Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(2*S*,4a*R*,6a*R*,7*R*,9*S*,10a*S*,10b*R*)-7-Carboxy-2-(3-furyl)-6a,10b-dimethyl-4,10-dioxoperhydrobenzo[*f*]isochromen-9-yl acetate

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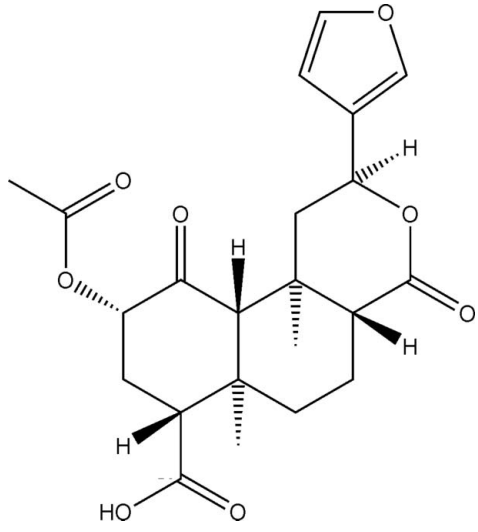
Received 18 December 2008; accepted 15 January 2009

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.029; wR factor = 0.074; data-to-parameter ratio = 13.2.

The asymmetric unit of the title compound, $C_{22}H_{26}O_8$, contains two crystallographically independent molecules with closely comparable conformations (r.m.s. overlay = 0.54 Å for 30 non-H atoms). All six-membered rings display chair conformations, with a slight distortion for the lactone ring. The molecules are connected by O—H···O hydrogen bonds into chains along [010], with the independent molecules segregated into separate chains. The two molecules in the asymmetric unit face each other in a head-to-tail fashion, with the furan ring of one molecule turned towards the carboxylic acid terminal of the other molecule.

Related literature

For the biosynthesis of Salvinorin A, see: Kutrzeba *et al.* (2007). For the isolation of Salvinorin A and further synthesis details, see: Lee, Karnati *et al.* (2005); Lee, He *et al.* (2005); Stewart (2005). For details on epimerization at the C-8 stereogenic center, see: Harding *et al.* (2005).



Experimental

Crystal data

$C_{22}H_{26}O_8$
M_r = 418.43
Monoclinic, $P2_1$
a = 11.2735 (6) Å
b = 16.8015 (9) Å
c = 11.3765 (6) Å
β = 111.934 (3)°
V = 1998.86 (18) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 0.89 mm^{-1}
T = 100 (2) K
0.19 × 0.12 × 0.09 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: none
33644 measured reflections
7269 independent reflections
6962 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.074
S = 1.10
7269 reflections
549 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Absolute structure: Flack (1983), 3360 Friedel pairs
Flack parameter: −0.09 (13)

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

D—H···A	D—H	H···A	D···A	D—H···A
O8—H8A···O6^{i}	0.82	1.92	2.7264 (18)	168
O8′—H8′···O3′^{ii}	0.82	1.92	2.7308 (17)	172

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* .

The authors thank the Center for Disease Control and Prevention, USA, for providing financial assistance (CDC

cooperative agreements 1UO1CI000211–03 and1UO1 CI000362–01). This Investigation was conducted in a facility constructed with support from Research Facilities Improvement Program grant No. C06 RR-14503–01 from the National Center for Research Resources, National Institutes of Health.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2338).

References

Bruker (2005). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Harding, W. W., Tidgewell, K., Byrd, N., Cobb, H., Dersch, C. M., Butelman, E. R., Rothman, R. B. & Prisinzano, T. E. (2005). *J. Med. Chem.* **48**, 4765–4771.

Kutrzeba, L., Dayan, F. E., Howell, J. L., Feng, J., Giner, J.-L. & Zjawiony, J. K. (2007). *Phytochemistry*, **68**, 1872–1881.

Lee, D. Y., He, M., Kondaveti, L., Liu-Chen, L. Y., Ma, Z., Wang, Y., Chen, Y., Li, J. G., Beguin, C., Carlezon, W. A. J. & Cohen, B. (2005). *Bioorg. Med. Chem. Lett.* **15**, 4169–4173.

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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Stewart, D. J. (2005). Dissertation thesis, University of Mississippi, USA.

supporting information

Acta Cryst. (2009). E**65**, o471–o472 [doi:10.1107/S1600536809002074]

(2*S*,4a*R*,6a*R*,7*R*,9*S*,10a*S*,10b*R*)-7-Carboxy-2-(3-furyl)-6a,10b-dimethyl-4,10-dioxoperhydrobenzo[*f*]isochromen-9-yl acetate

Paulo Carvalho, Lukasz M. Kutrzeba, Jordan K. Zjawiony and Mitchell A. Avery

S1. Comment

Diterpenoid salvinorin A is a potent kappa opioid agonist isolated from Mexican mint *Salvia divinorum*. The biosynthesis of this natural product was studied using synthetic analogues of salvinorin A modified at C-2 and C-18 as chemical probes for *in vitro* enzymatic reactions. Salvinorin A and its C-8 epimeric counterpart revealed low affinity to the kappa-opioid receptor.

