recognized as a journal reference

Oxford Diffraction (2009). *CrysAlis PRO*, version 171.33.48. Oxford Diffraction Ltd, Abingdon, Oxfordshire, England.

The following references may be incorrectly formatted

Cremer, D. & Pople, J. A. (1975). *J. Amer. Chem. Soc.*, 97,1354-1358.
[*Unrecognized journal title.*]

Ley, S. V. & Mahon, M. (1983). *J. Chem. Soc., Perkin Trans. 1*, 1983, 1379-1381.
[*Unrecognized journal title.*]

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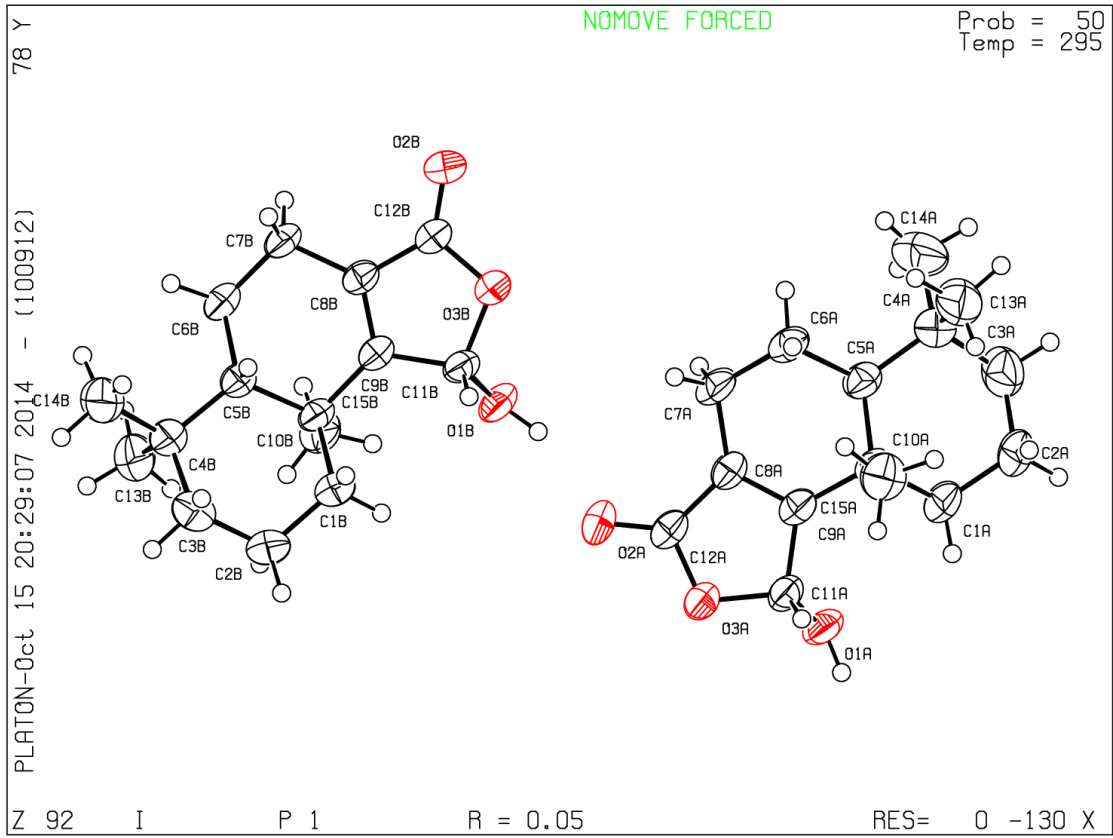
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1 supporting information

2 **Two drimane lactones, Valdiviolide and 11-epivaldiviolide, in the form of a (1:1)**
3 **co-crystal obtained from *Drimys winteri* extracts.**4 **Cristian Paz Robles,* Darío Mercado, Sebastián Suarez and Ricardo Baggio**5 **Computing details**6 Data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*;
7 program(s) used to solve structure: *SHELXS* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013*
8 (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008); software used to prepare material for
9 publication: *SHELXL2013*, *PLATON* (Spek, 2009).10 **(I)**11 *Crystal data*

