

**Methyl 2,3,4-tri-O-pivaloyl- $\beta$ -D-xylopyranoside**

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**Key indicators**

Single-crystal X-ray study  
 $T = 100\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$   
 $R$  factor = 0.039  
 $wR$  factor = 0.094  
Data-to-parameter ratio = 8.3

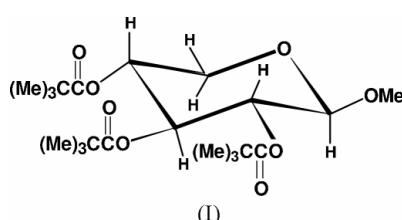
For details of how these key indicators were automatically derived from the article, see  
<http://journals.iucr.org/e>.

The crystal structure of the title compound,  $C_{21}H_{36}O_8$ , has been determined by X-ray analysis at 100 K. The six-membered pyranosyl ring adopts a chair conformation.

Received 9 September 2004  
Accepted 14 September 2004  
Online 25 September 2004

**Comment**

Several series of acylated monosaccharides, the acyl groups being pivaloyls, acetyls or a combination of the two, have been synthesized (Ljevaković *et al.*, 1992; Tomić *et al.*, 1991; Petrović *et al.*, 2002). One of the series of acylated monosaccharides previously prepared was that of methyl  $\beta$ -D-xylopyranoside, of which methyl 2,3,4-tri-O-pivaloyl- $\beta$ -D-xylopyranoside, (I), is the completely acylated analog (Petrović *et al.*, 1997). In carbohydrates, the hydroxyl groups are of comparable reactivity. Therefore, continued interest exists in investigating the characteristics of protective groups such as acyls in order to find suitable conditions to transform these molecules in a selective manner. The pivaloyl group is of special interest since it can be introduced into carbohydrates regioselectively, making further transformations selective as well. It can also be removed selectively by enzymes isolated from mammalian sera (Petrović *et al.*, 1997; Tomić *et al.*, 1991). Therefore, hydroxyl-group protection by pivaloyls leads to interesting new substrates in the investigation of enzymes as catalysts in organic synthesis.



The structure of methyl  $\beta$ -D-xylopyranoside was determined by X-ray analysis (Brown *et al.*, 1996) and by neutron diffraction methods (Takagi & Jeffrey, 1977). Several crystal structures of methyl  $\beta$ -D-xylopyranoside substituted at positions 2, 3 and 4 have been published, *e.g.* methyl 2,3,4-tri-O-benzoyl- $\beta$ -D-xylopyranoside (Vangehr *et al.*, 1980) and methyl 2,3,4-tri-O-acetyl- $\beta$ -methyl-D-xylopyranoside (James & Stevens, 1981). We have previously reported the crystal structure of the 3,4-substituted compound methyl 3,4-di-O-pivaloyl- $\beta$ -D-xylopyranoside (Prugovečki *et al.*, 2004). It is interesting to compare the geometrical parameters of (I) with those in the crystal structures of methyl  $\beta$ -D-xylopyranosides with substituents at positions 2, 3 and 4. The bond lengths within the pyranoside moiety agree with the values reported by Vangehr *et al.* (1980) and James & Stevens (1981). The

anomeric effect can be seen, with the anomeric C1—O1 bond length slightly shorter than the endocyclic C1—O5 and C5—O5 bonds. The six-membered ring in (I) adopts the  $\beta$ - $^4C_1$  conformation with a slightly distorted chair geometry. The puckering parameters are  $q_2 = 0.016(2)$  Å,  $q_3 = 0.615(2)$  Å,  $\varphi_2 = 79(6)^\circ$ ,  $Q = 0.616(2)$  Å and  $\Theta = 1.4(2)^\circ$  (Cremer & Pople, 1975). The same conformation is found in methyl 2,3,4-tri-*O*-acetyl- $\beta$ -methyl- $\alpha$ -xylopyranoside (James & Stevens, 1981); however, in methyl 2,3,4-tri-*O*-benzoyl- $\beta$ - $\alpha$ -xylopyranoside (Vangehr *et al.*, 1980), the pyranosyl ring adopts a twist-boat conformation due to the large benzoyl groups. The molecules are connected only by van der Waals contacts.

