IUCrData

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(1*R*,2*R*,7*S*,8*R*,9*R*)-*N*-(2-Hydroxy-2,6,6,9-tetramethyl-12-oxatricyclo[7.2.1.0^{1,7}]dodecan-8-yl)-acetamide

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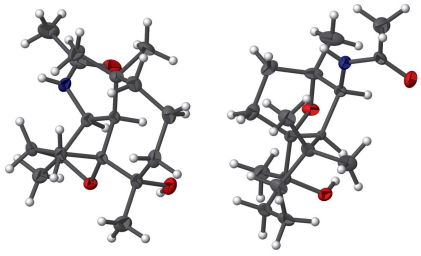
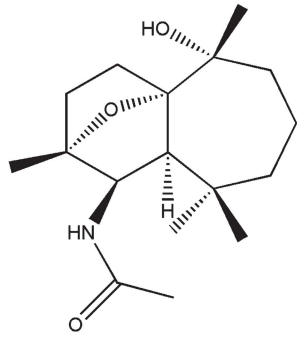
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{17}H_{29}NO_3$, was synthesized in two steps from β-himachalene (2,6,6,9-tetramethylbicyclo[5.4.0^{1,7}]undeca-1,8-diene), which was isolated from an oil of the Atlas cedar (*Cedrus Atlantica*). The asymmetric unit contains two independent molecules (*A* and *B*): *B* features an intramolecular O—H···O hydrogen bond whereas *A* forms an intermolecular O—H···O link to *B*. Each molecule is built up from a seven-membered ring to which a bridged six-membered ring is fused: the cycloheptane rings have twist-chair conformations, while the bridged cyclohexane rings display near-perfect boat conformations. In the crystal, the molecules are linked by N—H···O and O—H···O hydrogen bonds forming helical chains propagating along the *b*-axis direction. Some weak C—H···O interactions are also observed.

3D view



Chemical scheme



Structure description

The bicyclic sesquiterpene β-himachalene is the main constituent of the essential oil of the Atlas cedar (*Cedrus Atlantica*) (El Haib *et al.*, 2010; Loubidi *et al.*, 2014). The reactivity of this sesquiterpene and its derivatives has been studied extensively by our group (Benharref *et al.*, 2017 and references therein) in order to prepare new products with potential biological properties. Indeed, these compounds have been tested for their potential antifungal activity against the phytopathogen *Botrytis cinerea* (Daoubi *et al.*, 2004). Herein, we report on the synthesis and crystal structure of the title compound.

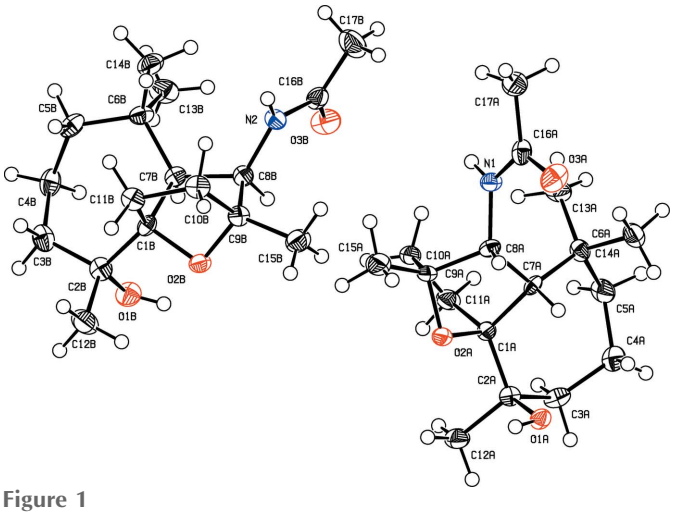


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level.

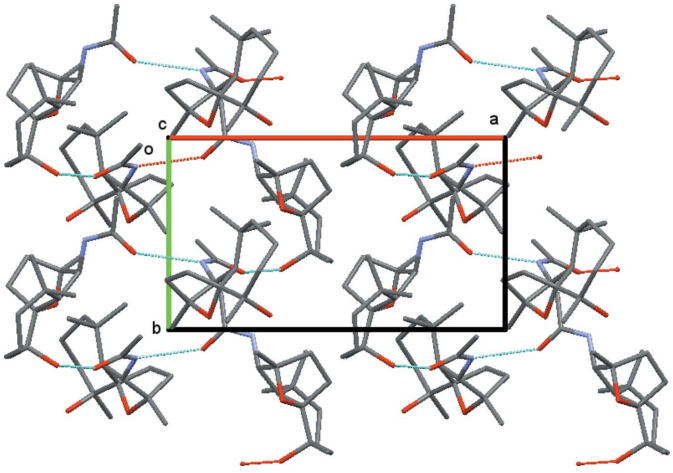


Figure 2
A view along the *c* axis of the crystal packing of the title compound, showing molecules linked by N—H···O hydrogen bonds (dashed lines; see Table 1), forming helical chains along [010]. For clarity, C-bound H atoms have been omitted.

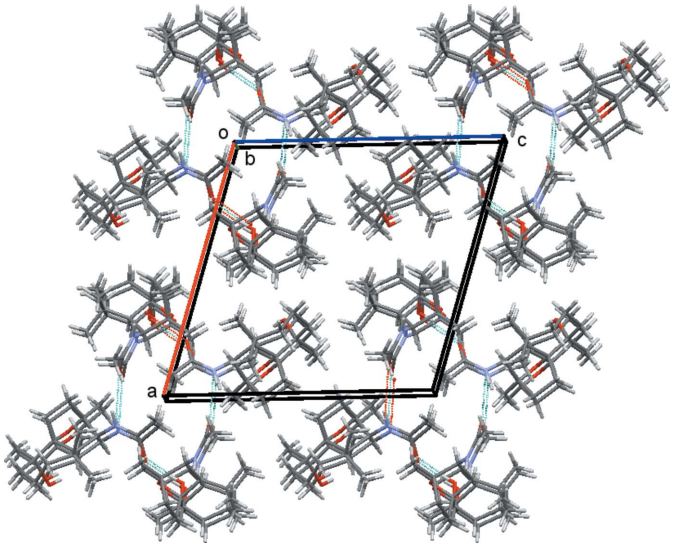


Figure 3
Unit-cell packing of the title compound viewed along [010].