S2. Experimental

The starting material for synthesis, salvinorin A, was isolated from dry plant material, and purified according to a previously published procedure (Stewart, 2005). Salvinorin A acid was synthesized following Lee, He *et al.* (2005), by using LiI in pyridine as a selective hydrolyzing agent of C-4 methyl ester.

S3. Refinement

All H atoms were visible in difference maps, but were placed geometrically and treated as riding atoms for refinement, with the following constraints: C—H = 0.93 Å, U_{iso}(H) = 1.2U_{eq}(C) for Csp^2, C—H = 0.98 Å, U_{iso}(H) = 1.2U_{eq}(C) for CH, C—H = 0.97 Å, U_{iso}(H) = 1.2U_{eq}(C) for CH_2, C—H = 0.96 Å, U_{iso}(H) = 1.5U_{eq}(C) CH_3, O—H = 0.82 Å, U_{iso}(H) = 1.5U_{eq}(O) for OH.

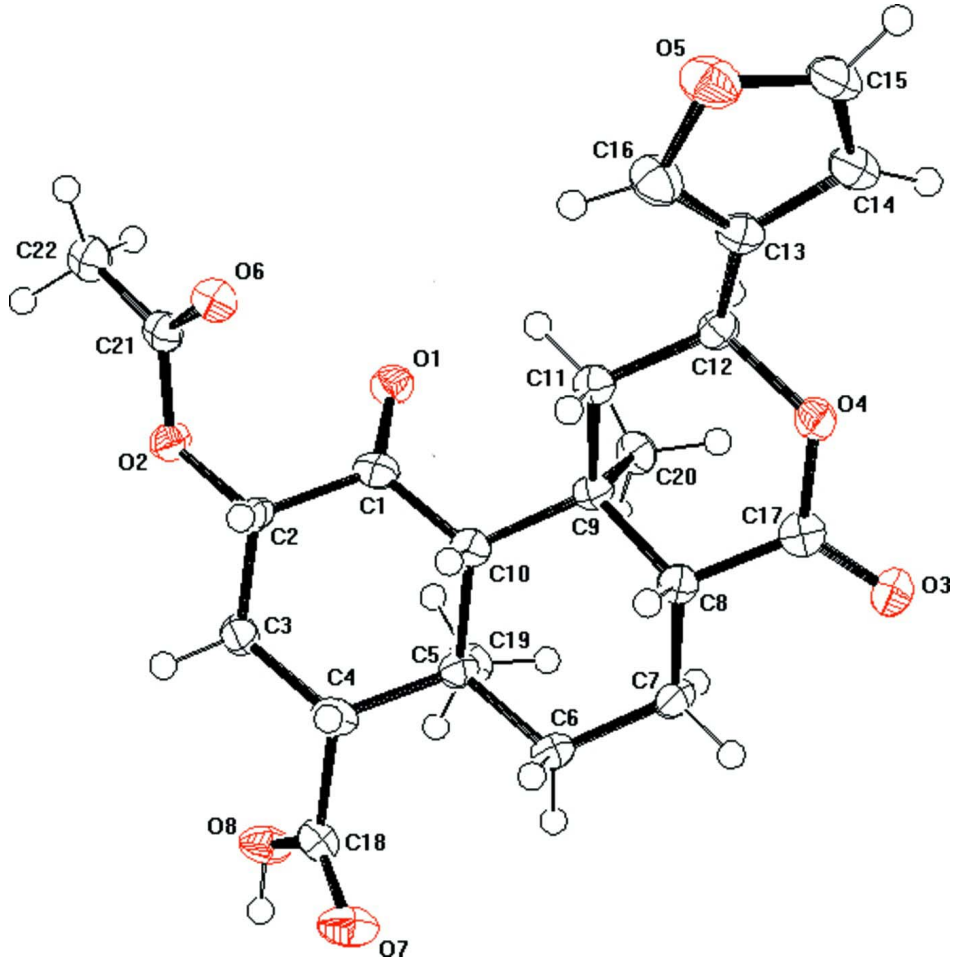

Figure 1

One molecule in the asymmetric unit of the title compound with displacement ellipsoids drawn at the 50% probability level for non-H atoms. The second molecule is closely comparable.

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Crystal data

$C_{22}H_{26}O_8$
$M_r = 418.43$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 11.2735\ (6)$ Å
$b = 16.8015\ (9)$ Å
$c = 11.3765\ (6)$ Å
$\beta = 111.934\ (3)°$
$V = 1998.86\ (18)$ Å^3
$Z = 4$
$F(000) = 888$
$D_x = 1.390$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 9847 reflections
$\theta = 4.2–69.3°$
$\mu = 0.89$ mm^{-1}
$T = 100$ K
Block, colourless
0.19 × 0.12 × 0.09 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube, Siemens KFF Cu 2 K90
Graphite monochromator
φ and ω scans
33644 measured reflections
7269 independent reflections
6962 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 69.7°$, $\theta_{min} = 4.2°$
$h = -13 \rightarrow 13$
$k = -20 \rightarrow 20$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.074$
$S = 1.10$
7269 reflections
549 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0314P)^2 + 0.6446P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}
Absolute structure: Flack (1983), 3360 Friedel pairs
Absolute structure parameter: −0.09 (13)