## Experimental

The title compound was synthesized according to the method of Petrović *et al.* (1997).

### Crystal data

$C_{21}H_{36}O_8$	$D_x = 1.173 \text{ Mg m}^{-3}$
$M_r = 416.50$	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 1573 reflections
$a = 9.828(1)$ Å	$\theta = 15.0\text{--}25.0^\circ$
$b = 6.849(1)$ Å	$\mu = 0.09 \text{ mm}^{-1}$
$c = 17.634(1)$ Å	$T = 100(2)$ K
$\beta = 96.36(1)^\circ$	Prism, colorless
$V = 1179.7(2)$ Å <sup>3</sup>	0.3 × 0.3 × 0.2 mm
$Z = 2$	

### Data collection

Oxford Diffraction Xcalibur CCD diffractometer	3234 reflections with $I > 2\sigma(I)$
$\omega$ scans	$R_{\text{int}} = 0.088$
Absorption correction: none	$\theta_{\text{max}} = 29.0^\circ$
38147 measured reflections	$h = -13 \rightarrow 13$
3376 independent reflections	$k = -9 \rightarrow 9$
All H-atom parameters refined	$l = -24 \rightarrow 24$

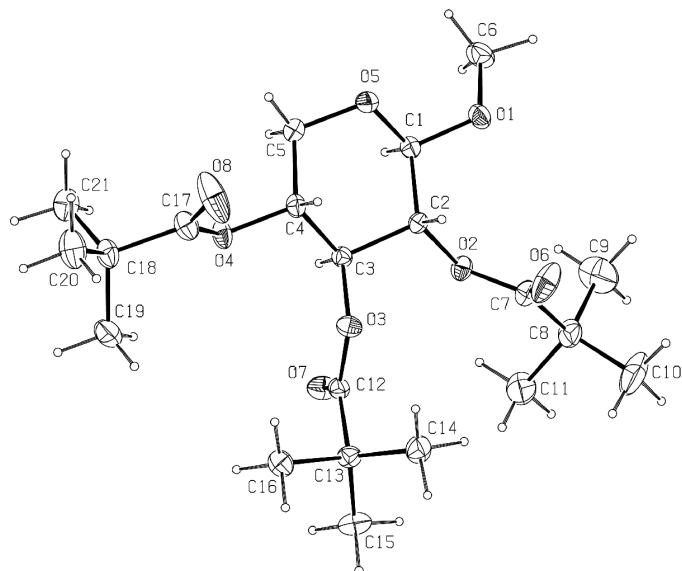
### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0504P)^2 + 0.2141P]$
$R[F^2 > 2\sigma(F^2)] = 0.039$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.094$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.14$	$\Delta\rho_{\text{max}} = 0.37 \text{ e } \text{\AA}^{-3}$
3376 reflections	$\Delta\rho_{\text{min}} = -0.21 \text{ e } \text{\AA}^{-3}$
406 parameters	
All H-atom parameters refined	

**Table 1**  
Selected geometric parameters (Å, °).

O1—C1	1.3881(19)	O5—C5	1.431(2)
O2—C2	1.4352(18)	C1—C2	1.524(2)
O3—C3	1.443(2)	C2—C3	1.527(2)
O4—C4	1.4421(19)	C3—C4	1.515(2)
O5—C1	1.4278(19)	C4—C5	1.527(2)
C1—O5—C5	110.88(12)	O3—C3—C4	109.13(13)
O1—C1—O5	108.55(12)	O3—C3—C2	108.55(14)
O1—C1—C2	108.35(14)	C4—C3—C2	107.77(12)
O5—C1—C2	108.59(12)	O4—C4—C3	108.50(12)
O2—C2—C1	108.51(12)	O4—C4—C5	108.97(15)
O2—C2—C3	108.88(12)	C3—C4—C5	109.00(13)
C1—C2—C3	108.82(13)	O5—C5—C4	109.61(14)

H atoms were found in a difference Fourier map and refined isotropically, giving C—H distances in the range 0.918(2)–1.03(3) Å. The absolute configuration could not be determined from the



**Figure 1**

View of methyl 2,3,4-tri-*O*-pivaloyl- $\beta$ -D-xylopyranoside with the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

diffraction data because of the absence of significant anomalous scatterers; Friedel equivalents were merged in the final refinement and the absolute configuration was assigned in accordance with the known chirality of the methyl  $\beta$ -D-xylopyranoside precursor.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2003); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON98* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

The authors thank the Ministry of Science and Technology of the Republic of Croatia for financial support (grants No. 119632 and 119610).

