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2-*C*-Benzyloxymethyl-2,3:5,6-di-*O*-isopropylidene-D-allono-1,4-lactone

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Key indicators

Single-crystal X-ray study
T = 190 K
Mean σ(C–C) = 0.004 Å
R factor = 0.049
wR factor = 0.097
Data-to-parameter ratio = 11.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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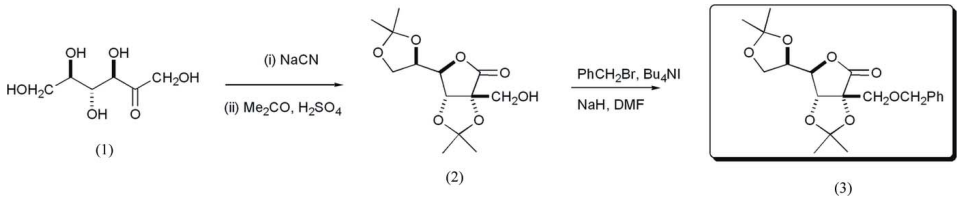
The crystal structure of the title benzyl ether, $C_{20}H_{26}O_7$, establishes the stereochemistry of the major diacetonide product from the Kiliani reaction of D-psicose. There are two independent molecules in the cell related by a pseudo-twofold screw axis. There are no suitable donors for strong hydrogen bonds: the molecules are simply in van der Waals contact.

Comment

Almost all of the readily available carbohydrate building blocks have linear carbon chains (Bols, 1996; Lichtenthaler & Peters, 2004). However, the Kiliani reaction on ketohexoses provides branched sugar lactones bearing a hydroxymethyl substituent at C-2. Acetonation of the crude mixtures of the resulting lactones gives reasonable yields of crystalline diacetonides (Hotchkiss *et al.*, 2004) which are suitable chiral starting materials for the synthesis of branched sugar mimics (Simone *et al.*, 2005). Ambiguities in the structures of the acetonides arise from (i) the stereochemistry at the quaternary carbon and (ii) the ring size of the ketal protecting groups. The structures of diacetonides derived from three of the four diastereomeric ketohexoses [D-fructose (Cowley *et al.*, 2004; van Ameijde *et al.*, 2004), L-sorbose (Anderson *et al.*, 1977) and D-tagatose (Harding *et al.*, 2005; Shallard-Brown *et al.*, 2004)] have been firmly established by X-ray crystallographic analysis. The fourth diastereomeric ketohexose D-psicose (1) is available from equilibration of D-fructose by D-tagatose 3-epimerase (Granstrom *et al.*, 2004; Izumori, 2002; Takeshita *et al.*, 2000; Itoh & Izumori, 1996).



(1) (2) (3)

The sequential treatment of D-psicose (1) with sodium cyanide, followed by extraction of the crude lactones with acetone in the presence of sulfuric acid gave, as the major product (Soengas *et al.*, 2005), a diacetonide tentatively assigned as structure (2); although the *cis*-fused diacetonide (2) was easily crystallized, the crystals were not suitable for X-ray crystallographic analysis. However, reaction of (2) with benzyl bromide and sodium hydride in the presence of tetra-n-butylammonium iodide in dimethylformamide afforded the corresponding benzyl ether (3), which formed crystals for X-;ray suitable analysis. This paper reports the crystal structure of (3) (Fig. 1) which unequivocally establishes the stereo-

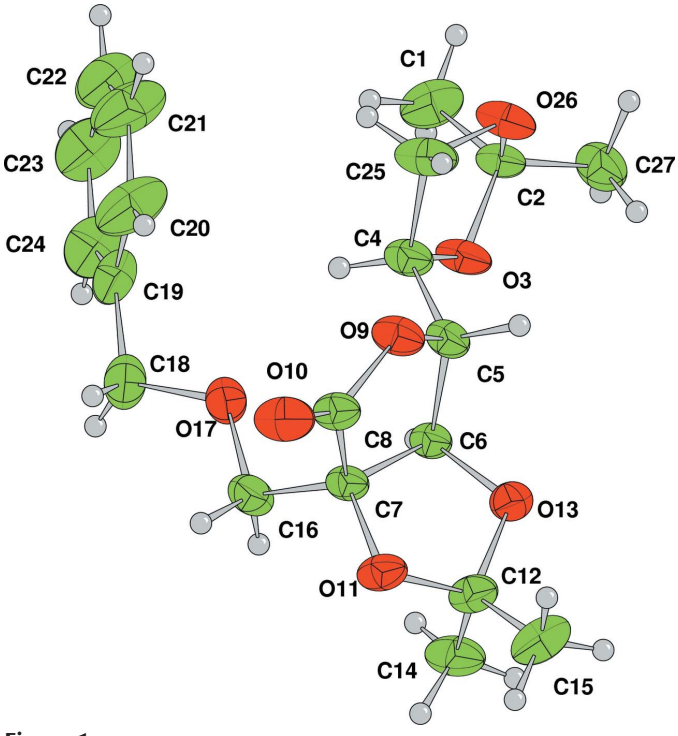


Figure 1
View of one of the two independent molecules in the asymmetric unit, with atomic displacement ellipsoids drawn at the 50% probability level. H-atom radii are arbitary.