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.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C17	0.15137 (16)	0.28832 (12)	0.49869 (18)	0.0206 (4)
C21	−0.01037 (17)	0.36406 (10)	−0.21255 (17)	0.0184 (4)
C10	0.05570 (16)	0.40092 (10)	0.18239 (17)	0.0171 (4)
H10	0.1460	0.4079	0.1953	0.021*
C2	0.02643 (18)	0.44739 (11)	−0.03640 (17)	0.0200 (4)
H2	0.1187	0.4446	−0.0167	0.024*
C13	0.22704 (17)	0.12615 (11)	0.32982 (18)	0.0209 (4)
C3	−0.00836 (19)	0.53085 (11)	−0.00734 (19)	0.0216 (4)
H3A	−0.1005	0.5356	−0.0341	0.026*
H3B	0.0208	0.5699	−0.0533	0.026*
C2′	0.52134 (17)	0.21987 (11)	0.57588 (17)	0.0184 (4)
H2′	0.4293	0.2214	0.5260	0.022*
C7	0.07910 (19)	0.43150 (12)	0.44293 (18)	0.0230 (4)
H7A	−0.0087	0.4227	0.4347	0.028*
H7B	0.1289	0.4445	0.5307	0.028*

C1	−0.01037 (16)	0.38607 (10)	0.04262 (18)	0.0184 (4)
C14	0.25349 (19)	0.05608 (11)	0.4068 (2)	0.0260 (4)
H14	0.2213	0.0437	0.4688	0.031*
C6	0.08460 (18)	0.50046 (11)	0.35771 (18)	0.0220 (4)
H6A	0.1734	0.5116	0.3723	0.026*
H6B	0.0490	0.5475	0.3816	0.026*
C20	−0.08067 (17)	0.30143 (11)	0.25474 (18)	0.0200 (4)
H20A	−0.1174	0.2710	0.1782	0.030*
H20B	−0.1339	0.3466	0.2514	0.030*
H20C	−0.0744	0.2688	0.3261	0.030*
C12	0.13705 (17)	0.19141 (11)	0.33016 (17)	0.0198 (4)
H12	0.0513	0.1681	0.3033	0.024*
C5	0.01218 (16)	0.48472 (11)	0.21544 (17)	0.0188 (4)
C8	0.13200 (17)	0.35598 (11)	0.40574 (17)	0.0194 (4)
H8	0.2173	0.3698	0.4079	0.023*
C22	−0.09694 (18)	0.34694 (12)	−0.34524 (18)	0.0229 (4)
H22A	−0.0585	0.3075	−0.3809	0.034*
H22B	−0.1110	0.3949	−0.3946	0.034*
H22C	−0.1772	0.3274	−0.3459	0.034*
C9	0.05392 (16)	0.33013 (11)	0.26855 (17)	0.0177 (4)
C16	0.2942 (2)	0.11820 (13)	0.2545 (2)	0.0297 (4)
H16	0.2956	0.1555	0.1947	0.036*
C11	0.13007 (17)	0.26038 (11)	0.24228 (17)	0.0184 (4)
H11A	0.0888	0.2432	0.1550	0.022*
H11B	0.2158	0.2780	0.2547	0.022*
C18	0.03699 (18)	0.63203 (11)	0.17068 (19)	0.0227 (4)
C19	−0.13340 (17)	0.49154 (11)	0.18207 (19)	0.0220 (4)
H19A	−0.1551	0.4673	0.2478	0.033*
H19B	−0.1781	0.4649	0.1032	0.033*
H19C	−0.1576	0.5467	0.1744	0.033*
C4	0.05507 (17)	0.54636 (11)	0.13572 (19)	0.0202 (4)
H4	0.1472	0.5387	0.1585	0.024*
C15	0.33360 (19)	0.01185 (12)	0.3722 (2)	0.0296 (4)
H15	0.3667	−0.0370	0.4078	0.036*
O6	0.07866 (12)	0.32321 (8)	−0.14752 (12)	0.0217 (3)
O2	−0.04114 (12)	0.43277 (8)	−0.16960 (12)	0.0213 (3)
O4	0.17443 (12)	0.21504 (8)	0.46368 (12)	0.0229 (3)
O1	−0.08635 (12)	0.33368 (8)	−0.00508 (12)	0.0223 (3)
O7	0.11380 (13)	0.66586 (8)	0.25955 (14)	0.0309 (3)
O8	−0.07111 (13)	0.66571 (8)	0.09307 (13)	0.0281 (3)
H8A	−0.0768	0.7108	0.1179	0.042*
O3	0.15754 (12)	0.29677 (8)	0.60630 (12)	0.0246 (3)
O5	0.36018 (14)	0.04804 (9)	0.27771 (16)	0.0341 (3)
O4′	0.33614 (12)	0.44634 (7)	0.94502 (12)	0.0201 (3)
O3′	0.34121 (12)	0.36271 (7)	1.09451 (11)	0.0196 (3)
O2′	0.58986 (12)	0.23688 (7)	0.49500 (12)	0.0196 (3)
O1′	0.62849 (12)	0.33263 (8)	0.69732 (12)	0.0220 (3)
O8′	0.61511 (12)	0.00106 (7)	0.76991 (13)	0.0221 (3)

