

Methyl α -L-rhamnosyl-(1 \rightarrow 2)[α -L-rhamnosyl-(1 \rightarrow 3)]- α -L-rhamnoside pentahydrate: synchrotron study

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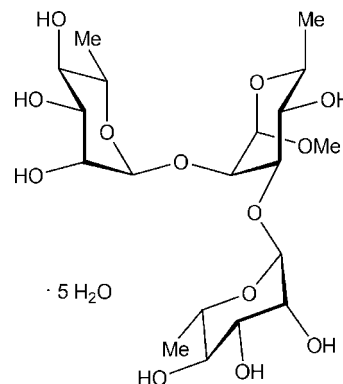
Received 8 June 2012; accepted 16 June 2012

Key indicators: single-crystal synchrotron study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.033; wR factor = 0.087; data-to-parameter ratio = 7.7.

The title hydrate, $\text{C}_{19}\text{H}_{34}\text{O}_{13}\cdot 5\text{H}_2\text{O}$, contains a vicinally disubstituted trisaccharide in which the two terminal rhamnosyl sugar groups are positioned adjacent to each other. The conformation of the trisaccharide is described by the glycosidic torsion angles $\varphi_2 = 48$ (1)°, $\psi_2 = -29$ (1)°, $\varphi_3 = 44$ (1)° and $\psi_3 = 4$ (1)°, whereas the ψ_2 torsion angle represents a conformation from the major state in solution, the ψ_3 torsion angle conformation may have been caught near a potential energy saddle-point when compared to its solution structure, in which at least two but probably three conformational states are populated. Extensive intermolecular O—H \cdots O hydrogen bonding is present in the crystal and a water-containing channel is formed along the b -axis direction.

Related literature

For a description of L-rhamnose as part of polysaccharides, see: Marie *et al.* (1998); Perry & MacLean (2000). For a description of the conformational dynamics of the title trisaccharide, see: Eklund *et al.* (2005); Jonsson *et al.* (2011). For a description of the puckering analysis of the residues, see: Cremer & Pople (1975). For further background to L-rhamnose, see: Ansaruzzaman *et al.* (1996); Varki *et al.* (1999); Kulber-Kielb *et al.* (2007); Lindberg (1998); Säwén *et al.* (2010).



Experimental

Crystal data

$\text{C}_{19}\text{H}_{34}\text{O}_{13}\cdot 5\text{H}_2\text{O}$

$M_r = 560.54$

Monoclinic, C_2

$a = 19.345$ (3) Å

$b = 6.4870$ (13) Å

$c = 21.145$ (3) Å

$\beta = 97.617$ (14)°

$V = 2630.0$ (8) Å³

$Z = 4$

Synchrotron radiation

$\lambda = 0.8970$ Å

$\mu = 0.22$ mm⁻¹

$T = 100$ K

$0.20 \times 0.05 \times 0.01$ mm

Data collection

Bruker SMART 1K CCD

diffractometer

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 2002)

$T_{\min} = 0.97$, $T_{\max} = 0.99$

17172 measured reflections

2906 independent reflections

2655 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.046$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$