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# supporting information

*Acta Cryst.* (2004). E60, o1840–o1841 [https://doi.org/10.1107/S1600536804022925]

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### Methyl 2,3,4-tri-O-pivaloyl- $\beta$ -D-xylopyranoside

#### Crystal data

C<sub>21</sub>H<sub>36</sub>O<sub>8</sub>  
 $M_r = 416.50$   
Monoclinic, P2<sub>1</sub>  
Hall symbol: P 2yb  
 $a = 9.828$  (1) Å  
 $b = 6.849$  (1) Å  
 $c = 17.634$  (1) Å  
 $\beta = 96.36$  (1) $^\circ$   
 $V = 1179.7$  (2) Å<sup>3</sup>  
 $Z = 2$

$F(000) = 452$   
 $D_x = 1.173$  Mg m<sup>-3</sup>  
Mo  $K\alpha$  radiation,  $\lambda = 0.71069$  Å  
Cell parameters from 1573 reflections  
 $\theta = 15.0\text{--}25.0^\circ$   
 $\mu = 0.09$  mm<sup>-1</sup>  
 $T = 100$  K  
Prismatic, colorless  
0.3 × 0.3 × 0.2 mm

#### Data collection

Xcalibur CCD  
diffractometer  
Radiation source: fine-focus sealed tube  
Graphite monochromator  
 $\omega$  scans  
38147 measured reflections  
3376 independent reflections

3234 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.088$   
 $\theta_{\text{max}} = 29.0^\circ$ ,  $\theta_{\text{min}} = 4.6^\circ$   
 $h = -13 \rightarrow 13$   
 $k = -9 \rightarrow 9$   
 $l = -24 \rightarrow 24$

#### Refinement

Refinement on  $F^2$   
Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.039$   
 $wR(F^2) = 0.094$   
 $S = 1.14$   
3376 reflections  
406 parameters  
1 restraint  
Primary atom site location: structure-invariant  
direct methods

Secondary atom site location: difference Fourier map  
Hydrogen site location: difference Fourier map  
H atoms treated by a mixture of independent and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.0504P)^2 + 0.2141P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.37$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.21$  e Å<sup>-3</sup>

#### Special details

**Experimental.** The data did not permit the determination of the absolute structure; (the stereochemistry is the relative configuration based on the absolute stereochemistry of related compounds).