H8′	0.6248	−0.0429	0.8036	0.033*
O6′	0.45406 (13)	0.33889 (8)	0.41371 (13)	0.0287 (3)
O7′	0.42832 (12)	−0.00338 (7)	0.79601 (14)	0.0253 (3)
O5′	0.12409 (13)	0.60011 (8)	0.65291 (13)	0.0253 (3)
C17′	0.35561 (16)	0.37244 (10)	0.99497 (17)	0.0173 (4)
C9′	0.47726 (16)	0.33266 (10)	0.85748 (16)	0.0166 (4)
C3′	0.55835 (18)	0.13668 (10)	0.63141 (18)	0.0183 (4)
H3′1	0.6498	0.1341	0.6789	0.022*
H3′2	0.5364	0.0977	0.5638	0.022*
C21′	0.54660 (18)	0.30114 (11)	0.41882 (17)	0.0211 (4)
C1′	0.55327 (16)	0.27937 (10)	0.68357 (16)	0.0165 (3)
C13′	0.30056 (17)	0.53252 (10)	0.76765 (17)	0.0184 (4)
C16′	0.17621 (19)	0.54538 (11)	0.74745 (19)	0.0232 (4)
H16′	0.1320	0.5205	0.7917	0.028*
C14′	0.32831 (18)	0.58268 (11)	0.67923 (19)	0.0233 (4)
H14′	0.4063	0.5874	0.6695	0.028*
C12′	0.39011 (17)	0.47249 (11)	0.85204 (17)	0.0185 (4)
H12′	0.4720	0.4988	0.8979	0.022*
C4′	0.48608 (17)	0.11855 (10)	0.71910 (17)	0.0180 (4)
H4′	0.3949	0.1247	0.6677	0.022*
C11′	0.41363 (17)	0.40455 (10)	0.77524 (17)	0.0175 (4)
H11C	0.4677	0.4233	0.7318	0.021*
H11D	0.3327	0.3881	0.7114	0.021*
C5′	0.51693 (16)	0.17822 (10)	0.83185 (17)	0.0170 (4)
C15′	0.22000 (19)	0.62152 (11)	0.61333 (19)	0.0241 (4)
H15′	0.2113	0.6581	0.5493	0.029*
C6′	0.42909 (18)	0.16156 (11)	0.90570 (18)	0.0210 (4)
H6′1	0.3432	0.1515	0.8453	0.025*
H6′2	0.4585	0.1136	0.9555	0.025*
C10′	0.48050 (16)	0.26283 (10)	0.76984 (16)	0.0160 (4)
H10′	0.3914	0.2571	0.7116	0.019*
C22′	0.62893 (19)	0.31710 (12)	0.34423 (19)	0.0250 (4)
H22D	0.5924	0.3596	0.2853	0.037*
H22E	0.6341	0.2700	0.2987	0.037*
H22F	0.7131	0.3320	0.4008	0.037*
C8′	0.38328 (17)	0.30620 (10)	0.91970 (17)	0.0179 (4)
H8′1	0.3022	0.2948	0.8499	0.021*
C19′	0.65936 (17)	0.17049 (12)	0.92042 (18)	0.0225 (4)
H19D	0.6715	0.1930	1.0017	0.034*
H19E	0.7117	0.1984	0.8841	0.034*
H19F	0.6831	0.1153	0.9304	0.034*
C18′	0.50473 (17)	0.03240 (11)	0.76516 (17)	0.0183 (4)
C7′	0.42364 (19)	0.22877 (11)	0.99402 (18)	0.0223 (4)
H7′1	0.3630	0.2153	1.0329	0.027*
H7′2	0.5070	0.2355	1.0609	0.027*
C20′	0.60724 (17)	0.35762 (11)	0.95727 (18)	0.0213 (4)
H20D	0.5946	0.3991	1.0095	0.032*
H20E	0.6615	0.3767	0.9155	0.032*