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1*A*^{i}	0.86	2.33	3.084 (4)	147
N2—H2···O3*A*^{ii}	0.86	2.15	2.851 (5)	139
O1*A*—H1*A*···O3*B*^{iii}	0.82	1.90	2.712 (4)	169
O1*B*—H1*B*···O2*B*	0.82	2.32	2.752 (4)	114
C3*A*—H3*A*2···O1*B*	0.97	2.58	3.516 (5)	162
C15*A*—H15*C*···O3*B*^{iv}	0.96	2.43	3.369 (6)	167
C17*A*—H17*C*···O1*A*^{i}	0.96	2.35	3.252 (6)	156

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{17}H_{29}NO_3$
M_r	295.42
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	293
a, *b*, *c* (Å)	14.5077 (10), 7.9149 (4), 14.8297 (8)
β (°)	106.803 (7)
V (Å^3)	1630.15 (17)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.08
Crystal size (mm)	0.50 × 0.25 × 0.12
Data collection	
Diffractometer	Rigaku Oxford Diffraction Xcalibur Eos Gemini ultra
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku Oxford Diffraction, 2015)
T_{min}, T_{max}	0.628, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	16240, 6178, 4204
R_{int}	0.063
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.625
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.055, 0.115, 0.99
No. of reflections	6178
No. of parameters	391
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.20, −0.23

Computer programs: *CrysAlis PRO* (Rigaku Oxford Diffraction, 2015), *SHELXS2014*(-(Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2008).

The asymmetric unit contains two independent molecules having similar, but not identical conformations: they differ by the presence of an intramolecular hydrogen bond in the molecule *B* involving the O1*B* and O2*B* atoms (Table 1). Each molecule is built up from a seven-membered ring, which is fused to a bridged cyclohexane ring as shown in Fig. 1. In both molecules, the cycloheptane rings displays a twist-chair conformation as indicated by the total puckering amplitude Q_T = 0.7876 (5) Å and spherical polar angle θ = 34.56 (3)°, φ_2 = 215.0 (5)° and φ_3 = 13.7 (4)° for molecule *B* and Q_T = 0.809 (4) Å, θ_2 = 38.4 (3)°, φ_2 =112.2 (5)°, φ_3 = 219.3 (4)° for molecule *A*, whereas the cyclohexane rings shows a near-perfect boat conformation with Q_T = 0.952 (5) Å and spherical

polar angle θ = 89.4 (3)° and φ_2 = 7.0 (3)° for molecule *B* and Q_T = 0.937 (5) Å, θ_2 = 89.2 (3)°, φ_2 = 8.0 (3)° for molecule *A*.

The crystal structure features C—H···O, N—H···O and O—H···O hydrogen bonds (Table 1). Classical hydrogen bonds link the molecules into helical chains propagating along the *b*-axis direction (Fig. 2 and Table 1). The unit-cell packing exhibits layered stacking when viewed along the *b*-axis direction, as shown in Fig. 3.

Synthesis and crystallization

4.0 g (17 mmol) of 2α,3α:6α,7α-diepoxyhimachalane (Lassaba *et al.*, 1998) was dissolved in 30 ml of CH_3CN and stirred at 273 K under argon. BF_3OEt (3% mmol) was added and the reaction mixture was stirred and monitored by TLC. After completion of the reaction, the solvent was removed and the residue obtained was chromatographed on silica, eluting with hexane–ethylacetate (88:12), which allowed the isolation of the title compound (yield: 3 g, 10 mmol, 60%). Colourless plates were recrystallized from ethyl acetate solution.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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References

Benharref, A., El Ammari, L., Saadi, M., Mazoir, N., Daran, J.-C. & Berraho, M. (2017). *IUCrData*, **2**, x170584.
Daoubi, M., Durán-Patrón, R., Hmamouchi, M., Hernández-Galán, R., Benharref, A. & Collado, I. G. (2004). *Pest Manag. Sci.* **60**, 927–932.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
El Haib, A., Benharref, A., Parrès-Maynadié, S., Manoury, E., Daran, J. C., Urrutigoïty, M. & Gouygou, M. (2010). *Tetrahedron Asymmetry*, **21**, 1272–1277.
Lassaba, E., ElJamili, H., Chekroun, A., Benharref, A., Chiaroni, A., Riche, C. & Lavergne, J.-P. (1998). *Synth. Commun.* **28**, 2641–2651.
Loubidi, M., Agustin, D., Benharref, A. & Poli, R. (2014). *C. R. Chim.* **17**, 549–556.
Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
Rigaku Oxford Diffraction (2015). *CrysAlis PRO*. Rigaku Oxford Diffraction Ltd, Yarnton, England.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* A**71**, 3–8.

full crystallographic data

IUCrData (2017). **2**, x170970 [https://doi.org/10.1107/S2414314617009701]

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Crystal data

$C_{17}H_{29}NO_3$
M_r = 295.42
Monoclinic, $P2_1$
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b = 7.9149 (4) Å
c = 14.8297 (8) Å
β = 106.803 (7)°
V = 1630.15 (17) Å^3
Z = 4
$F(000)$ = 648
D_x = 1.204 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6178 reflections
θ = 2.9–26.4°
μ = 0.08 mm^{-1}
T = 293 K
Plate, colourless
0.50 × 0.25 × 0.12 mm

Data collection

Rigaku Oxford Diffraction Xcalibur Eos Gemini ultra diffractometer
Radiation source: fine-focus sealed X-ray tube, Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.1978 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku Oxford Diffraction, 2015)
T_{min} = 0.628, T_{max} = 1.000
16240 measured reflections
6178 independent reflections
4204 reflections with $I > 2\sigma(I)$
R_{int} = 0.063
θ_{max} = 26.4°, θ_{min} = 3.0°
h = −18→18
k = −9→9
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.115
S = 0.99
6178 reflections
391 parameters
1 restraint
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0496P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-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.