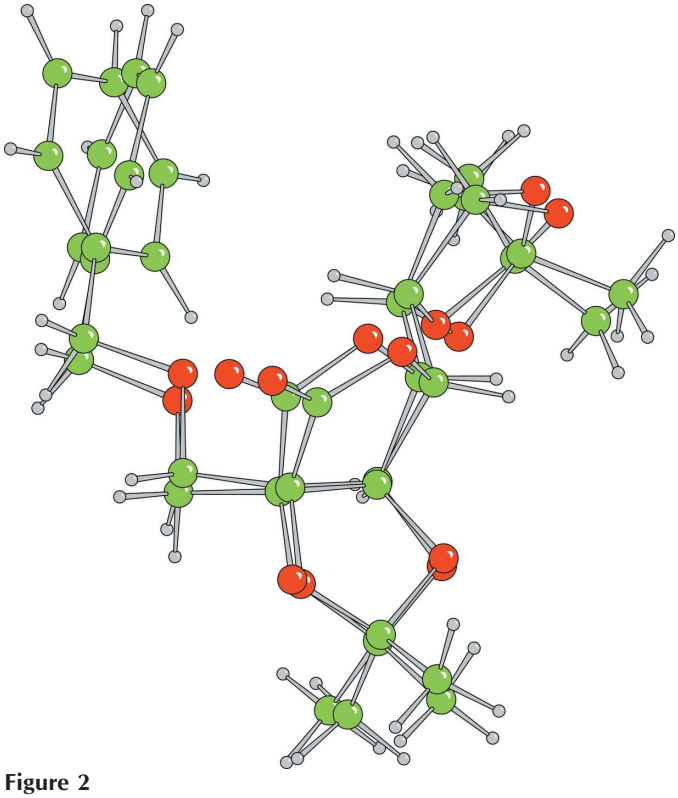


Figure 2
View of the two independent molecules superimposed to give the least-squares best fit between the atom coordinates.

chemistry of the major lactone product (2) from the Kiliani–acetonation sequence on D-psicose.

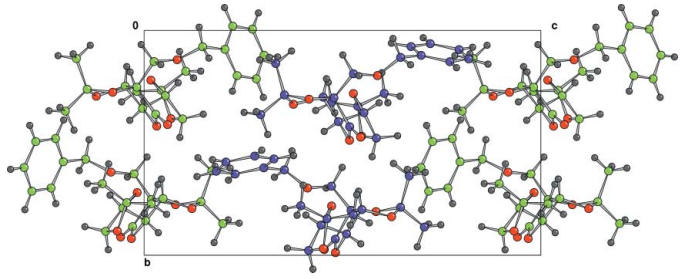


Figure 3
An *a* axis projection of the title compound. The C atoms in one of the independent molecules are coloured green, those in the other blue.

The crystals were in the form of large fragile prisms which crushed easily. A long sample was eventually selected and mounted on a nylon loop using perfluoropolyether oil so that the damaged end of the crystal was outside the X-ray beam. By mounting the crystal approximately parallel to the φ axis, the changes in illuminated volume were kept to a minimum, and were taken into account (Görbitz, 1999) by the multi-scan inter-frame scaling (*DENZO*/*SCALEPACK*, Otwinowski & Minor, 1997).

There are two independent molecules in the asymmetric unit related by a pseudo-twofold screw axis ($0.51 - x, 0.46 + y, 1.50 - z$). The molecules differ mainly in the orientation of the phenyl group (Fig. 2). The structure contains no hydrogen bonds and consists of layers loosely packed perpendicular to the *c* axis (Fig. 3). Alternate layers contain only molecules of one type.

Experimental

The benzyl ether (3) was crystallized from ethyl acetate–cyclohexane (m.p. 345–346 K); $[\alpha]_{22}^{D} - 30.0$ (*c*, 1.06 in chloroform); ν_{max} (NaCl): 1781 (–C═O) cm-1; ^{1}H NMR ($CDCl_3$): δ 1.27, 1.40, 1.46 [3 × *s*, 12 H, 2 × –C(CH_3)$_2$], 3.73 (*d*, 1H, $J_{2',2''}$ 9.0 Hz, H2′), 3.92–3.97 (*m*, 3H, H6, H6?, H4), 4.12–4.15 (*m*, 1 H, H5), 4.29 (*d*, 1H, $J_{2',2''}$ 9.0 Hz, H2″), 4.57 (*s*, 2H, –OCH_2Ph), 4.85 (*s*, 1H, H3); ^{13}C NMR ($CDCl_3$): δ 24.60, 26.07, 26.60, 26.63 [2 × –C(CH_3)$_2$], 66.47 (C2'), 69.34 (C6), 73.37 (C5), 73.99 (–OCH_2Ph), 80.05 (C3), 83.13 (C4), 83.70 (C2), 109.91, 113.33 [2 × –C(CH_3)$_2$], 127.76, 127.91, 128.29 (–CHAr), 136.70 (–CAr) 174.56 (C1); *m*/*z* (NH_3, ES+): 401 $(M + Na)^+$.