12	$C_{30}H_{44}O_6$	$Z = 1$
13	$M_r = 500.65$	$F(000) = 272$
14	Triclinic, <i>P1</i>	$D_x = 1.217 \text{ Mg m}^{-3}$
15	$a = 6.6766 (4) \text{ \AA}$	Mo $K\alpha$ radiation, $\lambda = 0.71069 \text{ \AA}$
16	$b = 7.1292 (4) \text{ \AA}$	Cell parameters from 3755 reflections
17	$c = 16.0995 (10) \text{ \AA}$	$\theta = 3.7\text{--}22.1^\circ$
18	$\alpha = 78.423 (5)^\circ$	$\mu = 0.08 \text{ mm}^{-1}$
19	$\beta = 84.663 (5)^\circ$	$T = 295 \text{ K}$
20	$\gamma = 65.460 (5)^\circ$	Blocks, colourless
21	$V = 682.88 (7) \text{ \AA}^3$	$0.30 \times 0.22 \times 0.22 \text{ mm}$

22 *Data collection*

23	Oxford Diffraction Gemini CCD S Ultra	6463 independent reflections
	diffractometer	4929 reflections with $I > 2\sigma(I)$
24	ω scans, thick slices	$R_{\text{int}} = 0.053$
25	Absorption correction: multi-scan	$\theta_{\text{max}} = 29.3^\circ$, $\theta_{\text{min}} = 3.7^\circ$
	<i>CrysAlis PRO</i> (Oxford Diffraction, 2009)	$h = -9 \rightarrow 8$
26	$T_{\text{min}} = 0.96$, $T_{\text{max}} = 0.97$	$k = -9 \rightarrow 9$
27	21131 measured reflections	$l = -20 \rightarrow 21$

28 *Refinement*

29	Refinement on F^2	Hydrogen site location: mixed
30	Least-squares matrix: full	H atoms treated by a mixture of independent
31	$R[F^2 > 2\sigma(F^2)] = 0.049$	and constrained refinement
32	$wR(F^2) = 0.130$	$w = 1/[\sigma^2(F_o^2) + (0.0657P)^2 + 0.0278P]$
33	$S = 1.01$	where $P = (F_o^2 + 2F_c^2)/3$
34	6463 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
35	339 parameters	$\Delta\rho_{\text{max}} = 0.24 \text{ e \AA}^{-3}$
36	7 restraints	$\Delta\rho_{\text{min}} = -0.17 \text{ e \AA}^{-3}$

37 *Special details*

38 **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.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	
41	C1A	0.8421 (7)	-0.1903 (5)	0.2474 (2)	0.0595 (9)
42	H1AA	0.9698	-0.3132	0.2705	0.071*
43	H1AB	0.7116	-0.2126	0.2688	0.071*
44	C2A	0.8491 (8)	-0.1639 (5)	0.1512 (2)	0.0696 (11)
45	H2AA	0.9829	-0.1483	0.1297	0.084*
46	H2AB	0.8504	-0.2879	0.1346	0.084*
47	C3A	0.6514 (8)	0.0263 (6)	0.1133 (3)	0.0703 (10)
48	H3AA	0.5192	0.0047	0.1324	0.084*
49	H3AB	0.6598	0.0388	0.0521	0.084*
50	C4A	0.6308 (6)	0.2334 (5)	0.1361 (2)	0.0584 (9)
51	C5A	0.6450 (5)	0.2018 (4)	0.2338 (2)	0.0453 (7)
52	H5AA	0.5128	0.1799	0.2553	0.054*
53	C6A	0.6246 (6)	0.3961 (4)	0.2678 (2)	0.0552 (8)
54	H6AA	0.7623	0.4129	0.2579	0.066*
55	H6AB	0.5103	0.5198	0.2371	0.066*
56	C7A	0.5697 (6)	0.3783 (4)	0.3617 (2)	0.0534 (8)
57	H7AA	0.6128	0.4708	0.3853	0.064*
58	H7AB	0.4119	0.4225	0.3697	0.064*
59	C8A	0.6831 (5)	0.1623 (4)	0.40701 (19)	0.0416 (7)
60	C9A	0.7984 (5)	-0.0069 (4)	0.3714 (2)	0.0463 (7)
61	C10A	0.8393 (5)	0.0028 (4)	0.2773 (2)	0.0420 (7)
62	C11A	0.8961 (5)	-0.1959 (4)	0.43973 (19)	0.0428 (6)
63	H11A	1.0572	-0.2527	0.4360	0.051*
64	C12A	0.6990 (5)	0.0963 (4)	0.4984 (2)	0.0427 (7)
65	C13A	0.8045 (8)	0.3023 (6)	0.0870 (3)	0.0751 (11)
66	H13A	0.9482	0.1918	0.0982	0.113*
67	H13B	0.7978	0.4256	0.1047	0.113*
68	H13C	0.7759	0.3327	0.0273	0.113*
69	C14A	0.4012 (8)	0.4007 (8)	0.1074 (3)	0.0957 (15)
70	H14A	0.3915	0.5365	0.1119	0.144*
71	H14B	0.2904	0.3711	0.1430	0.144*
72	H14C	0.3788	0.3987	0.0496	0.144*
73	C15A	1.0689 (6)	0.0062 (6)	0.2628 (3)	0.0641 (9)
74	H15A	1.1710	-0.1059	0.3016	0.096*
75	H15B	1.0619	0.1377	0.2719	0.096*
76	H15C	1.1169	-0.0110	0.2056	0.096*
77	O1A	0.8250 (4)	-0.3516 (3)	0.43921 (15)	0.0510 (5)
78	O2A	0.6240 (4)	0.1988 (3)	0.55499 (15)	0.0566 (6)
79	O3A	0.8205 (3)	-0.1130 (3)	0.51828 (14)	0.0474 (5)

