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Isopropyl 2,3,4,6-tetra-*O*-acetyl-β-D-glucopyranoside

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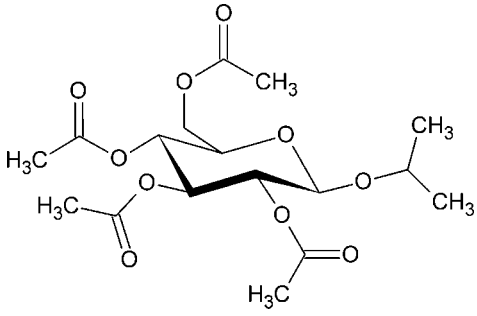
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.008 Å; R factor = 0.058; wR factor = 0.151; data-to-parameter ratio = 9.3.

The title compound, $C_{17}H_{26}O_{10}$, was formed by a Koenigs–Knorr reaction of 2,3,4,6-tetra-*O*-acetyl-α-D-glucopyranosyl bromide and propan-2-ol. The central ring adopts a chair conformation. The crystal does not contain any significant intermolecular interactions.

Related literature

Metabolites of alcohol are important markers for previous alcohol consumption, see: Joya *et al.* (2012); Helander *et al.* (2012). For investigation of the short-chain alkyl alcohol content in alcoholic beverages, see: Lachenmeier & Musshoff (2004). For the relevance of short-chain alkyl alcohol glucuronides as alcohol markers, see; Sticht & Käferstein (1999). For related synthesis, see: Baer & Abbas (1979).



Experimental

Crystal data

$C_{17}H_{26}O_{10}$
M_r = 390.38
Monoclinic, $P2_1$
a = 9.4225 (12) Å
b = 9.9313 (12) Å
c = 11.3641 (15) Å
β = 98.482 (9)°
V = 1051.8 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 296 K
0.28 × 0.12 × 0.11 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.221, T_{max} = 0.364
11225 measured reflections
2274 independent reflections
1411 reflections with $I > 2\sigma(I)$
R_{int} = 0.107

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.151
S = 1.07
2274 reflections
244 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

Data collection: *APEX2* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); 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) and *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT6870).

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supporting information

Acta Cryst. (2013). E**69**, o157 [doi:10.1107/S1600536812051483]

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S1. Comment

In recent years the determination of alcohol metabolites gained importance for screening previous alcohol consumption (Joya *et al.*, Helander *et al.*, 2012). Beside ethanol several short-chain alkyl alcohols, *e.g. i*-propanol, are found in alcoholic beverages as a result of the fermentation process (Lachenmeier & Musshoff, 2004). The glucuronides of these so-called fusel alcohols are interesting markers for the consumption of alcohol (Sticht & Käferstein, 1999). Hence, the analysis of these glucuronic metabolites, including their synthesis and full characterization is mandatory.

The title compound was formed by a Koenigs-Knorr-reaction of 2,3,4,6-tetra-*O*-acetyl-*α*-*D*-glucopyranosyl bromide and propan-2-ol (related synthesis Baer & Abbas, 1979) as an intermediate product towards synthesis of *n*-propyl-glucuronide.

The central ring has a chair conformation (Fig 1). The absolute configuration could not be defined confidently based on the single-crystal diffraction data. The isomeric purity of the title compound was confirmed by ^{1}H-NMR.

S2. Experimental

i-Propyl 2,3,4,6-tetra-*O*-acetyl-*β*-*D*-glucopyranoside was synthesized by a Koenigs-Knorr-reaction of 2,3,4,6-tetra-*O*-acetyl-*α*-*D*-glucopyranosyl bromide and propan-2-ol. In order to obtain crystals suitable for single-crystal analysis, about 10 mg of the compound were dissolved in 2 ml propan-2-ol. Colourless crystals of the title compound were formed after 4 days of slow solvent evaporation at room temperature.