Crystal data

$C_{20}H_{26}O_7$
M_r = 378.42
Monoclinic, $P2_1$
a = 10.3839 (2) Å
b = 10.4574 (2) Å
c = 19.0310 (5) Å
β = 103.8061 (9)°
V = 2006.84 (8) Å^3
Z = 4
D_x = 1.252 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 3433 reflections
θ = 1–30°
μ = 0.09 mm^{-1}
T = 190 K
Prism, colourless
1.20 × 0.75 × 0.45 mm

Data collection

Nonius Kappa CCD diffractometer
ω scans
Absorption correction: multi-scan (*DENZO*/*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.81, T_{max} = 0.96
11499 measured reflections
5664 independent reflections
5635 reflections with $I > -3\sigma(I)$
R_{int} = 0.027
θ_{max} = 30.1°
h = −14 → 14
k = −7 → 14
l = −26 → 26

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F^2) + (0.03P)^2 + 0.6P]$ where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$R[F^2 > 2\sigma(F^2)] = 0.049$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.097$	$\Delta\rho_{max} = 0.23$ e Å^{-3}
$S = 0.95$	$\Delta\rho_{min} = -0.17$ e Å^{-3}
5635 reflections	
487 parameters	
H-atom parameters constrained	

The H atoms were all located in a difference map, but those attached to C atoms were repositioned geometrically. The H atoms were initially refined with soft restraints on the bond lengths and angles to regularize their geometry (C—H = 0.93–98 Å) and isotropic displacement parameters [U_{iso}(H) = 1.2–1.5U_{eq}(C)], after which they were refined with riding constraints. In the absence of significant anomalous dispersion effects, Friedel pairs were averaged.

Data collection: *COLLECT* (Nonius, 2001); cell refinement: *DENZO*/*SCALEPACK*; data reduction: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR92* (Altomare *et al*., 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al*., 2003); molecular graphics: *CAMERON* (Watkin *et al*., 1996); software used to prepare material for publication: *CRYSTALS*.

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supporting information

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$V = 2006.84$ (8) Å^3
$Z = 4$
$F(000) = 808$
$D_x = 1.252$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3433 reflections
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$\mu = 0.09$ mm^{-1}
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$\theta_{max} = 30.1°$, $\theta_{min} = 1.1°$
$h = -14 \rightarrow 14$
$k = -7 \rightarrow 14$
$l = -26 \rightarrow 26$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.097$
$S = 0.95$
5635 reflections
487 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F^2) + (0.03P)^2 + 0.6P]$ where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.6912 (3)	0.6817 (3)	0.89496 (16)	0.0613
C2	0.6732 (2)	0.7661 (2)	0.95659 (12)	0.0358
O3	0.56299 (14)	0.72111 (16)	0.98275 (10)	0.0429
C4	0.4476 (2)	0.7902 (2)	0.94647 (13)	0.0393