80	C1B	0.5087 (6)	0.6373 (5)	0.7510 (2)	0.0550 (8)
81	H1BA	0.5265	0.5127	0.7301	0.066*
82	H1BB	0.6234	0.6810	0.7253	0.066*
83	C2B	0.5350 (7)	0.5847 (6)	0.8471 (2)	0.0673 (10)
84	H2BA	0.6805	0.4762	0.8614	0.081*
85	H2BB	0.4272	0.5315	0.8728	0.081*
86	C3B	0.5037 (7)	0.7788 (6)	0.8823 (2)	0.0665 (10)
87	H3BA	0.6188	0.8246	0.8594	0.080*
88	H3BB	0.5202	0.7411	0.9434	0.080*
89	C4B	0.2785 (6)	0.9624 (5)	0.8616 (2)	0.0529 (8)
90	C5B	0.2462 (5)	1.0063 (4)	0.76401 (19)	0.0414 (6)
91	H5BA	0.3637	1.0497	0.7396	0.050*
92	C6B	0.0321 (5)	1.1914 (4)	0.7315 (2)	0.0495 (7)
93	H6BA	-0.0897	1.1492	0.7441	0.059*
94	H6BB	0.0048	1.3059	0.7610	0.059*
95	C7B	0.0426 (5)	1.2675 (4)	0.6360 (2)	0.0470 (7)
96	H7BA	-0.1054	1.3408	0.6136	0.056*
97	H7BB	0.1135	1.3645	0.6253	0.056*
98	C8B	0.1685 (4)	1.0862 (4)	0.59250 (18)	0.0382 (6)
99	C9B	0.2757 (4)	0.8877 (4)	0.62997 (18)	0.0370 (6)
100	C10B	0.2828 (5)	0.8123 (4)	0.72441 (19)	0.0392 (6)
101	C11B	0.3888 (5)	0.7548 (4)	0.56421 (19)	0.0398 (6)
102	H11B	0.5488	0.6978	0.5706	0.048*
103	C12B	0.1989 (4)	1.0967 (4)	0.50102 (19)	0.0393 (6)
104	C13B	0.0953 (7)	0.9203 (7)	0.9151 (3)	0.0733 (11)
105	H13D	0.0999	0.7877	0.9085	0.110*
106	H13E	-0.0448	1.0293	0.8967	0.110*
107	H13F	0.1156	0.9175	0.9737	0.110*
108	C14B	0.2907 (9)	1.1561 (6)	0.8864 (3)	0.0855 (15)
109	H14D	0.1500	1.2725	0.8776	0.128*
110	H14E	0.4000	1.1908	0.8519	0.128*
111	H14F	0.3296	1.1250	0.9450	0.128*
112	C15B	0.1025 (6)	0.7270 (5)	0.7448 (2)	0.0556 (8)
113	H15D	0.1247	0.6249	0.7101	0.083*
114	H15E	-0.0396	0.8402	0.7335	0.083*
115	H15F	0.1101	0.6628	0.8035	0.083*
116	O1B	0.3228 (4)	0.5946 (3)	0.56564 (15)	0.0507 (5)
117	O2B	0.1319 (4)	1.2476 (3)	0.44398 (14)	0.0533 (6)
118	O3B	0.3231 (3)	0.9033 (3)	0.48363 (13)	0.0462 (5)
119	H1OB	0.415 (3)	0.481 (4)	0.549 (3)	0.103 (16)*
120	H1OA	0.909 (3)	-0.480 (2)	0.453 (3)	0.119 (19)*