$wR(F^2) = 0.087$

$S = 1.07$

2906 reflections

376 parameters

16 restraints

H atoms treated by a mixture of independent and constrained refinement

$\Delta\rho_{\text{max}} = 0.56$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.29$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---------------------------------------|----------|-------------|-------------|---------------|
| OW1—H101 \cdots O33 ⁱ | 0.88 (3) | 1.85 (3) | 2.726 (2) | 176 (2) |
| OW1—H102 \cdots OW3 ⁱⁱ | 0.88 (3) | 1.95 (3) | 2.802 (2) | 162 (2) |
| OW1—H102 \cdots O32 ⁱⁱⁱ | 0.88 (3) | 2.55 (3) | 2.976 (2) | 110 (2) |
| OW2—H201 \cdots O12 ^{iv} | 0.88 (3) | 2.03 (3) | 2.875 (2) | 163 (2) |
| OW2—H202 \cdots O35 ⁱⁱ | 0.87 (3) | 2.08 (3) | 2.877 (2) | 153 (2) |
| OW3—H301 \cdots OW5 | 0.88 (3) | 2.04 (3) | 2.845 (2) | 151 (2) |
| OW3—H302 \cdots O13 ^v | 0.88 (3) | 1.96 (3) | 2.836 (2) | 176 (2) |
| OW4—H401 \cdots OW3 | 0.88 (3) | 1.97 (3) | 2.840 (2) | 168 (2) |
| OW4—H402 \cdots OW1 | 0.88 (3) | 1.92 (3) | 2.771 (2) | 160 (2) |
| OW5—H501 \cdots O33 ^{vi} | 0.87 (3) | 2.07 (3) | 2.918 (2) | 168 (2) |
| OW5—H502 \cdots OW5 ^{vii} | 0.87 (3) | 2.50 (3) | 3.333 (2) | 159 (2) |
| O12—H12A \cdots O32 ⁱⁱⁱ | 0.84 | 2.01 | 2.767 (2) | 149 |
| O13—H13A \cdots O15 ⁱⁱ | 0.84 | 2.10 | 2.858 (2) | 149 |
| O14—H14A \cdots O24 ⁱⁱⁱ | 0.84 | 1.95 | 2.733 (2) | 157 |
| O24—H24A \cdots OW2 | 0.84 | 1.88 | 2.722 (2) | 176 |
| O32—H32A \cdots OW5 ^{viii} | 0.84 | 2.13 | 2.864 (2) | 146 |
| O33—H33A \cdots O34 ⁱ | 0.84 | 1.91 | 2.684 (2) | 152 |
| O34—H34A \cdots OW4 | 0.84 | 1.86 | 2.687 (2) | 168 |

Symmetry codes: (i) $-x + \frac{3}{2}, y - \frac{1}{2}, -z + 1$; (ii) $x, y - 1, z$; (iii) $x + \frac{1}{2}, y - \frac{1}{2}, z$; (iv) $x - \frac{1}{2}, y - \frac{1}{2}, z$; (v) $x, y + 1, z$; (vi) $x + \frac{1}{2}, y + \frac{1}{2}, z$; (vii) $-x + 2, y, -z + 1$; (viii) $x - \frac{1}{2}, y + \frac{1}{2}, z$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINTE* (Bruker, 1997); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *PLATON* (Spek, 2009).

This work was supported by a grant from the Swedish Research Council (VR).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6841).

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supplementary materials

Acta Cryst. (2012). E68, o2221–o2222 [doi:10.1107/S1600536812027390]

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Comment

In carbohydrate structures from humans the number of different monosaccharides is quite limited; typically seven different sugars are present in glycoproteins and glycolipids (Varki *et al.*, 1999). Constituents of polysaccharides in man add a few more monosaccharides to the repertoire. In bacteria, however, more than 100 different monosaccharide components have been found (Lindberg, 1998). One of them, *L*-rhamnose (6-deoxy-*L*-mannose) is present as a major constituent of the O-antigen polysaccharides from *Shigella flexneri* (Kulber-Kielb *et al.*, 2007) and is the sole monosaccharide in the repeating unit of an O-antigen from a *Klebsiella pneumoniae* strain (Ansaruzzaman *et al.*, 1996). *L*-rhamnose is also found at the branch point sugar in some polysaccharides, *e.g.*, from *Escherichia coli* O139 (Marie *et al.*, 1998) and *Yersinia enterocolitica* serotype O:28 (Perry & MacLean, 2000).