C5	0.3862 (2)	0.8461 (2)	1.00390 (12)	0.0342
C6	0.32997 (19)	0.74764 (19)	1.04783 (11)	0.0309
C7	0.17916 (19)	0.7523 (2)	1.01840 (11)	0.0312
C8	0.1574 (2)	0.8728 (2)	0.97093 (12)	0.0351
O9	0.27461 (15)	0.92551 (15)	0.96805 (9)	0.0397
O10	0.05341 (16)	0.9189 (2)	0.94083 (10)	0.0526
O11	0.12530 (14)	0.77517 (17)	1.07923 (8)	0.0389
H11	0.7692	0.7111	0.8752	0.1072*
C12	0.2311 (2)	0.7618 (2)	1.14340 (12)	0.0385
H12	0.7122	0.5935	0.9107	0.1072*
O13	0.34809 (15)	0.79243 (17)	1.12045 (8)	0.0389
H13	0.6153	0.6811	0.8572	0.1070*
C14	0.2355 (3)	0.6250 (3)	1.17066 (15)	0.0499
C15	0.2127 (3)	0.8590 (3)	1.19836 (15)	0.0595
C16	0.1155 (2)	0.6383 (2)	0.97486 (13)	0.0409
O17	0.17799 (16)	0.63022 (18)	0.91661 (9)	0.0475
C18	0.1019 (3)	0.5727 (3)	0.85246 (14)	0.0523
C19	0.1805 (3)	0.5838 (2)	0.79633 (13)	0.0493
C20	0.1947 (4)	0.7012 (3)	0.76556 (17)	0.0745
C21	0.2743 (5)	0.7150 (4)	0.71718 (19)	0.0858
C22	0.3377 (4)	0.6098 (4)	0.69765 (17)	0.0723
C23	0.3234 (4)	0.4942 (3)	0.72696 (19)	0.0731
C24	0.2461 (3)	0.4807 (3)	0.77668 (17)	0.0645
C25	0.5008 (2)	0.8934 (3)	0.90304 (16)	0.0541
O26	0.63821 (16)	0.89243 (16)	0.93251 (10)	0.0468
C27	0.7940 (2)	0.7712 (3)	1.01804 (15)	0.0502
H41	0.3846	0.7327	0.9142	0.0519*
H51	0.4571	0.8998	1.0360	0.0470*
H61	0.3665	0.6607	1.0437	0.0466*
C101	−0.1863 (3)	0.2800 (3)	0.60522 (15)	0.0542
C102	−0.1537 (2)	0.3374 (2)	0.53881 (12)	0.0337
O103	−0.03441 (14)	0.28084 (15)	0.52740 (10)	0.0430
C104	0.0740 (2)	0.3658 (2)	0.55386 (13)	0.0365
C105	0.1294 (2)	0.40541 (19)	0.48991 (13)	0.0371
C106	0.18060 (19)	0.29669 (19)	0.45118 (11)	0.0303
C107	0.33235 (19)	0.30000 (18)	0.47837 (11)	0.0296
C108	0.3581 (2)	0.4284 (2)	0.51802 (12)	0.0360
O109	0.24325 (16)	0.48850 (14)	0.51772 (10)	0.0434
O110	0.46329 (17)	0.47463 (18)	0.54578 (10)	0.0520
O111	0.38350 (15)	0.30560 (16)	0.41557 (8)	0.0392
C112	0.2743 (2)	0.2868 (2)	0.35378 (12)	0.0404
O113	0.16033 (17)	0.32835 (17)	0.37628 (9)	0.0437
C114	0.2634 (3)	0.1469 (2)	0.33179 (14)	0.0505
C115	0.2938 (3)	0.3743 (3)	0.29361 (16)	0.0636
C116	0.3952 (2)	0.1928 (2)	0.52760 (11)	0.0325
O117	0.33834 (14)	0.20092 (15)	0.58821 (8)	0.0367
C118	0.3999 (2)	0.1202 (2)	0.64651 (11)	0.0358
C119	0.3113 (2)	0.11102 (19)	0.69815 (12)	0.0336

C120	0.3578 (2)	0.1389 (2)	0.77052 (12)	0.0412
C121	0.2762 (3)	0.1263 (3)	0.81823 (14)	0.0526
C122	0.1479 (3)	0.0854 (3)	0.79376 (16)	0.0584
C123	0.0992 (3)	0.0588 (3)	0.72118 (18)	0.0561
C124	0.1804 (2)	0.0716 (2)	0.67285 (14)	0.0436
C125	0.0123 (2)	0.4789 (2)	0.58536 (15)	0.0459
O126	−0.12454 (15)	0.47056 (15)	0.54808 (9)	0.0398
C127	−0.2637 (2)	0.3203 (2)	0.47264 (14)	0.0447
H141	0.2995	0.6122	1.2168	0.0839*
H142	0.1494	0.6041	1.1787	0.0838*
H143	0.2526	0.5660	1.1356	0.0836*
H151	0.2850	0.8572	1.2396	0.1017*
H152	0.1340	0.8423	1.2121	0.1012*
H153	0.2073	0.9437	1.1765	0.1018*
H161	0.1271	0.5628	1.0073	0.0582*
H162	0.0161	0.6520	0.9578	0.0579*
H181	0.0150	0.6230	0.8367	0.0714*
H182	0.0768	0.4857	0.8613	0.0722*
H201	0.1497	0.7703	0.7774	0.0980*
H211	0.2841	0.7975	0.6995	0.1165*
H221	0.3878	0.6180	0.6634	0.0961*
H231	0.3613	0.4222	0.7119	0.1003*
H241	0.2398	0.3982	0.7972	0.0892*
H251	0.4621	0.9758	0.9098	0.0691*
H252	0.4751	0.8727	0.8509	0.0691*
H271	0.7674	0.8179	1.0567	0.0819*
H272	0.8637	0.8151	1.0007	0.0826*
H273	0.8236	0.6850	1.0351	0.0821*
H1011	−0.2671	0.3219	0.6144	0.0981*
H1012	−0.1117	0.2928	0.6452	0.0979*
H1013	−0.2008	0.1881	0.5992	0.0982*
H1041	0.1422	0.3215	0.5924	0.0478*
H1051	0.0575	0.4492	0.4538	0.0514*
H1061	0.1396	0.2149	0.4596	0.0428*
H1141	0.3410	0.1196	0.3155	0.0940*
H1142	0.2614	0.0916	0.3717	0.0936*
H1143	0.1889	0.1311	0.2915	0.0936*
H1151	0.3760	0.3612	0.2829	0.1135*
H1152	0.2926	0.4627	0.3115	0.1137*
H1153	0.2242	0.3646	0.2522	0.1139*
H1161	0.4930	0.2011	0.5413	0.0455*
H1162	0.3764	0.1091	0.5006	0.0447*
H1181	0.4899	0.1526	0.6716	0.0480*
H1182	0.4170	0.0356	0.6273	0.0484*
H1201	0.4422	0.1657	0.7873	0.0534*
H1211	0.3084	0.1499	0.8666	0.0663*
H1221	0.0969	0.0750	0.8277	0.0864*
H1231	0.0120	0.0326	0.7035	0.0778*