Refinement. All H atoms attached to C atoms were fixed geometrically and treated as riding with C—H = 0.99 Å (methylene), 0.98 Å (methyl), 1.0Å (methine) with $U_{iso}(H) = 1.2U_{eq}$(CH and CH_2) or $U_{iso}(H) = 1.5U_{eq}(CH_3)$. The coordinates of H atoms attached to N atoms were freely refined with $U_{iso}(H) = 1.2U_{eq}(N)$ and the H attached to hydroxyl O atoms were fixed geometrically and treated as riding with O—H = 0.84Å and $U_{iso}(H) = 1.5U_{eq}(O)$.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1A	0.6042 (3)	0.9215 (5)	0.3367 (3)	0.0176 (9)
C1B	0.1521 (3)	0.7991 (5)	0.1239 (3)	0.0184 (9)
C2A	0.5787 (3)	1.1119 (5)	0.3300 (3)	0.0194 (9)
C2B	0.2228 (3)	0.8563 (5)	0.0698 (3)	0.0234 (10)
C3A	0.5175 (3)	1.1635 (5)	0.2328 (3)	0.0256 (11)
H3A1	0.5051	1.2838	0.2336	0.031*
H3A2	0.4559	1.1062	0.2201	0.031*
C3B	0.2688 (3)	0.7124 (6)	0.0319 (3)	0.0283 (11)
H3B1	0.3157	0.7598	0.0039	0.034*
H3B2	0.2193	0.6586	−0.0182	0.034*
C4A	0.5593 (3)	1.1271 (5)	0.1517 (3)	0.0277 (11)
H4A1	0.6267	1.1594	0.1703	0.033*
H4A2	0.5263	1.1964	0.0982	0.033*
C4B	0.3191 (3)	0.5749 (6)	0.1016 (3)	0.0285 (11)
H4B1	0.3597	0.5086	0.0733	0.034*
H4B2	0.3603	0.6285	0.1576	0.034*
C5A	0.5511 (4)	0.9415 (6)	0.1212 (3)	0.0282 (11)
H5A1	0.5453	0.9381	0.0544	0.034*
H5A2	0.4914	0.8977	0.1289	0.034*
C5B	0.2495 (3)	0.4569 (5)	0.1303 (3)	0.0284 (11)
H5B1	0.2798	0.3468	0.1431	0.034*
H5B2	0.1929	0.4441	0.0766	0.034*
C6A	0.6328 (3)	0.8185 (5)	0.1716 (3)	0.0216 (10)
C6B	0.2152 (3)	0.5072 (5)	0.2162 (3)	0.0241 (10)
C7A	0.6721 (3)	0.8589 (5)	0.2791 (3)	0.0162 (9)
H7A	0.7171	0.9526	0.2824	0.019*
C7B	0.1981 (3)	0.7020 (5)	0.2176 (3)	0.0188 (9)
H7B	0.2625	0.7511	0.2426	0.023*
C8A	0.7331 (3)	0.7213 (5)	0.3489 (3)	0.0182 (9)
H8A	0.7964	0.7716	0.3785	0.022*
C8B	0.1376 (3)	0.7772 (5)	0.2814 (3)	0.0204 (10)
H8B	0.1795	0.8595	0.3233	0.024*
C9A	0.6804 (4)	0.7145 (5)	0.4250 (3)	0.0248 (11)
C9B	0.0608 (3)	0.8793 (5)	0.2109 (3)	0.0202 (10)
C10A	0.5781 (3)	0.6436 (5)	0.3840 (3)	0.0274 (11)

H10A	0.5509	0.6087	0.4336	0.033*
H10B	0.5778	0.5483	0.3428	0.033*
C10B	−0.0064 (3)	0.7649 (5)	0.1357 (3)	0.0238 (10)
H10C	−0.0236	0.6636	0.1638	0.029*
H10D	−0.0646	0.8243	0.1020	0.029*
C11A	0.5228 (3)	0.7952 (5)	0.3287 (3)	0.0273 (11)
H11C	0.4912	0.7658	0.2635	0.033*
H11D	0.4753	0.8384	0.3574	0.033*
C11B	0.0558 (3)	0.7226 (6)	0.0701 (3)	0.0234 (10)
H11A	0.0608	0.6016	0.0625	0.028*
H11B	0.0303	0.7747	0.0087	0.028*
C12A	0.5288 (4)	1.1601 (5)	0.4038 (3)	0.0295 (11)
H12D	0.5188	1.2800	0.4024	0.044*
H12E	0.5684	1.1272	0.4650	0.044*
H12F	0.4678	1.1033	0.3903	0.044*
C12B	0.1739 (4)	0.9771 (6)	−0.0103 (3)	0.0367 (13)
H12A	0.2208	1.0201	−0.0387	0.055*
H12B	0.1455	1.0692	0.0142	0.055*
H12C	0.1247	0.9176	−0.0568	0.055*
C13A	0.5922 (4)	0.6396 (6)	0.1448 (3)	0.0292 (11)
H13D	0.6444	0.5607	0.1542	0.044*
H13E	0.5547	0.6382	0.0798	0.044*
H13F	0.5523	0.6084	0.1836	0.044*
C13B	0.2971 (4)	0.4656 (6)	0.3050 (3)	0.0340 (12)
H13A	0.3077	0.3457	0.3087	0.051*
H13B	0.2800	0.5030	0.3596	0.051*
H13C	0.3549	0.5218	0.3023	0.051*
C14A	0.7174 (4)	0.8430 (6)	0.1309 (3)	0.0321 (12)
H14D	0.6987	0.8084	0.0661	0.048*
H14E	0.7709	0.7759	0.1658	0.048*
H14F	0.7356	0.9600	0.1351	0.048*
C14B	0.1300 (4)	0.3921 (5)	0.2153 (3)	0.0328 (12)
H14A	0.1198	0.3926	0.2765	0.049*
H14B	0.1436	0.2790	0.1995	0.049*
H14C	0.0731	0.4326	0.1694	0.049*
C15A	0.7375 (4)	0.6418 (6)	0.5174 (3)	0.0401 (14)
H15A	0.7021	0.6542	0.5626	0.060*
H15B	0.7978	0.7005	0.5393	0.060*
H15C	0.7493	0.5242	0.5096	0.060*
C15B	0.0104 (4)	1.0103 (5)	0.2527 (3)	0.0305 (11)
H15D	−0.0238	1.0867	0.2042	0.046*
H15E	0.0570	1.0720	0.3005	0.046*
H15F	−0.0343	0.9558	0.2800	0.046*
C16A	0.8330 (3)	0.5161 (5)	0.2981 (3)	0.0244 (10)
C16B	0.1403 (4)	0.6587 (5)	0.4341 (3)	0.0252 (11)
C17A	0.8449 (4)	0.3346 (5)	0.2726 (3)	0.0346 (12)
H17A	0.9066	0.2938	0.3095	0.052*
H17B	0.8407	0.3273	0.2069	0.052*