121 *Atomic displacement parameters (\AA^2)*

122		U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
123	C1A	0.084 (3)	0.0356 (16)	0.060 (2)	-0.0222 (17)	0.0026 (17)	-0.0166 (14)
124	C2A	0.106 (3)	0.0478 (19)	0.061 (2)	-0.031 (2)	0.000 (2)	-0.0251 (17)
125	C3A	0.088 (3)	0.075 (2)	0.062 (2)	-0.041 (2)	-0.008 (2)	-0.0210 (19)

126	C4A	0.068 (2)	0.0492 (18)	0.052 (2)	-0.0189 (16)	-0.0049 (16)	-0.0059 (15)
127	C5A	0.0492 (17)	0.0343 (14)	0.0517 (18)	-0.0172 (13)	0.0021 (13)	-0.0072 (12)
128	C6A	0.073 (2)	0.0278 (14)	0.058 (2)	-0.0152 (14)	0.0042 (16)	-0.0060 (13)
129	C7A	0.067 (2)	0.0256 (13)	0.064 (2)	-0.0145 (13)	0.0019 (16)	-0.0107 (13)
130	C8A	0.0440 (16)	0.0327 (13)	0.0516 (18)	-0.0173 (12)	0.0030 (13)	-0.0130 (12)
131	C9A	0.0511 (18)	0.0322 (14)	0.0523 (18)	-0.0127 (13)	0.0005 (14)	-0.0105 (12)
132	C10A	0.0471 (16)	0.0278 (12)	0.0491 (17)	-0.0122 (11)	0.0012 (13)	-0.0101 (11)
133	C11A	0.0411 (15)	0.0334 (14)	0.0493 (17)	-0.0081 (12)	-0.0009 (12)	-0.0130 (12)
134	C12A	0.0384 (15)	0.0335 (14)	0.0586 (18)	-0.0139 (12)	0.0001 (13)	-0.0155 (13)
135	C13A	0.106 (3)	0.065 (2)	0.058 (2)	-0.042 (2)	0.009 (2)	-0.0062 (18)
136	C14A	0.092 (3)	0.089 (3)	0.075 (3)	-0.008 (3)	-0.025 (3)	0.003 (2)
137	C15A	0.056 (2)	0.069 (2)	0.068 (2)	-0.0249 (18)	0.0052 (17)	-0.0150 (19)
138	O1A	0.0578 (13)	0.0268 (10)	0.0637 (14)	-0.0104 (9)	-0.0078 (11)	-0.0096 (9)
139	O2A	0.0631 (15)	0.0456 (12)	0.0584 (14)	-0.0129 (11)	0.0055 (11)	-0.0265 (11)
140	O3A	0.0487 (12)	0.0370 (11)	0.0505 (13)	-0.0082 (9)	-0.0031 (9)	-0.0143 (9)
141	C1B	0.057 (2)	0.0374 (16)	0.0527 (19)	-0.0016 (14)	-0.0043 (15)	-0.0061 (13)
142	C2B	0.071 (2)	0.0456 (18)	0.058 (2)	0.0031 (16)	-0.0132 (18)	-0.0038 (15)
143	C3B	0.080 (3)	0.060 (2)	0.051 (2)	-0.0184 (19)	-0.0173 (18)	-0.0046 (16)
144	C4B	0.070 (2)	0.0390 (16)	0.0461 (18)	-0.0177 (15)	-0.0071 (15)	-0.0073 (13)
145	C5B	0.0487 (16)	0.0317 (13)	0.0441 (16)	-0.0163 (12)	-0.0022 (13)	-0.0068 (11)
146	C6B	0.0554 (19)	0.0309 (14)	0.0562 (19)	-0.0087 (13)	-0.0012 (14)	-0.0145 (13)
147	C7B	0.0554 (18)	0.0255 (13)	0.0544 (18)	-0.0084 (12)	-0.0053 (14)	-0.0107 (12)
148	C8B	0.0396 (15)	0.0293 (13)	0.0476 (16)	-0.0150 (11)	-0.0016 (12)	-0.0079 (11)
149	C9B	0.0352 (14)	0.0289 (13)	0.0471 (16)	-0.0125 (11)	0.0015 (11)	-0.0090 (11)
150	C10B	0.0402 (15)	0.0259 (12)	0.0467 (16)	-0.0092 (11)	0.0011 (12)	-0.0064 (11)
151	C11B	0.0372 (14)	0.0290 (12)	0.0460 (16)	-0.0067 (11)	-0.0006 (12)	-0.0062 (11)
152	C12B	0.0363 (15)	0.0309 (13)	0.0484 (16)	-0.0108 (11)	-0.0003 (12)	-0.0088 (12)
153	C13B	0.091 (3)	0.067 (2)	0.052 (2)	-0.023 (2)	0.0132 (19)	-0.0156 (17)
154	C14B	0.136 (4)	0.058 (2)	0.066 (3)	-0.035 (3)	-0.034 (3)	-0.0147 (19)
155	C15B	0.071 (2)	0.0441 (17)	0.060 (2)	-0.0321 (16)	0.0083 (16)	-0.0122 (14)
156	O1B	0.0568 (13)	0.0280 (10)	0.0644 (14)	-0.0123 (9)	0.0048 (10)	-0.0157 (9)
157	O2B	0.0630 (15)	0.0343 (11)	0.0513 (13)	-0.0113 (10)	-0.0062 (10)	0.0004 (9)
158	O3B	0.0527 (12)	0.0326 (10)	0.0443 (12)	-0.0083 (9)	0.0008 (9)	-0.0079 (8)