H1241	0.1458	0.0568	0.6234	0.0614*
H1251	0.0319	0.4686	0.6389	0.0615*
H1252	0.0532	0.5618	0.5746	0.0617*
H1271	−0.3419	0.3661	0.4805	0.0712*
H1272	−0.2812	0.2311	0.4641	0.0709*
H1273	−0.2314	0.3561	0.4323	0.0713*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.078 (2)	0.0607 (17)	0.0530 (16)	0.0116 (15)	0.0308 (15)	0.0019 (13)
C2	0.0313 (10)	0.0382 (10)	0.0431 (11)	0.0027 (8)	0.0194 (9)	0.0076 (9)
O3	0.0283 (7)	0.0490 (9)	0.0559 (10)	0.0048 (6)	0.0189 (7)	0.0216 (8)
C4	0.0284 (9)	0.0488 (12)	0.0433 (11)	0.0027 (9)	0.0136 (9)	0.0113 (10)
C5	0.0257 (9)	0.0366 (10)	0.0410 (11)	−0.0003 (7)	0.0093 (8)	0.0066 (8)
C6	0.0271 (9)	0.0338 (9)	0.0334 (10)	0.0020 (7)	0.0102 (8)	0.0052 (8)
C7	0.0254 (8)	0.0364 (9)	0.0339 (10)	0.0029 (7)	0.0114 (7)	0.0049 (8)
C8	0.0312 (10)	0.0398 (10)	0.0368 (11)	0.0073 (8)	0.0131 (8)	0.0074 (8)
O9	0.0354 (8)	0.0352 (7)	0.0514 (9)	0.0055 (6)	0.0163 (7)	0.0139 (7)
O10	0.0369 (9)	0.0685 (12)	0.0545 (10)	0.0182 (8)	0.0147 (8)	0.0266 (9)
O11	0.0327 (7)	0.0530 (9)	0.0346 (7)	0.0110 (7)	0.0147 (6)	0.0101 (7)
C12	0.0371 (10)	0.0473 (12)	0.0330 (10)	0.0087 (9)	0.0124 (8)	0.0100 (9)
O13	0.0347 (7)	0.0498 (9)	0.0324 (7)	−0.0016 (7)	0.0082 (6)	0.0048 (7)
C14	0.0446 (13)	0.0526 (14)	0.0568 (15)	0.0087 (11)	0.0204 (12)	0.0253 (12)
C15	0.080 (2)	0.0647 (17)	0.0388 (13)	0.0124 (15)	0.0235 (13)	0.0008 (12)
C16	0.0331 (11)	0.0443 (12)	0.0484 (13)	−0.0063 (9)	0.0156 (10)	−0.0027 (10)
O17	0.0386 (8)	0.0621 (11)	0.0432 (9)	−0.0114 (8)	0.0124 (7)	−0.0155 (8)
C18	0.0537 (14)	0.0534 (14)	0.0440 (13)	−0.0082 (12)	0.0000 (11)	−0.0052 (11)
C19	0.0648 (16)	0.0427 (12)	0.0341 (12)	0.0026 (11)	−0.0003 (11)	−0.0018 (9)
C20	0.128 (3)	0.0489 (15)	0.0520 (17)	0.0237 (18)	0.0317 (19)	0.0098 (13)
C21	0.148 (4)	0.061 (2)	0.0553 (19)	0.007 (2)	0.039 (2)	0.0144 (16)
C22	0.097 (3)	0.076 (2)	0.0456 (16)	0.0005 (19)	0.0204 (16)	−0.0011 (15)
C23	0.089 (2)	0.0637 (19)	0.068 (2)	0.0196 (18)	0.0212 (18)	−0.0010 (16)
C24	0.084 (2)	0.0454 (14)	0.0615 (18)	0.0088 (14)	0.0117 (16)	0.0011 (13)
C25	0.0395 (12)	0.0686 (17)	0.0599 (16)	0.0118 (12)	0.0230 (12)	0.0316 (14)
O26	0.0361 (8)	0.0436 (9)	0.0670 (12)	0.0043 (7)	0.0249 (8)	0.0207 (8)
C27	0.0363 (11)	0.0541 (14)	0.0593 (15)	0.0000 (11)	0.0094 (11)	0.0084 (12)
C101	0.0612 (16)	0.0611 (15)	0.0474 (14)	0.0054 (13)	0.0267 (12)	0.0070 (12)
C102	0.0303 (10)	0.0327 (9)	0.0409 (11)	0.0050 (7)	0.0139 (8)	−0.0020 (8)
O103	0.0314 (7)	0.0345 (7)	0.0682 (11)	0.0012 (6)	0.0221 (7)	−0.0129 (8)
C104	0.0286 (9)	0.0349 (10)	0.0476 (12)	0.0033 (8)	0.0123 (9)	−0.0044 (9)
C105	0.0321 (10)	0.0272 (9)	0.0524 (13)	0.0042 (8)	0.0107 (9)	0.0002 (9)
C106	0.0284 (9)	0.0278 (8)	0.0348 (10)	0.0006 (7)	0.0076 (8)	0.0029 (7)
C107	0.0284 (9)	0.0292 (9)	0.034 (1)	−0.0004 (7)	0.0133 (8)	0.0033 (7)
C108	0.0369 (11)	0.0317 (9)	0.0430 (11)	−0.0067 (8)	0.0165 (9)	0.0016 (8)
O109	0.0423 (9)	0.0258 (7)	0.0646 (11)	−0.0037 (6)	0.0180 (8)	−0.0051 (7)
O110	0.0431 (9)	0.0531 (10)	0.0629 (12)	−0.0206 (8)	0.0187 (8)	−0.0122 (9)
O111	0.0383 (8)	0.0483 (9)	0.0354 (8)	−0.0037 (7)	0.0177 (7)	0.0062 (6)