H17C	0.7950	0.2671	0.2852	0.052*
C17B	0.0852 (4)	0.5607 (6)	0.4879 (3)	0.0427 (14)
H17D	0.0274	0.6209	0.4869	0.064*
H17E	0.1241	0.5468	0.5518	0.064*
H17F	0.0687	0.4518	0.4593	0.064*
N1	0.7507 (3)	0.5542 (4)	0.3163 (2)	0.0207 (8)
H1	0.7067	0.4781	0.3084	0.025*
N2	0.0990 (3)	0.6698 (4)	0.3410 (2)	0.0207 (8)
H2	0.0481	0.6115	0.3158	0.025*
O1A	0.6662 (2)	1.2086 (3)	0.34675 (18)	0.0217 (7)
H1A	0.6966	1.2028	0.4026	0.033*
O1B	0.3005 (2)	0.9460 (4)	0.1336 (2)	0.0299 (8)
H1B	0.2790	1.0098	0.1662	0.045*
O2A	0.6627 (2)	0.8915 (3)	0.43286 (17)	0.0228 (7)
O2B	0.1198 (2)	0.9552 (3)	0.15845 (19)	0.0219 (7)
O3A	0.8965 (2)	0.6211 (4)	0.3013 (2)	0.0354 (8)
O3B	0.2193 (3)	0.7230 (4)	0.4729 (2)	0.0359 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1A	0.019 (3)	0.017 (2)	0.016 (2)	−0.0008 (19)	0.0030 (19)	0.0002 (18)
C1B	0.016 (3)	0.017 (2)	0.019 (2)	0.0011 (19)	0.001 (2)	−0.0040 (18)
C2A	0.016 (2)	0.020 (2)	0.023 (2)	−0.0002 (19)	0.005 (2)	−0.0009 (18)
C2B	0.018 (3)	0.028 (2)	0.021 (2)	−0.003 (2)	0.002 (2)	−0.0015 (19)
C3A	0.023 (3)	0.017 (2)	0.033 (2)	0.001 (2)	0.002 (2)	−0.0001 (19)
C3B	0.023 (3)	0.039 (3)	0.024 (2)	−0.005 (2)	0.008 (2)	−0.004 (2)
C4A	0.030 (3)	0.029 (2)	0.017 (2)	0.005 (2)	−0.004 (2)	0.006 (2)
C4B	0.023 (3)	0.033 (3)	0.030 (2)	0.002 (2)	0.009 (2)	−0.008 (2)
C5A	0.032 (3)	0.032 (3)	0.016 (2)	0.007 (2)	0.000 (2)	0.000 (2)
C5B	0.026 (3)	0.020 (2)	0.038 (3)	0.005 (2)	0.008 (2)	−0.005 (2)
C6A	0.022 (3)	0.026 (2)	0.016 (2)	0.003 (2)	0.003 (2)	0.0012 (18)
C6B	0.024 (3)	0.018 (2)	0.031 (2)	0.002 (2)	0.008 (2)	0.0037 (19)
C7A	0.012 (2)	0.013 (2)	0.022 (2)	−0.0017 (17)	0.0038 (19)	0.0044 (17)
C7B	0.015 (2)	0.018 (2)	0.020 (2)	−0.0020 (18)	0.001 (2)	−0.0019 (18)
C8A	0.018 (3)	0.014 (2)	0.020 (2)	−0.0010 (19)	0.001 (2)	−0.0009 (18)
C8B	0.020 (3)	0.022 (2)	0.019 (2)	−0.005 (2)	0.004 (2)	−0.0030 (18)
C9A	0.041 (3)	0.014 (2)	0.022 (2)	0.007 (2)	0.014 (2)	−0.0001 (19)
C9B	0.015 (2)	0.021 (2)	0.024 (2)	0.0012 (19)	0.005 (2)	0.0011 (19)
C10A	0.037 (3)	0.018 (2)	0.036 (3)	−0.004 (2)	0.024 (2)	−0.005 (2)
C10B	0.016 (3)	0.027 (3)	0.025 (2)	−0.0004 (19)	0.000 (2)	0.0019 (19)
C11A	0.028 (3)	0.024 (2)	0.035 (3)	−0.005 (2)	0.018 (2)	−0.008 (2)
C11B	0.018 (3)	0.028 (2)	0.021 (2)	−0.001 (2)	0.001 (2)	0.002 (2)
C12A	0.033 (3)	0.021 (2)	0.037 (3)	0.004 (2)	0.015 (2)	−0.002 (2)
C12B	0.041 (4)	0.038 (3)	0.036 (3)	0.001 (2)	0.018 (3)	0.009 (2)
C13A	0.033 (3)	0.029 (2)	0.021 (2)	0.001 (2)	0.000 (2)	−0.006 (2)
C13B	0.030 (3)	0.032 (3)	0.038 (3)	0.012 (2)	0.007 (3)	0.014 (2)
C14A	0.043 (3)	0.033 (3)	0.024 (2)	0.007 (2)	0.015 (2)	0.002 (2)