159 *Geometric parameters (Å, °)*

160	C1A—C2A	1.521 (5)	C1B—C2B	1.526 (5)
161	C1A—C10A	1.540 (4)	C1B—C10B	1.535 (4)
162	C1A—H1AA	0.9700	C1B—H1BA	0.9700
163	C1A—H1AB	0.9700	C1B—H1BB	0.9700
164	C2A—C3A	1.511 (6)	C2B—C3B	1.527 (5)
165	C2A—H2AA	0.9700	C2B—H2BA	0.9700
166	C2A—H2AB	0.9700	C2B—H2BB	0.9700
167	C3A—C4A	1.540 (5)	C3B—C4B	1.539 (5)
168	C3A—H3AA	0.9700	C3B—H3BA	0.9700
169	C3A—H3AB	0.9700	C3B—H3BB	0.9700
170	C4A—C13A	1.536 (6)	C4B—C13B	1.527 (6)
171	C4A—C14A	1.542 (6)	C4B—C14B	1.547 (5)

172	C4A—C5A	1.551 (5)	C4B—C5B	1.556 (4)
173	C5A—C6A	1.540 (4)	C5B—C6B	1.534 (4)
174	C5A—C10A	1.558 (4)	C5B—C10B	1.557 (4)
175	C5A—H5AA	0.9800	C5B—H5BA	0.9800
176	C6A—C7A	1.515 (5)	C6B—C7B	1.531 (5)
177	C6A—H6AA	0.9700	C6B—H6BA	0.9700
178	C6A—H6AB	0.9700	C6B—H6BB	0.9700
179	C7A—C8A	1.469 (4)	C7B—C8B	1.489 (4)
180	C7A—H7AA	0.9700	C7B—H7BA	0.9700
181	C7A—H7AB	0.9700	C7B—H7BB	0.9700
182	C8A—C9A	1.342 (4)	C8B—C9B	1.333 (4)
183	C8A—C12A	1.452 (4)	C8B—C12B	1.459 (4)
184	C9A—C11A	1.504 (4)	C9B—C11B	1.499 (4)
185	C9A—C10A	1.506 (4)	C9B—C10B	1.506 (4)
186	C10A—C15A	1.538 (5)	C10B—C15B	1.544 (4)
187	C11A—O1A	1.379 (3)	C11B—O1B	1.380 (3)
188	C11A—O3A	1.462 (4)	C11B—O3B	1.471 (4)
189	C11A—H11A	0.9800	C11B—H11B	0.9800
190	C12A—O2A	1.221 (4)	C12B—O2B	1.217 (3)
191	C12A—O3A	1.356 (3)	C12B—O3B	1.352 (3)
192	C13A—H13A	0.9600	C13B—H13D	0.9600
193	C13A—H13B	0.9600	C13B—H13E	0.9600
194	C13A—H13C	0.9600	C13B—H13F	0.9600
195	C14A—H14A	0.9600	C14B—H14D	0.9600
196	C14A—H14B	0.9600	C14B—H14E	0.9600
197	C14A—H14C	0.9600	C14B—H14F	0.9600
198	C15A—H15A	0.9600	C15B—H15D	0.9600
199	C15A—H15B	0.9600	C15B—H15E	0.9600
200	C15A—H15C	0.9600	C15B—H15F	0.9600
201	O1A—H10A	0.849 (14)	O1B—H10B	0.865 (14)
202				
203	C2A—C1A—C10A	111.7 (3)	C2B—C1B—C10B	111.9 (3)
204	C2A—C1A—H1AA	109.3	C2B—C1B—H1BA	109.2
205	C10A—C1A—H1AA	109.3	C10B—C1B—H1BA	109.2
206	C2A—C1A—H1AB	109.3	C2B—C1B—H1BB	109.2
207	C10A—C1A—H1AB	109.3	C10B—C1B—H1BB	109.2
208	H1AA—C1A—H1AB	107.9	H1BA—C1B—H1BB	107.9
209	C3A—C2A—C1A	110.5 (3)	C1B—C2B—C3B	110.6 (3)
210	C3A—C2A—H2AA	109.5	C1B—C2B—H2BA	109.5
211	C1A—C2A—H2AA	109.5	C3B—C2B—H2BA	109.5
212	C3A—C2A—H2AB	109.5	C1B—C2B—H2BB	109.5
213	C1A—C2A—H2AB	109.5	C3B—C2B—H2BB	109.5
214	H2AA—C2A—H2AB	108.1	H2BA—C2B—H2BB	108.1
215	C2A—C3A—C4A	114.2 (3)	C2B—C3B—C4B	113.6 (3)
216	C2A—C3A—H3AA	108.7	C2B—C3B—H3BA	108.8
217	C4A—C3A—H3AA	108.7	C4B—C3B—H3BA	108.8
218	C2A—C3A—H3AB	108.7	C2B—C3B—H3BB	108.8
219	C4A—C3A—H3AB	108.7	C4B—C3B—H3BB	108.8