C112	0.0479 (12)	0.0412 (11)	0.0338 (10)	−0.0025 (10)	0.0130 (9)	0.0056 (9)
O113	0.0428 (9)	0.0503 (9)	0.0357 (8)	0.0075 (7)	0.0049 (7)	0.0058 (7)
C114	0.0631 (16)	0.0447 (13)	0.0480 (14)	−0.0056 (11)	0.0220 (12)	−0.0037 (11)
C115	0.092 (2)	0.0594 (17)	0.0422 (14)	−0.0056 (16)	0.0214 (14)	0.0162 (12)
C116	0.0292 (9)	0.0361 (9)	0.0354 (10)	0.0062 (7)	0.0141 (8)	0.0056 (8)
O117	0.0380 (8)	0.0414 (8)	0.0346 (7)	0.0132 (6)	0.0163 (6)	0.0118 (6)
C118	0.0330 (10)	0.0402 (10)	0.0331 (10)	0.0080 (8)	0.0056 (8)	0.0072 (8)
C119	0.0369 (10)	0.0304 (9)	0.0338 (10)	0.0053 (8)	0.0091 (8)	0.0066 (8)
C120	0.0389 (11)	0.0478 (12)	0.0345 (11)	0.0105 (9)	0.0038 (9)	0.0045 (9)
C121	0.0577 (15)	0.0671 (16)	0.0345 (11)	0.0239 (13)	0.0142 (11)	0.0094 (11)
C122	0.0568 (16)	0.0704 (18)	0.0559 (16)	0.0205 (14)	0.0294 (14)	0.0281 (14)
C123	0.0424 (13)	0.0590 (16)	0.0701 (19)	−0.0047 (12)	0.0200 (13)	0.0119 (14)
C124	0.0415 (12)	0.0479 (12)	0.0411 (12)	−0.0063 (10)	0.0097 (10)	0.0014 (10)
C125	0.0308 (10)	0.0470 (12)	0.0586 (14)	0.0041 (9)	0.0081 (10)	−0.0192 (11)
O126	0.0301 (7)	0.0334 (7)	0.0569 (10)	0.0032 (6)	0.0122 (7)	−0.0111 (7)
C127	0.0435 (12)	0.0371 (11)	0.0496 (13)	−0.0023 (9)	0.0036 (10)	0.0009 (10)

Geometric parameters (Å, º)

C1—C2	1.515 (4)	C101—C102	1.509 (3)
C1—H11	1.018	C101—H1011	0.999
C1—H12	0.978	C101—H1012	0.957
C1—H13	0.932	C101—H1013	0.975
C2—O3	1.432 (2)	C102—O103	1.435 (2)
C2—O26	1.417 (3)	C102—O126	1.427 (3)
C2—C27	1.498 (3)	C102—C127	1.496 (3)
O3—C4	1.428 (3)	O103—C104	1.428 (3)
C4—C5	1.509 (3)	C104—C105	1.522 (3)
C4—C25	1.540 (3)	C104—C125	1.534 (3)
C4—H41	0.987	C104—H1041	1.003
C5—C6	1.527 (3)	C105—C106	1.518 (3)
C5—O9	1.456 (3)	C105—O109	1.461 (3)
C5—H51	1.008	C105—H1051	0.998
C6—C7	1.533 (3)	C106—C107	1.537 (3)
C6—O13	1.429 (2)	C106—O113	1.429 (2)
C6—H61	0.995	C106—H1061	0.985
C7—C8	1.535 (3)	C107—C108	1.532 (3)
C7—O11	1.421 (2)	C107—O111	1.420 (2)
C7—C16	1.511 (3)	C107—C116	1.506 (3)
C8—O9	1.349 (2)	C108—O109	1.347 (3)
C8—O10	1.197 (3)	C108—O110	1.196 (3)
O11—C12	1.441 (3)	O111—C112	1.439 (3)
C12—O13	1.422 (2)	C112—O113	1.419 (3)
C12—C14	1.518 (3)	C112—C114	1.518 (3)
C12—C15	1.503 (4)	C112—C115	1.517 (3)
C14—H141	0.976	C114—H1141	0.974
C14—H142	0.967	C114—H1142	0.959
C14—H143	0.956	C114—H1143	0.964