H20F	0.6467	0.3126	1.0091	0.032*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C17	0.0127 (8)	0.0244 (9)	0.0235 (10)	0.0000 (7)	0.0056 (7)	−0.0009 (8)
C21	0.0201 (9)	0.0183 (9)	0.0200 (9)	−0.0027 (7)	0.0112 (7)	0.0016 (8)
C10	0.0137 (8)	0.0179 (9)	0.0200 (9)	−0.0004 (7)	0.0066 (7)	−0.0011 (7)
C2	0.0213 (9)	0.0199 (9)	0.0175 (9)	0.0011 (7)	0.0057 (7)	−0.0005 (8)
C13	0.0174 (9)	0.0194 (9)	0.0232 (10)	−0.0038 (7)	0.0046 (7)	−0.0038 (8)
C3	0.0238 (9)	0.0183 (9)	0.0231 (9)	0.0012 (7)	0.0092 (7)	0.0023 (8)
C2′	0.0171 (9)	0.0194 (9)	0.0201 (9)	0.0009 (7)	0.0086 (7)	0.0024 (8)
C7	0.0256 (10)	0.0236 (9)	0.0198 (10)	0.0015 (8)	0.0084 (8)	−0.0037 (8)
C1	0.0154 (9)	0.0159 (9)	0.0252 (10)	0.0032 (7)	0.0092 (7)	−0.0010 (8)
C14	0.0279 (10)	0.0199 (9)	0.0272 (10)	−0.0013 (8)	0.0070 (8)	−0.0010 (8)
C6	0.0223 (9)	0.0205 (9)	0.0222 (9)	−0.0002 (7)	0.0069 (7)	−0.0053 (8)
C20	0.0180 (9)	0.0213 (9)	0.0210 (9)	−0.0006 (7)	0.0074 (7)	0.0021 (8)
C12	0.0188 (9)	0.0201 (9)	0.0197 (9)	−0.0014 (7)	0.0062 (7)	−0.0015 (7)
C5	0.0172 (9)	0.0176 (9)	0.0215 (9)	0.0008 (7)	0.0070 (7)	−0.0022 (8)
C8	0.0170 (9)	0.0217 (9)	0.0193 (9)	−0.0005 (7)	0.0064 (7)	0.0009 (8)
C22	0.0222 (9)	0.0272 (10)	0.0217 (9)	−0.0007 (8)	0.0108 (7)	0.0002 (8)
C9	0.0167 (8)	0.0181 (9)	0.0190 (9)	−0.0011 (7)	0.0076 (7)	−0.0012 (8)
C16	0.0296 (11)	0.0256 (10)	0.0369 (12)	0.0056 (8)	0.0160 (9)	0.0047 (9)
C11	0.0165 (8)	0.0203 (9)	0.0188 (9)	−0.0008 (7)	0.0071 (7)	−0.0023 (7)
C18	0.0200 (10)	0.0205 (9)	0.0285 (11)	−0.0002 (7)	0.0101 (8)	0.0022 (8)
C19	0.0185 (9)	0.0209 (9)	0.0286 (10)	0.0019 (7)	0.0111 (8)	−0.0006 (8)
C4	0.0160 (9)	0.0183 (9)	0.0264 (10)	−0.0006 (7)	0.0082 (7)	−0.0015 (8)
C15	0.0293 (11)	0.0166 (9)	0.0384 (12)	0.0013 (8)	0.0074 (9)	−0.0012 (9)
O6	0.0187 (6)	0.0196 (6)	0.0267 (7)	0.0009 (5)	0.0083 (5)	0.0008 (6)
O2	0.0253 (7)	0.0201 (6)	0.0174 (7)	0.0037 (5)	0.0069 (5)	0.0017 (5)
O4	0.0267 (7)	0.0221 (7)	0.0199 (7)	0.0041 (5)	0.0087 (5)	0.0027 (6)
O1	0.0206 (6)	0.0242 (7)	0.0212 (7)	−0.0027 (5)	0.0069 (5)	−0.0010 (6)
O7	0.0258 (7)	0.0225 (7)	0.0362 (8)	−0.0001 (6)	0.0021 (6)	−0.0070 (7)
O8	0.0260 (7)	0.0161 (6)	0.0354 (8)	0.0042 (5)	0.0039 (6)	−0.0037 (6)
O3	0.0232 (7)	0.0309 (7)	0.0199 (7)	0.0024 (5)	0.0084 (5)	0.0012 (6)
O5	0.0321 (8)	0.0276 (8)	0.0475 (9)	0.0056 (6)	0.0203 (7)	−0.0017 (7)
O4′	0.0247 (7)	0.0172 (6)	0.0204 (7)	0.0027 (5)	0.0107 (5)	0.0005 (5)
O3′	0.0228 (7)	0.0198 (6)	0.0177 (6)	−0.0008 (5)	0.0092 (5)	−0.0016 (5)
O2′	0.0233 (7)	0.0178 (6)	0.0203 (7)	0.0031 (5)	0.0112 (5)	0.0027 (5)
O1′	0.0227 (6)	0.0178 (6)	0.0292 (7)	−0.0028 (5)	0.0139 (6)	−0.0011 (6)
O8′	0.0239 (7)	0.0146 (6)	0.0318 (7)	0.0032 (5)	0.0149 (6)	0.0057 (6)
O6′	0.0266 (7)	0.0284 (7)	0.0334 (8)	0.0101 (6)	0.0138 (6)	0.0108 (6)
O7′	0.0249 (7)	0.0172 (6)	0.0395 (8)	−0.0016 (5)	0.0184 (6)	0.0005 (6)
O5′	0.0257 (7)	0.0217 (7)	0.0292 (7)	0.0066 (5)	0.0111 (6)	0.0031 (6)
C17′	0.0132 (8)	0.0174 (9)	0.0187 (9)	−0.0002 (6)	0.0030 (7)	−0.0002 (8)
C9′	0.0165 (8)	0.0155 (8)	0.0183 (9)	−0.0014 (7)	0.0071 (7)	−0.0020 (7)
C3′	0.0212 (9)	0.0148 (8)	0.0200 (9)	0.0003 (7)	0.0088 (7)	−0.0021 (7)
C21′	0.0239 (9)	0.0187 (9)	0.0183 (9)	0.0005 (7)	0.0051 (7)	0.0000 (7)