220	H3AA—C3A—H3AB	107.6	H3BA—C3B—H3BB	107.7
221	C13A—C4A—C3A	110.0 (3)	C13B—C4B—C3B	111.1 (3)
222	C13A—C4A—C14A	108.1 (3)	C13B—C4B—C14B	107.4 (3)
223	C3A—C4A—C14A	107.2 (4)	C3B—C4B—C14B	106.8 (3)
224	C13A—C4A—C5A	114.3 (3)	C13B—C4B—C5B	115.0 (3)
225	C3A—C4A—C5A	108.0 (3)	C3B—C4B—C5B	107.9 (3)
226	C14A—C4A—C5A	109.1 (3)	C14B—C4B—C5B	108.3 (3)
227	C6A—C5A—C4A	114.8 (2)	C6B—C5B—C10B	110.5 (2)
228	C6A—C5A—C10A	110.4 (2)	C6B—C5B—C4B	115.0 (3)
229	C4A—C5A—C10A	116.6 (2)	C10B—C5B—C4B	115.5 (2)
230	C6A—C5A—H5AA	104.5	C6B—C5B—H5BA	104.8
231	C4A—C5A—H5AA	104.5	C10B—C5B—H5BA	104.8
232	C10A—C5A—H5AA	104.5	C4B—C5B—H5BA	104.8
233	C7A—C6A—C5A	111.8 (2)	C7B—C6B—C5B	111.8 (3)
234	C7A—C6A—H6AA	109.3	C7B—C6B—H6BA	109.3
235	C5A—C6A—H6AA	109.3	C5B—C6B—H6BA	109.3
236	C7A—C6A—H6AB	109.3	C7B—C6B—H6BB	109.3
237	C5A—C6A—H6AB	109.3	C5B—C6B—H6BB	109.3
238	H6AA—C6A—H6AB	107.9	H6BA—C6B—H6BB	107.9
239	C8A—C7A—C6A	111.4 (3)	C8B—C7B—C6B	110.1 (2)
240	C8A—C7A—H7AA	109.4	C8B—C7B—H7BA	109.6
241	C6A—C7A—H7AA	109.4	C6B—C7B—H7BA	109.6
242	C8A—C7A—H7AB	109.4	C8B—C7B—H7BB	109.6
243	C6A—C7A—H7AB	109.4	C6B—C7B—H7BB	109.6
244	H7AA—C7A—H7AB	108.0	H7BA—C7B—H7BB	108.1
245	C9A—C8A—C12A	108.0 (3)	C9B—C8B—C12B	108.1 (2)
246	C9A—C8A—C7A	126.1 (3)	C9B—C8B—C7B	126.2 (3)
247	C12A—C8A—C7A	125.8 (3)	C12B—C8B—C7B	125.6 (2)
248	C8A—C9A—C11A	109.4 (3)	C8B—C9B—C11B	109.7 (3)
249	C8A—C9A—C10A	123.5 (3)	C8B—C9B—C10B	124.3 (3)
250	C11A—C9A—C10A	126.8 (3)	C11B—C9B—C10B	125.9 (2)
251	C9A—C10A—C1A	112.3 (2)	C9B—C10B—C1B	110.8 (2)
252	C9A—C10A—C15A	104.7 (3)	C9B—C10B—C15B	105.9 (2)
253	C1A—C10A—C15A	109.1 (3)	C1B—C10B—C15B	110.0 (3)
254	C9A—C10A—C5A	106.1 (2)	C9B—C10B—C5B	105.3 (2)
255	C1A—C10A—C5A	108.9 (2)	C1B—C10B—C5B	109.3 (2)