**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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.90467 (12)	0.9963 (2)	0.27885 (7)	0.0210 (3)
O2	0.67472 (12)	1.14493 (19)	0.33822 (6)	0.0177 (2)
O3	0.43427 (12)	1.17869 (19)	0.23208 (7)	0.0181 (2)
O4	0.40081 (11)	0.8702 (2)	0.12167 (6)	0.0206 (3)
O5	0.77279 (11)	0.8747 (2)	0.17460 (7)	0.0206 (3)
O6	0.73886 (19)	1.4510 (2)	0.31265 (8)	0.0374 (4)
O7	0.33988 (13)	1.0393 (2)	0.33006 (7)	0.0236 (3)
O8	0.41444 (17)	1.0010 (3)	0.00617 (9)	0.0502 (5)
C1	0.77493 (15)	0.9275 (3)	0.25305 (9)	0.0168 (3)
C2	0.67251 (16)	1.0922 (3)	0.25932 (9)	0.0143 (3)
C3	0.52954 (15)	1.0189 (3)	0.23020 (9)	0.0150 (3)
C4	0.53234 (16)	0.9531 (3)	0.14832 (9)	0.0171 (3)
C5	0.64225 (17)	0.7965 (3)	0.14567 (10)	0.0213 (3)
C6	1.00126 (17)	0.8407 (3)	0.29381 (12)	0.0262 (4)
C7	0.71448 (17)	1.3291 (3)	0.35805 (9)	0.0207 (3)
C8	0.72651 (18)	1.3565 (3)	0.44436 (10)	0.0258 (4)
C9	0.8546 (3)	1.2435 (6)	0.47813 (14)	0.0492 (7)
C10	0.7413 (4)	1.5740 (5)	0.46246 (15)	0.0500 (7)
C11	0.5994 (2)	1.2767 (4)	0.47661 (12)	0.0373 (5)
C12	0.34523 (16)	1.1722 (3)	0.28597 (9)	0.0163 (3)
C13	0.25306 (16)	1.3526 (3)	0.28059 (9)	0.0181 (3)
C14	0.3382 (2)	1.5371 (3)	0.27308 (12)	0.0254 (4)
C15	0.1789 (2)	1.3636 (3)	0.35238 (10)	0.0261 (4)
C16	0.14786 (17)	1.3264 (3)	0.20974 (10)	0.0226 (4)
C17	0.35381 (17)	0.9045 (3)	0.04765 (10)	0.0231 (4)
C18	0.21880 (18)	0.7985 (3)	0.02499 (10)	0.0234 (4)
C19	0.11581 (19)	0.8528 (4)	0.08105 (13)	0.0345 (5)
C20	0.1646 (2)	0.8584 (4)	-0.05658 (12)	0.0358 (5)
C21	0.2473 (2)	0.5787 (3)	0.02853 (12)	0.0297 (4)
H1	0.748 (2)	0.813 (3)	0.2817 (12)	0.012 (5)*
H2	0.696 (2)	1.203 (3)	0.2313 (12)	0.012 (5)*
H3	0.504 (2)	0.910 (3)	0.2612 (11)	0.011 (5)*
H4	0.554 (2)	1.060 (4)	0.1196 (12)	0.015 (5)*
H51	0.618 (2)	0.680 (4)	0.1751 (14)	0.024 (6)*
H52	0.650 (2)	0.759 (4)	0.0963 (13)	0.020 (5)*
H61	1.089 (3)	0.904 (4)	0.3153 (15)	0.033 (7)*
H62	0.970 (3)	0.749 (4)	0.3303 (16)	0.036 (7)*
H63	1.015 (3)	0.765 (4)	0.2466 (15)	0.031 (6)*
H91	0.846 (4)	1.107 (6)	0.466 (2)	0.057 (10)*
H92	0.863 (3)	1.261 (6)	0.533 (2)	0.066 (10)*