S3. Refinement

All H-atoms were positioned geometrically and refined using a riding model with d(C—H) = 0.93 Å, U_{iso}=1.2U_{eq} (C) for aromatic 0.98 Å, U_{iso} = 1.2U_{eq} (C) for CH, 0.97 Å, U_{iso} = 1.2U_{eq} (C) for CH_2, 0.96 Å, U_{iso} = 1.5U_{eq} (C) for CH_3 hydrogen atoms. In the absence of significant anomalous dispersion effects Friedel pairs were merged. The absolute configuration has not been determined by anomalous-dispersion effects in diffraction measurements of the crystal. The conformation has been assigned due to an unchanging chiral centre in the synthetic procedure.

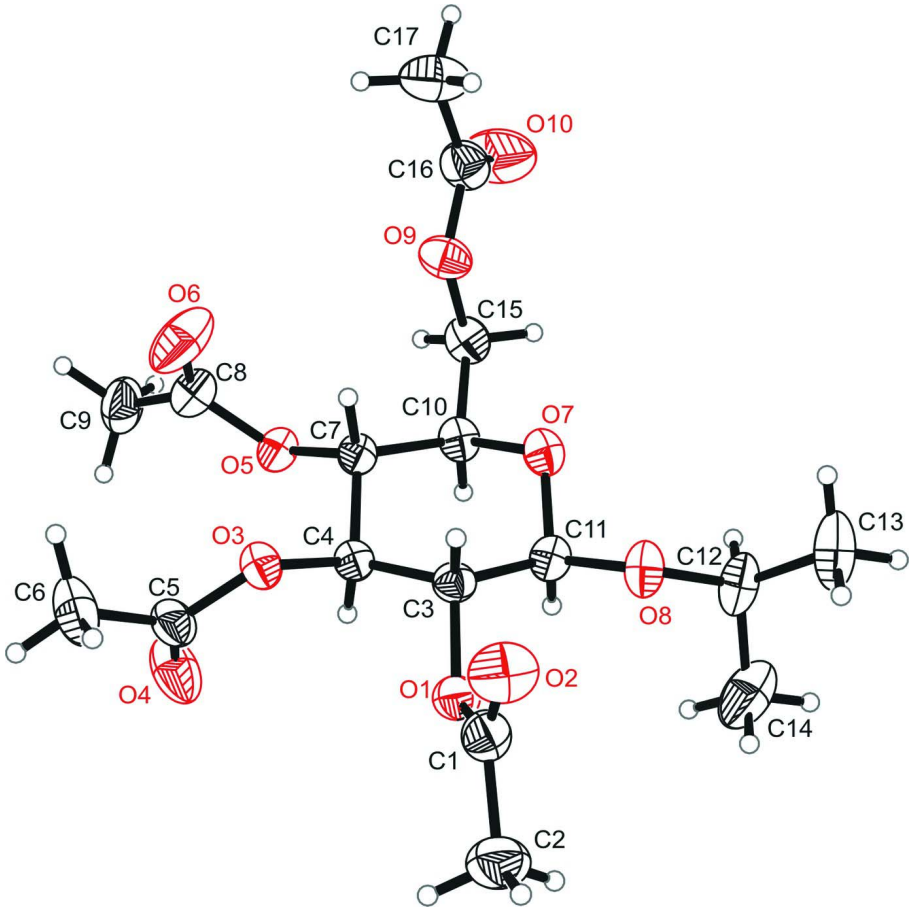

Figure 1

ORTEP representation of the title compound with atomic labeling shown with 30% probability displacement ellipsoids.