C14B	0.036 (3)	0.020 (2)	0.047 (3)	−0.005 (2)	0.018 (3)	−0.004 (2)
C15A	0.068 (4)	0.027 (2)	0.027 (3)	0.013 (3)	0.015 (3)	0.003 (2)
C15B	0.031 (3)	0.024 (2)	0.039 (3)	0.001 (2)	0.014 (3)	0.000 (2)
C16A	0.019 (3)	0.030 (3)	0.023 (2)	0.007 (2)	0.006 (2)	0.010 (2)
C16B	0.031 (3)	0.021 (2)	0.021 (2)	0.007 (2)	0.003 (2)	0.0014 (19)
C17A	0.035 (3)	0.030 (3)	0.046 (3)	0.008 (2)	0.023 (3)	0.004 (2)
C17B	0.053 (4)	0.048 (3)	0.030 (3)	0.010 (3)	0.015 (3)	0.014 (2)
N1	0.020 (2)	0.0172 (18)	0.0270 (19)	−0.0004 (16)	0.0094 (18)	−0.0022 (15)
N2	0.020 (2)	0.023 (2)	0.0179 (18)	−0.0069 (16)	0.0031 (17)	−0.0009 (15)
O1A	0.0243 (19)	0.0184 (15)	0.0208 (15)	−0.0048 (14)	0.0038 (14)	−0.0010 (14)
O1B	0.025 (2)	0.0289 (18)	0.0359 (18)	−0.0071 (15)	0.0092 (16)	−0.0043 (14)
O2A	0.034 (2)	0.0173 (15)	0.0175 (15)	0.0028 (14)	0.0072 (14)	−0.0005 (12)
O2B	0.0228 (19)	0.0168 (15)	0.0272 (15)	0.0009 (13)	0.0089 (15)	0.0019 (13)
O3A	0.022 (2)	0.0331 (18)	0.053 (2)	−0.0023 (16)	0.0156 (17)	0.0059 (16)
O3B	0.040 (2)	0.0319 (18)	0.0258 (17)	−0.0023 (17)	−0.0064 (17)	0.0021 (15)

Geometric parameters (Å, º)

C1A—O2A	1.453 (5)	C9B—C15B	1.502 (6)
C1A—C11A	1.526 (6)	C9B—C10B	1.544 (6)
C1A—C2A	1.548 (5)	C10A—C11A	1.543 (6)
C1A—C7A	1.560 (5)	C10A—H10A	0.9700
C1B—O2B	1.465 (5)	C10A—H10B	0.9700
C1B—C11B	1.520 (6)	C10B—C11B	1.542 (6)
C1B—C2B	1.542 (6)	C10B—H10C	0.9700
C1B—C7B	1.560 (6)	C10B—H10D	0.9700
C2A—O1A	1.442 (5)	C11A—H11C	0.9700
C2A—C3A	1.514 (6)	C11A—H11D	0.9700
C2A—C12A	1.524 (5)	C11B—H11A	0.9700
C2B—O1B	1.432 (5)	C11B—H11B	0.9700
C2B—C3B	1.508 (6)	C12A—H12D	0.9600
C2B—C12B	1.530 (6)	C12A—H12E	0.9600
C3A—C4A	1.524 (6)	C12A—H12F	0.9600
C3A—H3A1	0.9700	C12B—H12A	0.9600
C3A—H3A2	0.9700	C12B—H12B	0.9600
C3B—C4B	1.532 (6)	C12B—H12C	0.9600
C3B—H3B1	0.9700	C13A—H13D	0.9600
C3B—H3B2	0.9700	C13A—H13E	0.9600
C4A—C5A	1.532 (6)	C13A—H13F	0.9600
C4A—H4A1	0.9700	C13B—H13A	0.9600
C4A—H4A2	0.9700	C13B—H13B	0.9600
C4B—C5B	1.523 (6)	C13B—H13C	0.9600
C4B—H4B1	0.9700	C14A—H14D	0.9600
C4B—H4B2	0.9700	C14A—H14E	0.9600
C5A—C6A	1.549 (6)	C14A—H14F	0.9600
C5A—H5A1	0.9700	C14B—H14A	0.9600
C5A—H5A2	0.9700	C14B—H14B	0.9600
C5B—C6B	1.548 (5)	C14B—H14C	0.9600