H93	0.940 (4)	1.297 (6)	0.459 (2)	0.066 (10)*
H101	0.818 (4)	1.629 (6)	0.446 (2)	0.066 (11)*
H102	0.652 (4)	1.646 (7)	0.439 (2)	0.086 (14)*
H103	0.748 (4)	1.585 (7)	0.522 (2)	0.073 (11)*
H111	0.590 (3)	1.130 (5)	0.4669 (17)	0.044 (8)*
H112	0.610 (3)	1.298 (5)	0.5322 (17)	0.041 (7)*
H113	0.515 (3)	1.348 (6)	0.4560 (17)	0.051 (8)*
H141	0.280 (2)	1.655 (4)	0.2735 (13)	0.022 (5)*
H142	0.413 (3)	1.550 (5)	0.3189 (16)	0.038 (7)*
H143	0.383 (3)	1.538 (5)	0.2262 (16)	0.039 (7)*
H151	0.113 (3)	1.479 (5)	0.3487 (17)	0.040 (7)*
H152	0.125 (3)	1.246 (5)	0.3617 (18)	0.042 (8)*
H153	0.244 (3)	1.385 (5)	0.3980 (16)	0.043 (8)*
H161	0.091 (2)	1.210 (4)	0.2149 (13)	0.017 (5)*
H162	0.191 (2)	1.310 (4)	0.1663 (14)	0.025 (6)*
H163	0.085 (3)	1.445 (4)	0.2023 (15)	0.033 (7)*
H191	0.153 (3)	0.821 (5)	0.1338 (17)	0.039 (7)*
H192	0.029 (3)	0.781 (5)	0.0696 (16)	0.035 (7)*
H193	0.096 (3)	0.989 (6)	0.0821 (19)	0.052 (9)*
H201	0.145 (3)	0.992 (5)	-0.0610 (16)	0.037 (7)*
H202	0.078 (3)	0.792 (5)	-0.0687 (16)	0.040 (7)*
H203	0.229 (3)	0.820 (5)	-0.0927 (18)	0.052 (9)*
H211	0.279 (3)	0.542 (5)	0.0833 (17)	0.044 (8)*
H212	0.161 (3)	0.509 (6)	0.0114 (18)	0.056 (9)*
H213	0.313 (3)	0.542 (5)	-0.0068 (18)	0.050 (8)*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0145 (5)	0.0204 (7)	0.0271 (6)	0.0005 (5)	-0.0021 (4)	-0.0005 (5)
O2	0.0213 (5)	0.0181 (6)	0.0132 (5)	-0.0020 (5)	0.0003 (4)	-0.0006 (4)
O3	0.0171 (5)	0.0195 (6)	0.0179 (5)	0.0031 (5)	0.0032 (4)	0.0034 (5)
O4	0.0189 (5)	0.0271 (7)	0.0149 (5)	-0.0070 (5)	-0.0024 (4)	0.0004 (5)
O5	0.0166 (5)	0.0253 (7)	0.0200 (5)	0.0006 (5)	0.0029 (4)	-0.0057 (5)
O6	0.0682 (11)	0.0214 (7)	0.0231 (7)	-0.0134 (8)	0.0064 (6)	-0.0018 (6)
O7	0.0253 (6)	0.0206 (7)	0.0261 (6)	0.0046 (5)	0.0083 (5)	0.0057 (5)
O8	0.0357 (7)	0.0768 (14)	0.0339 (8)	-0.0261 (9)	-0.0151 (6)	0.0298 (9)
C1	0.0142 (6)	0.0173 (8)	0.0184 (7)	-0.0008 (6)	0.0005 (5)	-0.0011 (6)
C2	0.0159 (6)	0.0152 (7)	0.0117 (6)	-0.0003 (6)	0.0008 (5)	-0.0011 (6)
C3	0.0140 (6)	0.0145 (7)	0.0163 (6)	0.0002 (6)	0.0009 (5)	0.0002 (6)
C4	0.0158 (6)	0.0193 (8)	0.0158 (7)	-0.0032 (6)	-0.0001 (5)	-0.0010 (6)
C5	0.0193 (7)	0.0218 (9)	0.0226 (7)	-0.0011 (7)	0.0020 (6)	-0.0071 (7)
C6	0.0176 (7)	0.0268 (10)	0.0338 (9)	0.0043 (7)	0.0005 (6)	0.0022 (8)
C7	0.0237 (7)	0.0190 (9)	0.0190 (7)	-0.0008 (7)	0.0001 (6)	-0.0027 (6)
C8	0.0274 (8)	0.0333 (10)	0.0167 (7)	-0.0059 (8)	0.0021 (6)	-0.0065 (7)
C9	0.0379 (12)	0.085 (2)	0.0226 (10)	0.0080 (13)	-0.0066 (8)	-0.0003 (12)
C10	0.0763 (19)	0.0427 (15)	0.0329 (11)	-0.0258 (15)	0.0143 (12)	-0.0192 (11)
C11	0.0379 (11)	0.0528 (16)	0.0231 (9)	-0.0126 (10)	0.0115 (8)	-0.0096 (10)