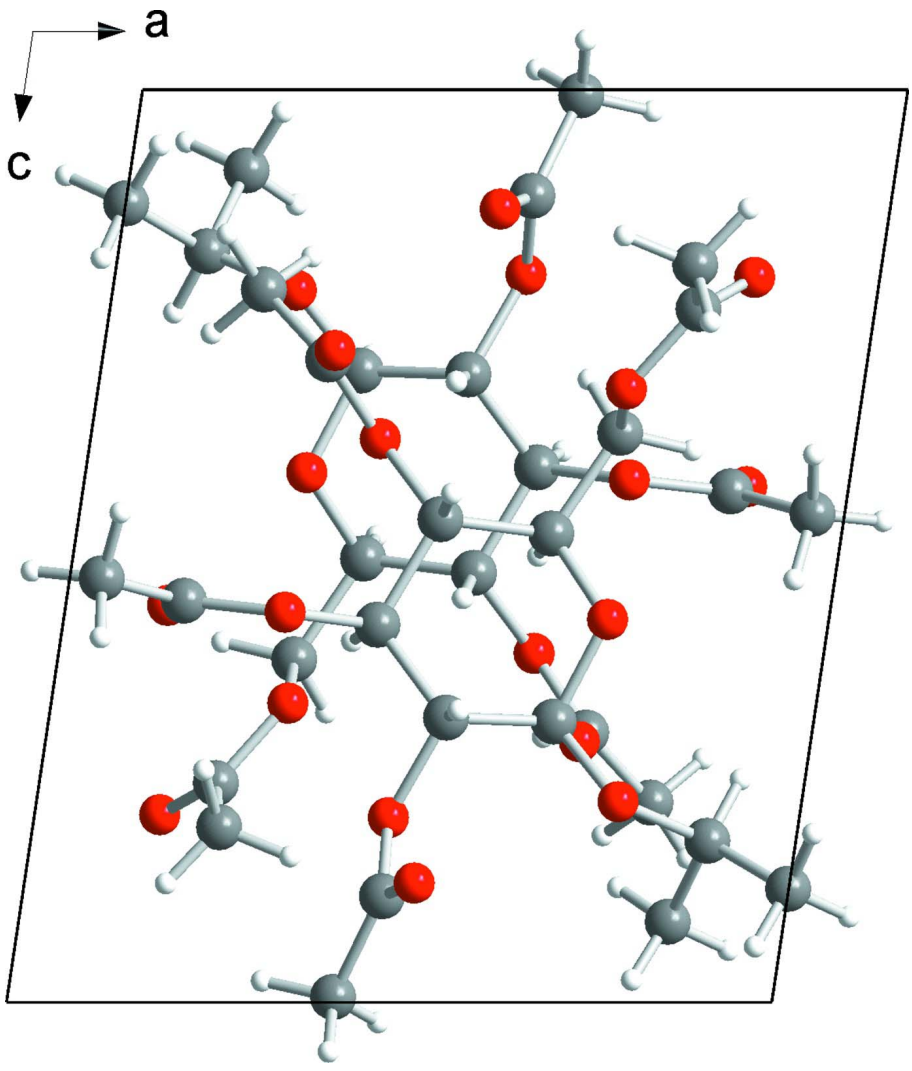

Figure 2

View of the unit cell of the title compound along the *b* axis.

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$c = 11.3641$ (15) Å
$\beta = 98.482$ (9)°
$V = 1051.8$ (2) Å^3
$Z = 2$
$F(000) = 416$
$D_x = 1.233$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1344 reflections
$\theta = 2.6$–19.8°
$\mu = 0.10$ mm^{-1}
$T = 296$ K
Block, colourless
0.28 × 0.12 × 0.11 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.221, T_{max} = 0.364
11225 measured reflections
2274 independent reflections
1411 reflections with $I > 2\sigma(I)$
R_{int} = 0.107
θ_{max} = 26.3°, θ_{min} = 1.8°
h = −11→11
k = −12→11
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.151
S = 1.07
2274 reflections
244 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
$w = 1/[\sigma^2(F_o^2) + (0.0659P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

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}
O1	0.5370 (3)	0.1530 (3)	0.2024 (3)	0.0679 (9)
O2	0.4905 (5)	0.3603 (4)	0.1307 (4)	0.1009 (14)
O3	0.7120 (3)	0.2202 (3)	0.4275 (3)	0.0599 (8)
O4	0.8668 (4)	0.0492 (4)	0.4350 (5)	0.1119 (16)
O5	0.6214 (3)	0.0547 (3)	0.6176 (3)	0.0608 (8)
O6	0.6978 (6)	0.2424 (5)	0.7143 (4)	0.1298 (19)
O7	0.2892 (3)	0.1260 (3)	0.4196 (3)	0.0651 (8)
O8	0.2399 (3)	0.1684 (3)	0.2197 (3)	0.0696 (9)
O9	0.3094 (4)	0.1908 (3)	0.6722 (3)	0.0752 (10)
O10	0.1637 (7)	0.1219 (6)	0.7944 (5)	0.149 (2)
C1	0.5300 (6)	0.2489 (6)	0.1162 (5)	0.0727 (14)
C2	0.5743 (7)	0.1902 (8)	0.0060 (5)	0.108 (2)
H2A	0.5697	0.2584	−0.0542	0.161*
H2B	0.5109	0.1176	−0.0220	0.161*
H2C	0.6707	0.1568	0.0235	0.161*
C3	0.4811 (4)	0.1820 (4)	0.3120 (4)	0.0542 (11)