C5B—H5B1	0.9700	C15A—H15A	0.9600
C5B—H5B2	0.9700	C15A—H15B	0.9600
C6A—C14A	1.529 (6)	C15A—H15C	0.9600
C6A—C13A	1.541 (6)	C15B—H15D	0.9600
C6A—C7A	1.563 (5)	C15B—H15E	0.9600
C6B—C13B	1.533 (6)	C15B—H15F	0.9600
C6B—C14B	1.533 (6)	C16A—O3A	1.232 (5)
C6B—C7B	1.563 (6)	C16A—N1	1.332 (5)
C7A—C8A	1.586 (5)	C16A—C17A	1.508 (6)
C7A—H7A	0.9800	C16B—O3B	1.234 (5)
C7B—C8B	1.580 (5)	C16B—N2	1.339 (5)
C7B—H7B	0.9800	C16B—C17B	1.498 (6)
C8A—N1	1.456 (5)	C17A—H17A	0.9600
C8A—C9A	1.536 (5)	C17A—H17B	0.9600
C8A—H8A	0.9800	C17A—H17C	0.9600
C8B—N2	1.451 (5)	C17B—H17D	0.9600
C8B—C9B	1.522 (6)	C17B—H17E	0.9600
C8B—H8B	0.9800	C17B—H17F	0.9600
C9A—O2A	1.435 (5)	N1—H1	0.8600
C9A—C15A	1.496 (6)	N2—H2	0.8600
C9A—C10A	1.537 (6)	O1A—H1A	0.8200
C9B—O2B	1.443 (5)	O1B—H1B	0.8200

O2A—C1A—C11A	100.9 (3)	C15B—C9B—C8B	115.5 (3)
O2A—C1A—C2A	106.4 (3)	O2B—C9B—C10B	101.8 (3)
C11A—C1A—C2A	117.9 (3)	C15B—C9B—C10B	114.9 (4)
O2A—C1A—C7A	101.7 (3)	C8B—C9B—C10B	111.7 (3)
C11A—C1A—C7A	110.3 (3)	C9A—C10A—C11A	102.7 (3)
C2A—C1A—C7A	116.9 (3)	C9A—C10A—H10A	111.2
O2B—C1B—C11B	100.4 (3)	C11A—C10A—H10A	111.2
O2B—C1B—C2B	105.2 (3)	C9A—C10A—H10B	111.2
C11B—C1B—C2B	119.6 (3)	C11A—C10A—H10B	111.2
O2B—C1B—C7B	101.6 (3)	H10A—C10A—H10B	109.1
C11B—C1B—C7B	111.3 (3)	C11B—C10B—C9B	102.7 (3)
C2B—C1B—C7B	115.5 (3)	C11B—C10B—H10C	111.2
O1A—C2A—C3A	105.4 (3)	C9B—C10B—H10C	111.2
O1A—C2A—C12A	108.7 (3)	C11B—C10B—H10D	111.2
C3A—C2A—C12A	110.3 (4)	C9B—C10B—H10D	111.2
O1A—C2A—C1A	108.9 (3)	H10C—C10B—H10D	109.1
C3A—C2A—C1A	112.7 (3)	C1A—C11A—C10A	101.3 (4)
C12A—C2A—C1A	110.6 (3)	C1A—C11A—H11C	111.5
O1B—C2B—C3B	105.8 (4)	C10A—C11A—H11C	111.5
O1B—C2B—C12B	107.6 (4)	C1A—C11A—H11D	111.5
C3B—C2B—C12B	109.8 (3)	C10A—C11A—H11D	111.5
O1B—C2B—C1B	108.5 (3)	H11C—C11A—H11D	109.3
C3B—C2B—C1B	113.9 (3)	C1B—C11B—C10B	101.5 (3)
C12B—C2B—C1B	111.0 (4)	C1B—C11B—H11A	111.5
C2A—C3A—C4A	116.3 (4)	C10B—C11B—H11A	111.5

C2A—C3A—H3A1	108.2	C1B—C11B—H11B	111.5
C4A—C3A—H3A1	108.2	C10B—C11B—H11B	111.5
C2A—C3A—H3A2	108.2	H11A—C11B—H11B	109.3
C4A—C3A—H3A2	108.2	C2A—C12A—H12D	109.5
H3A1—C3A—H3A2	107.4	C2A—C12A—H12E	109.5
C2B—C3B—C4B	117.4 (3)	H12D—C12A—H12E	109.5
C2B—C3B—H3B1	107.9	C2A—C12A—H12F	109.5
C4B—C3B—H3B1	107.9	H12D—C12A—H12F	109.5
C2B—C3B—H3B2	107.9	H12E—C12A—H12F	109.5
C4B—C3B—H3B2	107.9	C2B—C12B—H12A	109.5
H3B1—C3B—H3B2	107.2	C2B—C12B—H12B	109.5
C3A—C4A—C5A	113.5 (4)	H12A—C12B—H12B	109.5
C3A—C4A—H4A1	108.9	C2B—C12B—H12C	109.5
C5A—C4A—H4A1	108.9	H12A—C12B—H12C	109.5
C3A—C4A—H4A2	108.9	H12B—C12B—H12C	109.5
C5A—C4A—H4A2	108.9	C6A—C13A—H13D	109.5
H4A1—C4A—H4A2	107.7	C6A—C13A—H13E	109.5
C5B—C4B—C3B	113.6 (4)	H13D—C13A—H13E	109.5
C5B—C4B—H4B1	108.9	C6A—C13A—H13F	109.5
C3B—C4B—H4B1	108.9	H13D—C13A—H13F	109.5
C5B—C4B—H4B2	108.9	H13E—C13A—H13F	109.5
C3B—C4B—H4B2	108.9	C6B—C13B—H13A	109.5
H4B1—C4B—H4B2	107.7	C6B—C13B—H13B	109.5
C4A—C5A—C6A	118.5 (4)	H13A—C13B—H13B	109.5
C4A—C5A—H5A1	107.7	C6B—C13B—H13C	109.5
C6A—C5A—H5A1	107.7	H13A—C13B—H13C	109.5
C4A—C5A—H5A2	107.7	H13B—C13B—H13C	109.5
C6A—C5A—H5A2	107.7	C6A—C14A—H14D	109.5
H5A1—C5A—H5A2	107.1	C6A—C14A—H14E	109.5
C4B—C5B—C6B	118.0 (4)	H14D—C14A—H14E	109.5
C4B—C5B—H5B1	107.8	C6A—C14A—H14F	109.5
C6B—C5B—H5B1	107.8	H14D—C14A—H14F	109.5
C4B—C5B—H5B2	107.8	H14E—C14A—H14F	109.5
C6B—C5B—H5B2	107.8	C6B—C14B—H14A	109.5
H5B1—C5B—H5B2	107.2	C6B—C14B—H14B	109.5
C14A—C6A—C13A	107.8 (3)	H14A—C14B—H14B	109.5
C14A—C6A—C5A	108.6 (3)	C6B—C14B—H14C	109.5
C13A—C6A—C5A	105.7 (4)	H14A—C14B—H14C	109.5
C14A—C6A—C7A	106.2 (4)	H14B—C14B—H14C	109.5
C13A—C6A—C7A	116.8 (3)	C9A—C15A—H15A	109.5
C5A—C6A—C7A	111.5 (3)	C9A—C15A—H15B	109.5
C13B—C6B—C14B	107.6 (4)	H15A—C15A—H15B	109.5
C13B—C6B—C5B	107.3 (4)	C9A—C15A—H15C	109.5
C14B—C6B—C5B	106.9 (3)	H15A—C15A—H15C	109.5
C13B—C6B—C7B	106.7 (3)	H15B—C15A—H15C	109.5
C14B—C6B—C7B	117.1 (4)	C9B—C15B—H15D	109.5
C5B—C6B—C7B	110.8 (3)	C9B—C15B—H15E	109.5
C1A—C7A—C6A	121.4 (3)	H15D—C15B—H15E	109.5