C1′	0.0151 (8)	0.0146 (8)	0.0190 (9)	0.0037 (7)	0.0055 (7)	0.0043 (7)
C13′	0.0243 (10)	0.0122 (8)	0.0193 (9)	−0.0006 (7)	0.0088 (7)	−0.0045 (7)
C16′	0.0292 (10)	0.0191 (9)	0.0250 (10)	0.0042 (8)	0.0145 (8)	0.0033 (8)
C14′	0.0247 (10)	0.0189 (9)	0.0287 (11)	−0.0008 (8)	0.0126 (8)	0.0007 (8)
C12′	0.0179 (9)	0.0170 (9)	0.0207 (9)	−0.0030 (7)	0.0075 (7)	−0.0018 (7)
C4′	0.0158 (9)	0.0162 (9)	0.0225 (9)	−0.0004 (7)	0.0079 (7)	0.0010 (8)
C11′	0.0192 (9)	0.0163 (8)	0.0193 (9)	−0.0012 (7)	0.0096 (7)	−0.0006 (7)
C5′	0.0180 (9)	0.0141 (8)	0.0187 (9)	0.0006 (7)	0.0069 (7)	0.0005 (7)
C15′	0.0329 (11)	0.0156 (9)	0.0261 (10)	−0.0005 (8)	0.0138 (8)	0.0015 (8)
C6′	0.0273 (10)	0.0149 (8)	0.0230 (9)	0.0016 (7)	0.0121 (8)	0.0030 (8)
C10′	0.0135 (8)	0.0154 (9)	0.0180 (9)	0.0003 (6)	0.0045 (7)	0.0007 (7)
C22′	0.0278 (10)	0.0249 (10)	0.0232 (10)	0.0016 (8)	0.0108 (8)	0.0059 (8)
C8′	0.0172 (8)	0.0178 (9)	0.0194 (9)	−0.0003 (7)	0.0077 (7)	−0.0008 (7)
C19′	0.0233 (10)	0.0189 (9)	0.0219 (10)	0.0037 (7)	0.0045 (7)	−0.0004 (8)
C18′	0.0212 (9)	0.0154 (9)	0.0187 (9)	−0.0017 (7)	0.0079 (7)	−0.0035 (7)
C7′	0.0275 (10)	0.0196 (9)	0.0227 (10)	0.0035 (7)	0.0128 (8)	0.0017 (8)
C20′	0.0198 (9)	0.0190 (9)	0.0242 (10)	−0.0003 (7)	0.0070 (7)	−0.0041 (8)

Geometric parameters (Å, º)

C17—O3	1.208 (2)	C4—H4	0.980
C17—O4	1.349 (2)	C15—O5	1.362 (3)
C17—C8	1.512 (3)	C15—H15	0.930
C21—O6	1.213 (2)	O8—H8A	0.820
C21—O2	1.348 (2)	O4′—C17′	1.349 (2)
C21—C22	1.488 (3)	O4′—C12′	1.471 (2)
C10—C1	1.504 (3)	O3′—C17′	1.214 (2)
C10—C9	1.546 (3)	O2′—C21′	1.356 (2)
C10—C5	1.582 (2)	O1′—C1′	1.202 (2)
C10—H10	0.980	O8′—C18′	1.334 (2)
C2—O2	1.440 (2)	O8′—H8′	0.820
C2—C1	1.523 (3)	O6′—C21′	1.204 (2)
C2—C3	1.525 (3)	O7′—C18′	1.205 (2)
C2—H2	0.980	O5′—C15′	1.366 (2)
C13—C16	1.345 (3)	O5′—C16′	1.370 (2)
C13—C14	1.431 (3)	C17′—C8′	1.506 (2)
C13—C12	1.495 (3)	C9′—C11′	1.532 (2)
C3—C4	1.536 (3)	C9′—C20′	1.539 (2)
C3—H3A	0.970	C9′—C8′	1.543 (2)
C3—H3B	0.970	C9′—C10′	1.549 (2)
C2′—O2′	1.434 (2)	C3′—C4′	1.536 (2)
C2′—C1′	1.517 (3)	C3′—H3′1	0.970
C2′—C3′	1.526 (2)	C3′—H3′2	0.970
C2′—H2′	0.980	C21′—C22′	1.497 (3)
C7—C6	1.527 (3)	C1′—C10′	1.522 (2)
C7—C8	1.527 (3)	C13′—C16′	1.351 (3)
C7—H7A	0.970	C13′—C14′	1.434 (3)
C7—H7B	0.970	C13′—C12′	1.495 (3)

C1—O1	1.206 (2)	C16′—H16′	0.930
C14—C15	1.337 (3)	C14′—C15′	1.340 (3)
C14—H14	0.930	C14′—H14′	0.930
C6—C5	1.539 (2)	C12′—C11′	1.520 (2)
C6—H6A	0.970	C12′—H12′	0.980
C6—H6B	0.970	C4′—C18′	1.527 (2)
C20—C9	1.543 (2)	C4′—C5′	1.561 (3)
C20—H20A	0.960	C4′—H4′	0.980
C20—H20B	0.960	C11′—H11C	0.970
C20—H20C	0.960	C11′—H11D	0.970
C12—O4	1.471 (2)	C5′—C6′	1.545 (2)
C12—C11	1.513 (3)	C5′—C19′	1.550 (2)
C12—H12	0.980	C5′—C10′	1.572 (2)
C5—C19	1.544 (2)	C15′—H15′	0.930
C5—C4	1.567 (3)	C6′—C7′	1.528 (3)
C8—C9	1.541 (2)	C6′—H6′1	0.970
C8—H8	0.980	C6′—H6′2	0.970
C22—H22A	0.960	C10′—H10′	0.980
C22—H22B	0.960	C22′—H22D	0.960
C22—H22C	0.960	C22′—H22E	0.960
C9—C11	1.546 (2)	C22′—H22F	0.960
C16—O5	1.366 (3)	C8′—C7′	1.525 (2)
C16—H16	0.930	C8′—H8′1	0.980
C11—H11A	0.970	C19′—H19D	0.960
C11—H11B	0.970	C19′—H19E	0.960
C18—O7	1.200 (2)	C19′—H19F	0.960
C18—O8	1.335 (2)	C7′—H7′1	0.970
C18—C4	1.527 (3)	C7′—H7′2	0.970
C19—H19A	0.960	C20′—H20D	0.960
C19—H19B	0.960	C20′—H20E	0.960
C19—H19C	0.960	C20′—H20F	0.960