C12	0.0144 (6)	0.0190 (8)	0.0153 (6)	0.0000 (6)	0.0008 (5)	-0.0012 (6)
C13	0.0180 (6)	0.0171 (8)	0.0187 (7)	0.0038 (6)	0.0007 (5)	0.0009 (6)
C14	0.0255 (8)	0.0179 (9)	0.0321 (9)	0.0008 (7)	-0.0001 (7)	-0.0002 (8)
C15	0.0310 (8)	0.0259 (10)	0.0225 (8)	0.0102 (8)	0.0078 (7)	0.0021 (7)
C16	0.0200 (7)	0.0260 (10)	0.0209 (7)	0.0018 (7)	-0.0023 (6)	0.0030 (7)
C17	0.0200 (7)	0.0283 (10)	0.0197 (7)	-0.0012 (7)	-0.0037 (6)	0.0048 (7)
C18	0.0207 (7)	0.0293 (10)	0.0190 (7)	-0.0046 (7)	-0.0028 (6)	0.0000 (7)
C19	0.0188 (7)	0.0487 (15)	0.0356 (10)	-0.0022 (9)	0.0018 (7)	-0.0099 (10)
C20	0.0342 (10)	0.0431 (14)	0.0267 (9)	-0.0065 (10)	-0.0126 (8)	0.0056 (10)
C21	0.0331 (9)	0.0289 (11)	0.0263 (9)	-0.0051 (8)	-0.0006 (7)	-0.0018 (8)

*Geometric parameters ( $\text{\AA}$ ,  $^{\circ}$ )*

O1—C1	1.3881 (19)	C10—H101	0.92 (4)
O1—C6	1.432 (2)	C10—H102	1.05 (5)
O2—C7	1.355 (2)	C10—H103	1.05 (4)
O2—C2	1.4352 (18)	C11—H111	1.02 (3)
O3—C12	1.3620 (19)	C11—H112	0.99 (3)
O3—C3	1.443 (2)	C11—H113	1.00 (3)
O4—C17	1.3560 (19)	C12—C13	1.529 (2)
O4—C4	1.4421 (19)	C13—C14	1.530 (3)
O5—C1	1.4278 (19)	C13—C15	1.531 (2)
O5—C5	1.431 (2)	C13—C16	1.541 (2)
O6—C7	1.199 (2)	C14—H141	0.99 (3)
O7—C12	1.202 (2)	C14—H142	1.03 (3)
O8—C17	1.193 (2)	C14—H143	0.98 (3)
C1—C2	1.524 (2)	C15—H151	1.02 (3)
C1—H1	0.99 (2)	C15—H152	0.99 (3)
C2—C3	1.527 (2)	C15—H153	0.98 (3)
C2—H2	0.95 (2)	C16—H161	0.98 (2)
C3—C4	1.515 (2)	C16—H162	0.92 (2)
C3—H3	0.98 (2)	C16—H163	1.02 (3)
C4—C5	1.527 (2)	C17—C18	1.526 (2)
C4—H4	0.93 (2)	C18—C21	1.532 (3)
C5—H51	1.00 (3)	C18—C20	1.533 (3)
C5—H52	0.92 (2)	C18—C19	1.537 (3)
C6—H61	1.00 (3)	C19—H191	0.99 (3)
C6—H62	0.97 (3)	C19—H192	0.98 (3)
C6—H63	1.00 (3)	C19—H193	0.96 (4)
C7—C8	1.525 (2)	C20—H201	0.93 (3)
C8—C10	1.527 (4)	C20—H202	0.97 (3)
C8—C11	1.530 (3)	C20—H203	0.98 (3)
C8—C9	1.540 (3)	C21—H211	1.01 (3)
C9—H91	0.96 (4)	C21—H212	0.99 (3)
C9—H92	0.97 (4)	C21—H213	0.98 (3)
C9—H93	1.01 (4)		
C1—O1—C6	111.93 (14)	H102—C10—H103	108 (3)