C15—H151	0.948	C115—H1151	0.934
C15—H152	0.933	C115—H1152	0.986
C15—H153	0.974	C115—H1153	0.939
C16—O17	1.414 (3)	C116—O117	1.418 (2)
C16—H161	0.992	C116—H1161	0.991
C16—H162	1.016	C116—H1162	1.010
O17—C18	1.420 (3)	O117—C118	1.419 (2)
C18—C19	1.496 (4)	C118—C119	1.501 (3)
C18—H181	1.025	C118—H1181	1.001
C18—H182	0.972	C118—H1182	0.989
C19—C20	1.383 (4)	C119—C120	1.377 (3)
C19—C24	1.374 (4)	C119—C124	1.392 (3)
C20—C21	1.384 (5)	C120—C121	1.389 (3)
C20—H201	0.917	C120—H1201	0.902
C21—C22	1.378 (5)	C121—C122	1.371 (4)
C21—H211	0.941	C121—H1211	0.934
C22—C23	1.354 (5)	C122—C123	1.381 (4)
C22—H221	0.930	C122—H1221	0.934
C23—C24	1.386 (5)	C123—C124	1.395 (3)
C23—H231	0.927	C123—H1231	0.929
C24—H241	0.955	C124—H1241	0.937
C25—O26	1.403 (3)	C125—O126	1.431 (3)
C25—H251	0.973	C125—H1251	0.996
C25—H252	0.989	C125—H1252	1.008
C27—H271	0.976	C127—H1271	0.984
C27—H272	0.978	C127—H1272	0.957
C27—H273	0.983	C127—H1273	0.983

C2—C1—H11	111.4	C102—C101—H1011	109.8
C2—C1—H12	111.8	C102—C101—H1012	108.0
H11—C1—H12	105.1	H1011—C101—H1012	110.8
C2—C1—H13	111.3	C102—C101—H1013	110.7
H11—C1—H13	108.6	H1011—C101—H1013	109.8
H12—C1—H13	108.4	H1012—C101—H1013	107.7
C1—C2—O3	109.6 (2)	C101—C102—O103	109.69 (19)
C1—C2—O26	111.2 (2)	C101—C102—O126	111.6 (2)
O3—C2—O26	104.72 (16)	O103—C102—O126	104.83 (16)
C1—C2—C27	113.0 (2)	C101—C102—C127	112.2 (2)
O3—C2—C27	109.27 (19)	O103—C102—C127	109.69 (18)
O26—C2—C27	108.7 (2)	O126—C102—C127	108.53 (18)
C2—O3—C4	108.29 (16)	C102—O103—C104	109.05 (15)
O3—C4—C5	107.26 (19)	O103—C104—C105	107.69 (19)
O3—C4—C25	104.32 (16)	O103—C104—C125	104.46 (16)
C5—C4—C25	112.6 (2)	C105—C104—C125	112.05 (19)
O3—C4—H41	110.4	O103—C104—H1041	109.3
C5—C4—H41	110.7	C105—C104—H1041	112.0
C25—C4—H41	111.2	C125—C104—H1041	111.0
C4—C5—C6	114.72 (18)	C104—C105—C106	115.28 (17)