H3A	0.4669	0.2792	0.3197	0.065*
C4	0.5889 (4)	0.1315 (4)	0.4138 (4)	0.0514 (10)
H4A	0.6193	0.0404	0.3959	0.062*
C5	0.8464 (5)	0.1663 (6)	0.4401 (5)	0.0709 (14)
C6	0.9581 (6)	0.2722 (6)	0.4646 (7)	0.104 (2)
H6A	1.0514	0.2314	0.4728	0.156*
H6B	0.9468	0.3183	0.5368	0.156*
H6C	0.9483	0.3353	0.3999	0.156*
C7	0.5271 (4)	0.1297 (4)	0.5294 (4)	0.0526 (10)
H7A	0.5172	0.2221	0.5572	0.063*
C8	0.7049 (6)	0.1238 (7)	0.7044 (5)	0.0780 (15)
C9	0.8026 (6)	0.0292 (7)	0.7811 (5)	0.095 (2)
H9A	0.8613	0.0794	0.8420	0.143*
H9B	0.8626	−0.0172	0.7331	0.143*
H9C	0.7462	−0.0350	0.8171	0.143*
C10	0.3795 (5)	0.0587 (5)	0.5137 (4)	0.0574 (11)
H10A	0.3926	−0.0345	0.4892	0.069*
C11	0.3396 (5)	0.1088 (5)	0.3077 (4)	0.0580 (11)
H11A	0.3509	0.0130	0.2907	0.070*
C12	0.1157 (6)	0.0848 (6)	0.1770 (5)	0.0838 (17)
H12A	0.0844	0.0373	0.2443	0.101*
C13	0.0007 (7)	0.1795 (8)	0.1244 (7)	0.133 (3)
H13A	−0.0210	0.2409	0.1846	0.199*
H13B	−0.0839	0.1295	0.0938	0.199*
H13C	0.0331	0.2293	0.0610	0.199*
C14	0.1516 (9)	−0.0151 (8)	0.0883 (7)	0.132 (3)
H14A	0.2236	−0.0758	0.1259	0.198*
H14B	0.1874	0.0312	0.0245	0.198*
H14C	0.0671	−0.0649	0.0572	0.198*
C15	0.3091 (6)	0.0569 (5)	0.6241 (5)	0.0708 (14)
H15A	0.2113	0.0246	0.6051	0.085*
H15B	0.3609	−0.0034	0.6825	0.085*
C16	0.2309 (6)	0.2104 (7)	0.7598 (5)	0.0852 (17)
C17	0.2438 (9)	0.3493 (7)	0.8081 (6)	0.119 (3)
H17A	0.1849	0.3584	0.8698	0.178*
H17B	0.2127	0.4124	0.7456	0.178*
H17C	0.3420	0.3671	0.8403	0.178*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.071 (2)	0.073 (2)	0.0601 (19)	0.0155 (19)	0.0089 (16)	0.0013 (19)
O2	0.137 (4)	0.075 (3)	0.093 (3)	0.009 (3)	0.025 (3)	0.022 (2)
O3	0.0504 (17)	0.0541 (18)	0.075 (2)	0.0007 (15)	0.0081 (15)	0.0020 (16)
O4	0.069 (2)	0.069 (3)	0.197 (5)	0.014 (2)	0.016 (3)	−0.005 (3)
O5	0.0677 (18)	0.0512 (17)	0.060 (2)	−0.0004 (16)	−0.0015 (17)	0.0015 (17)
O6	0.190 (5)	0.077 (3)	0.104 (4)	−0.003 (3)	−0.039 (3)	−0.021 (3)
O7	0.0547 (17)	0.0625 (19)	0.077 (2)	−0.0016 (16)	0.0072 (16)	0.0041 (19)