256	C15A—C10A—C5A	115.7 (3)	C15B—C10B—C5B	115.5 (2)
257	O1A—C11A—O3A	108.8 (2)	O1B—C11B—O3B	109.5 (2)
258	O1A—C11A—C9A	114.2 (2)	O1B—C11B—C9B	113.4 (2)
259	O3A—C11A—C9A	103.8 (2)	O3B—C11B—C9B	103.6 (2)
260	O1A—C11A—H11A	110.0	O1B—C11B—H11B	110.1
261	O3A—C11A—H11A	110.0	O3B—C11B—H11B	110.1
262	C9A—C11A—H11A	110.0	C9B—C11B—H11B	110.1
263	O2A—C12A—O3A	119.7 (3)	O2B—C12B—O3B	120.5 (3)
264	O2A—C12A—C8A	130.1 (3)	O2B—C12B—C8B	129.6 (3)
265	O3A—C12A—C8A	110.1 (2)	O3B—C12B—C8B	109.9 (2)
266	C4A—C13A—H13A	109.5	C4B—C13B—H13D	109.5
267	C4A—C13A—H13B	109.5	C4B—C13B—H13E	109.5

268	H13A—C13A—H13B	109.5	H13D—C13B—H13E	109.5
269	C4A—C13A—H13C	109.5	C4B—C13B—H13F	109.5
270	H13A—C13A—H13C	109.5	H13D—C13B—H13F	109.5
271	H13B—C13A—H13C	109.5	H13E—C13B—H13F	109.5
272	C4A—C14A—H14A	109.5	C4B—C14B—H14D	109.5
273	C4A—C14A—H14B	109.5	C4B—C14B—H14E	109.5
274	H14A—C14A—H14B	109.5	H14D—C14B—H14E	109.5
275	C4A—C14A—H14C	109.5	C4B—C14B—H14F	109.5
276	H14A—C14A—H14C	109.5	H14D—C14B—H14F	109.5
277	H14B—C14A—H14C	109.5	H14E—C14B—H14F	109.5
278	C10A—C15A—H15A	109.5	C10B—C15B—H15D	109.5
279	C10A—C15A—H15B	109.5	C10B—C15B—H15E	109.5
280	H15A—C15A—H15B	109.5	H15D—C15B—H15E	109.5
281	C10A—C15A—H15C	109.5	C10B—C15B—H15F	109.5
282	H15A—C15A—H15C	109.5	H15D—C15B—H15F	109.5
283	H15B—C15A—H15C	109.5	H15E—C15B—H15F	109.5
284	C11A—O1A—H10A	121.4 (14)	C11B—O1B—H10B	118.5 (14)
285	C12A—O3A—C11A	108.7 (2)	C12B—O3B—C11B	108.6 (2)

286 *Hydrogen-bond geometry (Å, °)*

287	$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
288	O1B—H10B \cdots O2A	0.87 (1)	1.90 (2)	2.730 (3)	159 (5)
289	O1A—H10A \cdots O2B ⁱ	0.85 (1)	1.92 (2)	2.729 (3)	159 (5)

290 Symmetry code: (i) $x+1, y-2, z$.