C4—C5—O9	107.97 (18)	C104—C105—O109	107.44 (19)
C6—C5—O9	106.00 (15)	C106—C105—O109	105.91 (16)
C4—C5—H51	106.3	C104—C105—H1051	108.2
C6—C5—H51	111.3	C106—C105—H1051	107.5
O9—C5—H51	110.5	O109—C105—H1051	112.6
C5—C6—C7	105.60 (16)	C105—C106—C107	105.63 (16)
C5—C6—O13	109.23 (17)	C105—C106—O113	108.98 (17)
C7—C6—O13	103.30 (15)	C107—C106—O113	103.04 (15)
C5—C6—H61	111.7	C105—C106—H1061	110.9
C7—C6—H61	112.1	C107—C106—H1061	114.4
O13—C6—H61	114.2	O113—C106—H1061	113.4
C6—C7—C8	103.62 (16)	C106—C107—C108	103.30 (16)
C6—C7—O11	105.91 (16)	C106—C107—O111	106.07 (16)
C8—C7—O11	108.09 (16)	C108—C107—O111	108.78 (15)
C6—C7—C16	116.74 (17)	C106—C107—C116	116.72 (15)
C8—C7—C16	109.94 (18)	C108—C107—C116	110.32 (18)
O11—C7—C16	111.94 (16)	O111—C107—C116	111.13 (15)
C7—C8—O9	110.60 (17)	C107—C108—O109	110.89 (17)
C7—C8—O10	127.0 (2)	C107—C108—O110	127.3 (2)
O9—C8—O10	122.4 (2)	O109—C108—O110	121.8 (2)
C5—O9—C8	111.86 (16)	C105—O109—C108	111.30 (16)
C7—O11—C12	107.78 (15)	C107—O111—C112	107.57 (15)
O11—C12—O13	104.60 (16)	O111—C112—O113	105.24 (17)
O11—C12—C14	109.6 (2)	O111—C112—C114	110.5 (2)
O13—C12—C14	111.07 (18)	O113—C112—C114	111.4 (2)
O11—C12—C15	109.02 (19)	O111—C112—C115	108.2 (2)
O13—C12—C15	108.7 (2)	O113—C112—C115	108.3 (2)
C14—C12—C15	113.4 (2)	C114—C112—C115	112.8 (2)
C6—O13—C12	107.35 (16)	C106—O113—C112	107.15 (16)
C12—C14—H141	113.3	C112—C114—H1141	111.0
C12—C14—H142	108.2	C112—C114—H1142	112.2
H141—C14—H142	106.5	H1141—C114—H1142	104.5
C12—C14—H143	111.4	C112—C114—H1143	112.2
H141—C14—H143	109.8	H1141—C114—H1143	105.3
H142—C14—H143	107.3	H1142—C114—H1143	111.2
C12—C15—H151	110.8	C112—C115—H1151	111.8
C12—C15—H152	109.5	C112—C115—H1152	106.9
H151—C15—H152	109.7	H1151—C115—H1152	107.4
C12—C15—H153	108.8	C112—C115—H1153	110.7
H151—C15—H153	109.0	H1151—C115—H1153	111.1
H152—C15—H153	109.0	H1152—C115—H1153	108.7
C7—C16—O17	104.76 (16)	C107—C116—O117	104.97 (15)
C7—C16—H161	108.3	C107—C116—H1161	111.3
O17—C16—H161	115.5	O117—C116—H1161	112.4
C7—C16—H162	110.0	C107—C116—H1162	109.1
O17—C16—H162	112.3	O117—C116—H1162	113.5
H161—C16—H162	105.9	H1161—C116—H1162	105.7
C16—O17—C18	115.72 (18)	C116—O117—C118	113.76 (15)

O17—C18—C19	107.1 (2)	O117—C118—C119	108.54 (16)
O17—C18—H181	107.8	O117—C118—H1181	111.5
C19—C18—H181	110.1	C119—C118—H1181	111.0
O17—C18—H182	111.4	O117—C118—H1182	109.3
C19—C18—H182	114.2	C119—C118—H1182	112.1
H181—C18—H182	106.1	H1181—C118—H1182	104.4
C18—C19—C20	120.2 (3)	C118—C119—C120	121.2 (2)
C18—C19—C24	121.6 (3)	C118—C119—C124	119.5 (2)
C20—C19—C24	118.2 (3)	C120—C119—C124	119.3 (2)
C19—C20—C21	121.0 (3)	C119—C120—C121	120.7 (2)
C19—C20—H201	118.9	C119—C120—H1201	119.9
C21—C20—H201	120.1	C121—C120—H1201	119.3
C20—C21—C22	119.8 (3)	C120—C121—C122	120.2 (3)
C20—C21—H211	117.8	C120—C121—H1211	119.2
C22—C21—H211	122.5	C122—C121—H1211	120.5
C21—C22—C23	119.6 (3)	C121—C122—C123	119.8 (2)
C21—C22—H221	119.9	C121—C122—H1221	117.9
C23—C22—H221	120.4	C123—C122—H1221	122.3
C22—C23—C24	120.7 (3)	C122—C123—C124	120.4 (3)
C22—C23—H231	120.1	C122—C123—H1231	120.9
C24—C23—H231	119.1	C124—C123—H1231	118.7
C23—C24—C19	120.7 (3)	C123—C124—C119	119.6 (2)
C23—C24—H241	118.6	C123—C124—H1241	120.0
C19—C24—H241	120.7	C119—C124—H1241	120.3
C4—C25—O26	103.96 (18)	C104—C125—O126	102.55 (18)
C4—C25—H251	109.8	C104—C125—H1251	108.3
O26—C25—H251	111.5	O126—C125—H1251	115.9
C4—C25—H252	109.9	C104—C125—H1252	110.4
O26—C25—H252	113.9	O126—C125—H1252	111.6
H251—C25—H252	107.8	H1251—C125—H1252	107.9
C2—O26—C25	107.06 (18)	C125—O126—C102	106.07 (17)
C2—C27—H271	105.8	C102—C127—H1271	107.8
C2—C27—H272	107.6	C102—C127—H1272	109.6
H271—C27—H272	112.5	H1271—C127—H1272	111.7
C2—C27—H273	111.3	C102—C127—H1273	106.1
H271—C27—H273	109.4	H1271—C127—H1273	112.4
H272—C27—H273	110.1	H1272—C127—H1273	109.0