O8	0.0603 (18)	0.061 (2)	0.081 (2)	−0.0050 (16)	−0.0111 (17)	0.0118 (18)
O9	0.084 (2)	0.064 (2)	0.084 (2)	0.0017 (19)	0.035 (2)	0.006 (2)
O10	0.176 (5)	0.149 (4)	0.144 (5)	−0.045 (4)	0.099 (4)	−0.011 (4)
C1	0.071 (3)	0.080 (4)	0.067 (4)	0.001 (3)	0.009 (3)	0.010 (3)
C2	0.117 (5)	0.133 (6)	0.077 (4)	0.017 (5)	0.027 (4)	0.005 (4)
C3	0.059 (2)	0.050 (2)	0.054 (3)	0.006 (2)	0.009 (2)	0.000 (2)
C4	0.053 (2)	0.044 (2)	0.057 (3)	−0.001 (2)	0.008 (2)	0.001 (2)
C5	0.056 (3)	0.074 (4)	0.083 (4)	0.013 (3)	0.010 (3)	0.000 (3)
C6	0.055 (3)	0.084 (4)	0.170 (7)	−0.003 (3)	0.003 (4)	−0.007 (4)
C7	0.056 (2)	0.041 (2)	0.059 (3)	0.006 (2)	0.000 (2)	0.002 (2)
C8	0.086 (4)	0.076 (4)	0.069 (4)	−0.009 (3)	0.000 (3)	−0.004 (3)
C9	0.080 (4)	0.125 (5)	0.074 (4)	−0.006 (4)	−0.013 (3)	0.013 (4)
C10	0.059 (3)	0.042 (2)	0.070 (3)	0.000 (2)	0.007 (2)	0.009 (2)
C11	0.063 (3)	0.046 (2)	0.063 (3)	−0.001 (2)	0.001 (2)	0.003 (2)
C12	0.076 (3)	0.083 (4)	0.082 (4)	−0.020 (3)	−0.022 (3)	0.017 (3)
C13	0.078 (4)	0.155 (7)	0.148 (7)	−0.014 (5)	−0.043 (4)	0.015 (6)
C14	0.167 (8)	0.108 (5)	0.104 (6)	−0.009 (5)	−0.034 (5)	−0.016 (5)
C15	0.070 (3)	0.064 (3)	0.080 (4)	−0.005 (3)	0.018 (3)	0.010 (3)
C16	0.081 (4)	0.099 (5)	0.078 (4)	0.004 (4)	0.022 (3)	0.013 (4)
C17	0.163 (7)	0.098 (5)	0.104 (5)	0.029 (5)	0.049 (5)	−0.005 (4)

Geometric parameters (Å, º)

O1—C1	1.361 (6)	C6—H6B	0.9600
O1—C3	1.451 (5)	C6—H6C	0.9600
O2—C1	1.187 (6)	C7—C10	1.546 (6)
O3—C5	1.363 (6)	C7—H7A	0.9800
O3—C4	1.446 (5)	C8—C9	1.500 (8)
O4—C5	1.181 (6)	C9—H9A	0.9600
O5—C8	1.354 (6)	C9—H9B	0.9600
O5—C7	1.445 (5)	C9—H9C	0.9600
O6—C8	1.187 (7)	C10—C15	1.503 (6)
O7—C10	1.431 (5)	C10—H10A	0.9800
O7—C11	1.432 (5)	C11—H11A	0.9800
O8—C11	1.398 (5)	C12—C14	1.489 (9)
O8—C12	1.459 (6)	C12—C13	1.491 (8)
O9—C16	1.339 (6)	C12—H12A	0.9800
O9—C15	1.438 (6)	C13—H13A	0.9600
O10—C16	1.184 (7)	C13—H13B	0.9600
C1—C2	1.495 (8)	C13—H13C	0.9600
C2—H2A	0.9600	C14—H14A	0.9600
C2—H2B	0.9600	C14—H14B	0.9600
C2—H2C	0.9600	C14—H14C	0.9600
C3—C4	1.508 (6)	C15—H15A	0.9700
C3—C11	1.514 (6)	C15—H15B	0.9700
C3—H3A	0.9800	C16—C17	1.484 (9)
C4—C7	1.513 (6)	C17—H17A	0.9600
C4—H4A	0.9800	C17—H17B	0.9600