C1A—C7A—C8A	100.5 (3)	C9B—C15B—H15F	109.5
C6A—C7A—C8A	119.7 (3)	H15D—C15B—H15F	109.5
C1A—C7A—H7A	104.4	H15E—C15B—H15F	109.5
C6A—C7A—H7A	104.4	O3A—C16A—N1	122.9 (4)
C8A—C7A—H7A	104.4	O3A—C16A—C17A	120.9 (4)
C1B—C7B—C6B	120.2 (3)	N1—C16A—C17A	116.2 (4)
C1B—C7B—C8B	100.3 (3)	O3B—C16B—N2	122.1 (4)
C6B—C7B—C8B	119.8 (3)	O3B—C16B—C17B	122.2 (4)
C1B—C7B—H7B	104.9	N2—C16B—C17B	115.8 (4)
C6B—C7B—H7B	104.9	C16A—C17A—H17A	109.5
C8B—C7B—H7B	104.9	C16A—C17A—H17B	109.5
N1—C8A—C9A	112.6 (3)	H17A—C17A—H17B	109.5
N1—C8A—C7A	121.6 (3)	C16A—C17A—H17C	109.5
C9A—C8A—C7A	101.6 (3)	H17A—C17A—H17C	109.5
N1—C8A—H8A	106.7	H17B—C17A—H17C	109.5
C9A—C8A—H8A	106.7	C16B—C17B—H17D	109.5
C7A—C8A—H8A	106.7	C16B—C17B—H17E	109.5
N2—C8B—C9B	113.3 (4)	H17D—C17B—H17E	109.5
N2—C8B—C7B	121.5 (3)	C16B—C17B—H17F	109.5
C9B—C8B—C7B	102.2 (3)	H17D—C17B—H17F	109.5
N2—C8B—H8B	106.3	H17E—C17B—H17F	109.5
C9B—C8B—H8B	106.3	C16A—N1—C8A	121.8 (4)
C7B—C8B—H8B	106.3	C16A—N1—H1	119.1
O2A—C9A—C15A	111.4 (3)	C8A—N1—H1	119.1
O2A—C9A—C8A	99.5 (3)	C16B—N2—C8B	122.1 (4)
C15A—C9A—C8A	115.1 (4)	C16B—N2—H2	119.0
O2A—C9A—C10A	102.3 (4)	C8B—N2—H2	119.0
C15A—C9A—C10A	115.9 (4)	C2A—O1A—H1A	109.5
C8A—C9A—C10A	110.6 (3)	C2B—O1B—H1B	109.5
O2B—C9B—C15B	111.7 (3)	C9A—O2A—C1A	98.5 (3)
O2B—C9B—C8B	99.2 (3)	C9B—O2B—C1B	97.8 (3)

O2A—C1A—C2A—O1A	63.2 (4)	C14B—C6B—C7B—C8B	40.9 (6)
C11A—C1A—C2A—O1A	175.4 (3)	C5B—C6B—C7B—C8B	163.8 (4)
C7A—C1A—C2A—O1A	−49.5 (4)	C1A—C7A—C8A—N1	133.3 (4)
O2A—C1A—C2A—C3A	179.8 (3)	C6A—C7A—C8A—N1	−2.5 (6)
C11A—C1A—C2A—C3A	−68.0 (4)	C1A—C7A—C8A—C9A	7.3 (4)
C7A—C1A—C2A—C3A	67.0 (5)	C6A—C7A—C8A—C9A	−128.5 (4)
O2A—C1A—C2A—C12A	−56.2 (4)	C1B—C7B—C8B—N2	135.4 (4)
C11A—C1A—C2A—C12A	56.0 (5)	C6B—C7B—C8B—N2	1.5 (6)
C7A—C1A—C2A—C12A	−169.0 (3)	C1B—C7B—C8B—C9B	8.0 (4)
O2B—C1B—C2B—O1B	59.7 (4)	C6B—C7B—C8B—C9B	−125.9 (4)
C11B—C1B—C2B—O1B	171.4 (3)	N1—C8A—C9A—O2A	−172.7 (3)
C7B—C1B—C2B—O1B	−51.5 (4)	C7A—C8A—C9A—O2A	−40.9 (4)
O2B—C1B—C2B—C3B	177.2 (3)	N1—C8A—C9A—C15A	68.1 (5)
C11B—C1B—C2B—C3B	−71.1 (5)	C7A—C8A—C9A—C15A	−160.1 (4)
C7B—C1B—C2B—C3B	66.0 (4)	N1—C8A—C9A—C10A	−65.6 (4)
O2B—C1B—C2B—C12B	−58.4 (4)	C7A—C8A—C9A—C10A	66.2 (4)