O3—C17—O4	117.51 (17)	C14—C15—H15	124.6
O3—C17—C8	123.97 (17)	O5—C15—H15	124.6
O4—C17—C8	118.25 (15)	C21—O2—C2	114.88 (14)
O6—C21—O2	121.98 (16)	C17—O4—C12	122.60 (14)
O6—C21—C22	126.11 (17)	C18—O8—H8A	109.5
O2—C21—C22	111.91 (16)	C15—O5—C16	105.86 (16)
C1—C10—C9	115.10 (15)	C17′—O4′—C12′	122.14 (14)
C1—C10—C5	108.90 (14)	C21′—O2′—C2′	114.68 (14)
C9—C10—C5	117.10 (15)	C18′—O8′—H8′	109.5
C1—C10—H10	104.8	C15′—O5′—C16′	106.00 (14)
C9—C10—H10	104.8	O3′—C17′—O4′	117.37 (16)
C5—C10—H10	104.8	O3′—C17′—C8′	124.00 (16)
O2—C2—C1	110.60 (14)	O4′—C17′—C8′	118.44 (15)
O2—C2—C3	107.88 (15)	C11′—C9′—C20′	109.58 (14)
C1—C2—C3	110.20 (15)	C11′—C9′—C8′	105.19 (14)
O2—C2—H2	109.4	C20′—C9′—C8′	111.58 (14)

C1—C2—H2	109.4	C11′—C9′—C10′	108.80 (14)
C3—C2—H2	109.4	C20′—C9′—C10′	115.87 (14)
C16—C13—C14	105.66 (17)	C8′—C9′—C10′	105.21 (14)
C16—C13—C12	128.21 (18)	C2′—C3′—C4′	108.73 (14)
C14—C13—C12	126.05 (17)	C2′—C3′—H3′1	109.9
C2—C3—C4	109.27 (15)	C4′—C3′—H3′1	109.9
C2—C3—H3A	109.8	C2′—C3′—H3′2	109.9
C4—C3—H3A	109.8	C4′—C3′—H3′2	109.9
C2—C3—H3B	109.8	H3′1—C3′—H3′2	108.3
C4—C3—H3B	109.8	O6′—C21′—O2′	123.28 (17)
H3A—C3—H3B	108.3	O6′—C21′—C22′	126.00 (17)
O2′—C2′—C1′	111.40 (14)	O2′—C21′—C22′	110.72 (16)
O2′—C2′—C3′	108.58 (14)	O1′—C1′—C2′	122.59 (16)
C1′—C2′—C3′	108.88 (15)	O1′—C1′—C10′	125.18 (16)
O2′—C2′—H2′	109.3	C2′—C1′—C10′	112.22 (14)
C1′—C2′—H2′	109.3	C16′—C13′—C14′	105.75 (16)
C3′—C2′—H2′	109.3	C16′—C13′—C12′	128.77 (17)
C6—C7—C8	109.98 (15)	C14′—C13′—C12′	125.13 (16)
C6—C7—H7A	109.7	C13′—C16′—O5′	110.76 (16)
C8—C7—H7A	109.7	C13′—C16′—H16′	124.6
C6—C7—H7B	109.7	O5′—C16′—H16′	124.6
C8—C7—H7B	109.7	C15′—C14′—C13′	106.67 (17)
H7A—C7—H7B	108.2	C15′—C14′—H14′	126.7
O1—C1—C10	125.78 (17)	C13′—C14′—H14′	126.7
O1—C1—C2	122.15 (17)	O4′—C12′—C13′	107.57 (14)
C10—C1—C2	112.04 (15)	O4′—C12′—C11′	113.40 (14)
C15—C14—C13	106.63 (18)	C13′—C12′—C11′	111.01 (15)
C15—C14—H14	126.7	O4′—C12′—H12′	108.2
C13—C14—H14	126.7	C13′—C12′—H12′	108.2
C7—C6—C5	113.95 (15)	C11′—C12′—H12′	108.2
C7—C6—H6A	108.8	C18′—C4′—C3′	111.86 (14)
C5—C6—H6A	108.8	C18′—C4′—C5′	111.53 (15)
C7—C6—H6B	108.8	C3′—C4′—C5′	113.38 (14)
C5—C6—H6B	108.8	C18′—C4′—H4′	106.5
H6A—C6—H6B	107.7	C3′—C4′—H4′	106.5
C9—C20—H20A	109.5	C5′—C4′—H4′	106.5
C9—C20—H20B	109.5	C12′—C11′—C9′	112.55 (14)
H20A—C20—H20B	109.5	C12′—C11′—H11C	109.1
C9—C20—H20C	109.5	C9′—C11′—H11C	109.1
H20A—C20—H20C	109.5	C12′—C11′—H11D	109.1
H20B—C20—H20C	109.5	C9′—C11′—H11D	109.1
O4—C12—C13	105.53 (14)	H11C—C11′—H11D	107.8
O4—C12—C11	113.56 (14)	C6′—C5′—C19′	110.49 (15)
C13—C12—C11	115.33 (15)	C6′—C5′—C4′	109.82 (14)
O4—C12—H12	107.3	C19′—C5′—C4′	109.82 (14)
C13—C12—H12	107.3	C6′—C5′—C10′	107.14 (14)
C11—C12—H12	107.3	C19′—C5′—C10′	113.65 (14)
C6—C5—C19	110.08 (14)	C4′—C5′—C10′	105.75 (14)