C5—C6	1.485 (8)	C17—H17C	0.9600
C6—H6A	0.9600		
C1—O1—C3	119.6 (4)	C8—C9—H9C	109.5
C5—O3—C4	119.4 (4)	H9A—C9—H9C	109.5
C8—O5—C7	118.5 (4)	H9B—C9—H9C	109.5
C10—O7—C11	111.7 (3)	O7—C10—C15	110.1 (4)
C11—O8—C12	114.8 (3)	O7—C10—C7	107.4 (3)
C16—O9—C15	116.5 (4)	C15—C10—C7	114.2 (4)
O2—C1—O1	122.4 (5)	O7—C10—H10A	108.4
O2—C1—C2	127.7 (6)	C15—C10—H10A	108.4
O1—C1—C2	109.9 (5)	C7—C10—H10A	108.4
C1—C2—H2A	109.5	O8—C11—O7	108.0 (4)
C1—C2—H2B	109.5	O8—C11—C3	108.4 (3)
H2A—C2—H2B	109.5	O7—C11—C3	108.6 (3)
C1—C2—H2C	109.5	O8—C11—H11A	110.6
H2A—C2—H2C	109.5	O7—C11—H11A	110.6
H2B—C2—H2C	109.5	C3—C11—H11A	110.6
O1—C3—C4	107.7 (3)	O8—C12—C14	110.7 (5)
O1—C3—C11	107.8 (3)	O8—C12—C13	105.9 (5)
C4—C3—C11	110.9 (3)	C14—C12—C13	111.9 (6)
O1—C3—H3A	110.1	O8—C12—H12A	109.4
C4—C3—H3A	110.1	C14—C12—H12A	109.4
C11—C3—H3A	110.1	C13—C12—H12A	109.4
O3—C4—C3	108.6 (3)	C12—C13—H13A	109.5
O3—C4—C7	108.6 (3)	C12—C13—H13B	109.5
C3—C4—C7	111.6 (3)	H13A—C13—H13B	109.5
O3—C4—H4A	109.3	C12—C13—H13C	109.5
C3—C4—H4A	109.3	H13A—C13—H13C	109.5
C7—C4—H4A	109.3	H13B—C13—H13C	109.5
O4—C5—O3	122.4 (5)	C12—C14—H14A	109.5
O4—C5—C6	126.2 (5)	C12—C14—H14B	109.5
O3—C5—C6	111.3 (5)	H14A—C14—H14B	109.5
C5—C6—H6A	109.5	C12—C14—H14C	109.5
C5—C6—H6B	109.5	H14A—C14—H14C	109.5
H6A—C6—H6B	109.5	H14B—C14—H14C	109.5
C5—C6—H6C	109.5	O9—C15—C10	109.2 (4)
H6A—C6—H6C	109.5	O9—C15—H15A	109.8
H6B—C6—H6C	109.5	C10—C15—H15A	109.8
O5—C7—C4	109.4 (3)	O9—C15—H15B	109.8
O5—C7—C10	107.2 (3)	C10—C15—H15B	109.8
C4—C7—C10	111.1 (3)	H15A—C15—H15B	108.3
O5—C7—H7A	109.7	O10—C16—O9	121.4 (6)
C4—C7—H7A	109.7	O10—C16—C17	125.9 (6)
C10—C7—H7A	109.7	O9—C16—C17	112.7 (6)
O6—C8—O5	122.5 (6)	C16—C17—H17A	109.5
O6—C8—C9	127.2 (6)	C16—C17—H17B	109.5
O5—C8—C9	110.3 (5)	H17A—C17—H17B	109.5

C8—C9—H9A	109.5	C16—C17—H17C	109.5
C8—C9—H9B	109.5	H17A—C17—H17C	109.5
H9A—C9—H9B	109.5	H17B—C17—H17C	109.5