C7—O2—C2	117.23 (13)	C8—C11—H111	110.7 (17)
C12—O3—C3	117.38 (13)	C8—C11—H112	108.6 (17)
C17—O4—C4	116.30 (13)	H111—C11—H112	108 (2)
C1—O5—C5	110.88 (12)	C8—C11—H113	112.0 (19)
O1—C1—O5	108.55 (12)	H111—C11—H113	111 (3)
O1—C1—C2	108.35 (14)	H112—C11—H113	106 (2)
O5—C1—C2	108.59 (12)	O7—C12—O3	123.62 (16)
O1—C1—H1	113.0 (12)	O7—C12—C13	125.74 (14)
O5—C1—H1	108.6 (12)	O3—C12—C13	110.63 (14)
C2—C1—H1	109.6 (12)	C12—C13—C14	110.32 (13)
O2—C2—C1	108.51 (12)	C12—C13—C15	108.77 (14)
O2—C2—C3	108.88 (12)	C14—C13—C15	110.23 (16)
C1—C2—C3	108.82 (13)	C12—C13—C16	107.22 (14)
O2—C2—H2	108.9 (13)	C14—C13—C16	110.49 (15)
C1—C2—H2	110.9 (12)	C15—C13—C16	109.75 (14)
C3—C2—H2	110.9 (12)	C13—C14—H141	110.7 (14)
O3—C3—C4	109.13 (13)	C13—C14—H142	110.7 (17)
O3—C3—C2	108.55 (14)	H141—C14—H142	106 (2)
C4—C3—C2	107.77 (12)	C13—C14—H143	112.3 (18)
O3—C3—H3	111.0 (12)	H141—C14—H143	108 (2)
C4—C3—H3	109.9 (12)	H142—C14—H143	108 (2)
C2—C3—H3	110.5 (12)	C13—C15—H151	110.1 (17)
O4—C4—C3	108.50 (12)	C13—C15—H152	114.1 (18)
O4—C4—C5	108.97 (15)	H151—C15—H152	107 (2)
C3—C4—C5	109.00 (13)	C13—C15—H153	111.1 (16)
O4—C4—H4	112.6 (13)	H151—C15—H153	107 (3)
C3—C4—H4	108.4 (14)	H152—C15—H153	107 (3)
C5—C4—H4	109.2 (13)	C13—C16—H161	110.8 (13)
O5—C5—C4	109.61 (14)	C13—C16—H162	111.0 (15)
O5—C5—H51	112.0 (14)	H161—C16—H162	107 (2)
C4—C5—H51	109.4 (14)	C13—C16—H163	110.8 (16)
O5—C5—H52	105.9 (14)	H161—C16—H163	108.4 (19)
C4—C5—H52	111.2 (15)	H162—C16—H163	108 (2)
H51—C5—H52	109 (2)	O8—C17—O4	123.09 (16)
O1—C6—H61	105.9 (17)	O8—C17—C18	125.57 (16)
O1—C6—H62	110.7 (16)	O4—C17—C18	111.31 (14)
H61—C6—H62	110 (2)	C17—C18—C21	107.88 (16)
O1—C6—H63	112.3 (16)	C17—C18—C20	108.69 (16)
H61—C6—H63	111 (2)	C21—C18—C20	110.12 (18)
H62—C6—H63	107 (2)	C17—C18—C19	109.41 (15)
O6—C7—O2	123.31 (16)	C21—C18—C19	109.99 (19)
O6—C7—C8	125.55 (18)	C20—C18—C19	110.71 (18)
O2—C7—C8	111.13 (15)	C18—C19—H191	110.5 (16)
C7—C8—C10	108.88 (18)	C18—C19—H192	111.4 (16)
C7—C8—C11	110.53 (15)	H191—C19—H192	108 (2)
C10—C8—C11	109.5 (2)	C18—C19—H193	114 (2)
C7—C8—C9	107.08 (17)	H191—C19—H193	105 (3)
C10—C8—C9	110.8 (2)	H192—C19—H193	109 (3)

C11—C8—C9	110.1 (2)	C18—C20—H201	112.7 (18)
C8—C9—H91	110 (2)	C18—C20—H202	106.7 (17)
C8—C9—H92	108 (2)	H201—C20—H202	106 (3)
H91—C9—H92	110 (3)	C18—C20—H203	110.7 (18)
C8—C9—H93	111 (2)	H201—C20—H203	110 (3)
H91—C9—H93	110 (3)	H202—C20—H203	110 (2)
H92—C9—H93	108 (3)	C18—C21—H211	108.7 (18)
C8—C10—H101	113 (3)	C18—C21—H212	108 (2)
C8—C10—H102	109 (3)	H211—C21—H212	110 (3)
H101—C10—H102	111 (4)	C18—C21—H213	111 (2)
C8—C10—H103	106 (2)	H211—C21—H213	113 (3)
H101—C10—H103	109 (3)	H212—C21—H213	107 (3)