C6—C5—C4	109.75 (15)	C14′—C15′—O5′	110.82 (17)
C19—C5—C4	109.75 (15)	C14′—C15′—H15′	124.6
C6—C5—C10	108.25 (14)	O5′—C15′—H15′	124.6
C19—C5—C10	113.76 (14)	C7′—C6′—C5′	114.53 (15)
C4—C5—C10	105.10 (14)	C7′—C6′—H6′1	108.6
C17—C8—C7	113.59 (15)	C5′—C6′—H6′1	108.6
C17—C8—C9	112.03 (15)	C7′—C6′—H6′2	108.6
C7—C8—C9	112.36 (15)	C5′—C6′—H6′2	108.6
C17—C8—H8	106.1	H6′1—C6′—H6′2	107.6
C7—C8—H8	106.1	C1′—C10′—C9′	115.14 (14)
C9—C8—H8	106.1	C1′—C10′—C5′	109.95 (14)
C21—C22—H22A	109.5	C9′—C10′—C5′	117.47 (14)
C21—C22—H22B	109.5	C1′—C10′—H10′	104.2
H22A—C22—H22B	109.5	C9′—C10′—H10′	104.2
C21—C22—H22C	109.5	C5′—C10′—H10′	104.2
H22A—C22—H22C	109.5	C21′—C22′—H22D	109.5
H22B—C22—H22C	109.5	C21′—C22′—H22E	109.5
C8—C9—C20	111.05 (14)	H22D—C22′—H22E	109.5
C8—C9—C10	106.71 (14)	C21′—C22′—H22F	109.5
C20—C9—C10	114.89 (14)	H22D—C22′—H22F	109.5
C8—C9—C11	105.14 (14)	H22E—C22′—H22F	109.5
C20—C9—C11	109.83 (15)	C17′—C8′—C7′	113.41 (14)
C10—C9—C11	108.72 (14)	C17′—C8′—C9′	111.80 (14)
C13—C16—O5	110.99 (19)	C7′—C8′—C9′	112.71 (15)
C13—C16—H16	124.5	C17′—C8′—H8′1	106.1
O5—C16—H16	124.5	C7′—C8′—H8′1	106.1
C12—C11—C9	110.38 (15)	C9′—C8′—H8′1	106.1
C12—C11—H11A	109.6	C5′—C19′—H19D	109.5
C9—C11—H11A	109.6	C5′—C19′—H19E	109.5
C12—C11—H11B	109.6	H19D—C19′—H19E	109.5
C9—C11—H11B	109.6	C5′—C19′—H19F	109.5
H11A—C11—H11B	108.1	H19D—C19′—H19F	109.5
O7—C18—O8	123.35 (18)	H19E—C19′—H19F	109.5
O7—C18—C4	122.76 (17)	O7′—C18′—O8′	123.29 (17)
O8—C18—C4	113.89 (16)	O7′—C18′—C4′	123.43 (16)
C5—C19—H19A	109.5	O8′—C18′—C4′	113.26 (15)
C5—C19—H19B	109.5	C8′—C7′—C6′	110.04 (15)
H19A—C19—H19B	109.5	C8′—C7′—H7′1	109.7
C5—C19—H19C	109.5	C6′—C7′—H7′1	109.7
H19A—C19—H19C	109.5	C8′—C7′—H7′2	109.7
H19B—C19—H19C	109.5	C6′—C7′—H7′2	109.7
C18—C4—C3	112.41 (16)	H7′1—C7′—H7′2	108.2
C18—C4—C5	111.84 (15)	C9′—C20′—H20D	109.5
C3—C4—C5	112.27 (15)	C9′—C20′—H20E	109.5
C18—C4—H4	106.6	H20D—C20′—H20E	109.5
C3—C4—H4	106.6	C9′—C20′—H20F	109.5
C5—C4—H4	106.6	H20D—C20′—H20F	109.5
C14—C15—O5	110.86 (18)	H20E—C20′—H20F	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O8—H8*A*···O6^{i}	0.82	1.92	2.7264 (18)	168
O8′—H8′···O3′^{ii}	0.82	1.92	2.7308 (17)	172

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