C11B—C1B—C2B—C12B	53.3 (5)	N2—C8B—C9B—O2B	−174.4 (3)
C7B—C1B—C2B—C12B	−169.5 (4)	C7B—C8B—C9B—O2B	−41.9 (3)
O1A—C2A—C3A—C4A	61.4 (4)	N2—C8B—C9B—C15B	66.0 (5)
C12A—C2A—C3A—C4A	178.6 (4)	C7B—C8B—C9B—C15B	−161.5 (4)
C1A—C2A—C3A—C4A	−57.2 (5)	N2—C8B—C9B—C10B	−67.7 (4)
O1B—C2B—C3B—C4B	65.9 (5)	C7B—C8B—C9B—C10B	64.8 (4)
C12B—C2B—C3B—C4B	−178.2 (4)	O2A—C9A—C10A—C11A	28.0 (4)
C1B—C2B—C3B—C4B	−53.1 (5)	C15A—C9A—C10A—C11A	149.5 (4)
C2A—C3A—C4A—C5A	77.3 (5)	C8A—C9A—C10A—C11A	−77.2 (4)
C2B—C3B—C4B—C5B	73.9 (5)	O2B—C9B—C10B—C11B	28.4 (4)
C3A—C4A—C5A—C6A	−87.9 (5)	C15B—C9B—C10B—C11B	149.3 (3)
C3B—C4B—C5B—C6B	−88.7 (5)	C8B—C9B—C10B—C11B	−76.7 (4)
C4A—C5A—C6A—C14A	−78.9 (4)	O2A—C1A—C11A—C10A	−38.5 (3)
C4A—C5A—C6A—C13A	165.7 (4)	C2A—C1A—C11A—C10A	−153.8 (3)
C4A—C5A—C6A—C7A	37.8 (5)	C7A—C1A—C11A—C10A	68.4 (4)
C4B—C5B—C6B—C13B	−77.7 (5)	C9A—C10A—C11A—C1A	6.5 (4)
C4B—C5B—C6B—C14B	167.1 (4)	O2B—C1B—C11B—C10B	−39.5 (4)
C4B—C5B—C6B—C7B	38.4 (5)	C2B—C1B—C11B—C10B	−153.8 (3)
O2A—C1A—C7A—C6A	163.0 (3)	C7B—C1B—C11B—C10B	67.5 (4)
C11A—C1A—C7A—C6A	56.6 (5)	C9B—C10B—C11B—C1B	6.9 (4)
C2A—C1A—C7A—C6A	−81.6 (5)	O3A—C16A—N1—C8A	−5.1 (6)
O2A—C1A—C7A—C8A	28.2 (3)	C17A—C16A—N1—C8A	175.0 (3)
C11A—C1A—C7A—C8A	−78.2 (4)	C9A—C8A—N1—C16A	−140.3 (4)
C2A—C1A—C7A—C8A	143.6 (3)	C7A—C8A—N1—C16A	98.9 (5)
C14A—C6A—C7A—C1A	154.1 (3)	O3B—C16B—N2—C8B	−8.7 (6)
C13A—C6A—C7A—C1A	−85.7 (5)	C17B—C16B—N2—C8B	172.3 (4)
C5A—C6A—C7A—C1A	35.9 (5)	C9B—C8B—N2—C16B	−135.4 (4)
C14A—C6A—C7A—C8A	−79.4 (4)	C7B—C8B—N2—C16B	102.4 (5)
C13A—C6A—C7A—C8A	40.8 (5)	C15A—C9A—O2A—C1A	−177.5 (4)
C5A—C6A—C7A—C8A	162.5 (4)	C8A—C9A—O2A—C1A	60.7 (4)
O2B—C1B—C7B—C6B	161.6 (4)	C10A—C9A—O2A—C1A	−52.9 (3)
C11B—C1B—C7B—C6B	55.4 (5)	C11A—C1A—O2A—C9A	57.6 (3)
C2B—C1B—C7B—C6B	−85.1 (5)	C2A—C1A—O2A—C9A	−178.9 (3)
O2B—C1B—C7B—C8B	27.9 (4)	C7A—C1A—O2A—C9A	−56.0 (3)
C11B—C1B—C7B—C8B	−78.3 (4)	C15B—C9B—O2B—C1B	−176.7 (3)
C2B—C1B—C7B—C8B	141.2 (3)	C8B—C9B—O2B—C1B	61.0 (3)
C13B—C6B—C7B—C1B	155.4 (4)	C10B—C9B—O2B—C1B	−53.6 (3)
C14B—C6B—C7B—C1B	−84.0 (5)	C11B—C1B—O2B—C9B	58.7 (3)
C5B—C6B—C7B—C1B	38.9 (5)	C2B—C1B—O2B—C9B	−176.6 (3)
C13B—C6B—C7B—C8B	−79.6 (5)	C7B—C1B—O2B—C9B	−55.8 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1*A*^{i}	0.86	2.33	3.084 (4)	147
N2—H2···O3*A*^{ii}	0.86	2.15	2.851 (5)	139
O1*A*—H1*A*···O3*B*^{iii}	0.82	1.90	2.712 (4)	169
O1*B*—H1*B*···O2*B*	0.82	2.32	2.752 (4)	114

C3*A*—H3*A*2···O1*B*	0.97	2.58	3.516 (5)	162
C15*A*—H15*C*···O3*B*^{iv}	0.96	2.43	3.369 (6)	167
C17*A*—H17*C*···O1*A*^{i}	0.96	2.35	3.252 (6)	156

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