In the title compound (I) the three sugar components are all *L*-rhamnose residues having the α -anomeric configuration. The *O*-methyl residue (*a*) is vicinally disubstituted at O2 (residue b) and O3 (residue c) which leads to spatial proximity of also the two latter rhamnosyl groups. The major degrees of freedom in trisaccharide (I) are present at the (1 \rightarrow 2)- and (1 \rightarrow 3)-linkages, *i.e.*, between residues b and a as well as between residues c and a, respectively. The torsion angles are given by $\varphi_2 = 48^\circ$, $\psi_2 = -29^\circ$, $\varphi_3 = 44^\circ$ and $\psi_3 = 4^\circ$. In a recent NMR and molecular dynamics (MD) simulation study of (I) in water solution $\langle\varphi\rangle \approx 40^\circ$, when the *exo*-anomeric conformation was populated, but non-*exo* conformations with $\varphi < 0^\circ$ were also significantly populated (Eklund *et al.*, 2005). The dynamics of the ψ torsion angles were found to be highly correlated with both ψ_2 and ψ_3 being either $> 0^\circ$ or $< 0^\circ$. The conformation of the X-ray structure (Figure 1) is reminiscent of the conformational states found from the MD simulation and the values of the glycosidic torsion angles are observed to correspond to conformational regions that are highly populated, albeit the ψ torsion angles in the solid state structure deviate somewhat from the pattern observed from the molecular simulations with water as a solvent.

In studies of the conformational dynamics of the title trisaccharide *trans*-glycosidic heteronuclear carbon-proton coupling constants were measured (Eklund *et al.*, 2005; Jonsson *et al.*, 2011) which, when interpreted by Karplus-type relationships (Säwén *et al.*, 2010), can yield information on conformation *via* torsion angles at the glycosidic linkages. Calculation of the three-bond coupling constants based on the torsion angles in the crystal structure of the trisaccharide showed that for the φ torsion angles and the ψ torsion angle at the α -(1 \rightarrow 2)-linkage the differences to the experimental data were not larger than *ca* 0.5 Hz, indicating that for these torsions the conformation in the solid state is similar to that populated to a large extent in solution. However, for the ψ torsion angle at the α -(1 \rightarrow 3)-linkage the corresponding difference was larger, *ca* 1 Hz, suggesting that in the crystal structure the latter torsion describes a conformation that is less populated in water solution. The crystal structure conformation is still, however, one in a low potential energy region, since conformational exchange occurs for both of the ψ torsion angles between states for which ψ takes either positive or negative values according to the molecular dynamics simulation (Eklund *et al.*, 2005).

The calculated Cremer & Pople (1975) parameters for the three different rings are: ring O15 → C15 [$Q=0.570$ (2) Å, $\theta=177.9$ (2) ° and $\varphi=20$ (9) °], ring O25 → C25 [$Q=0.580$ (2) Å, $\theta=171.4$ (2) ° and $\varphi=72.5$ (14) °] and for the ring O35 → C35 [$Q=0.582$ (2) Å, $\theta=177.1$ (2) ° and $\varphi=131$ (5) °].

Extensive water-water hydrogen bonding was observed (Table 1) between the title compound and water molecules leading to a water channel in the b-direction (Fig. 2 and Fig. 3). The title compound showed hydrogen bonds to water and to other adjacent (symmetry related) trisaccharides, but no intra-molecular hydrogen bonds were found.

Experimental

The synthesis of (I) was described by Eklund *et al.* (2005) in which all three rhamnosyl residues have the *L* absolute configuration. The trisaccharide was crystallized at ambient temperature by slow evaporation from a mixture of water and ethanol (1:1). The crystal was mounted in a capillary tube and diffraction data were collected at 100 K on beamline I711 at the Swedish synchrotron radiation facility, MAXLAB, Lund.

Refinement

All hydrogen atoms, except those on the water molecules, were geometrically placed and constrained to ride on the parent atom. The C—H bond distances are 0.98 Å for CH₃, 0.99 Å for CH₂, 1.00 Å for CH. The O—H bond distance is 0.84 Å for OH groups. The $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{C}, \text{O})$ for the CH₃ and OH while it was set to $1.2 U_{\text{eq}}(\text{C})$ for all other H atoms. Due to the absence of significant anomalous scatterers, the value of the Flack parameter was not meaningful, thus the 3220 Friedel equivalents were included in the merging process (MERG 4 in *SHELXL*). The absolute configuration of each sugar residue is known from the starting compounds used in the synthesis. The hydrogen atoms of the water molecule were located from difference density map, given $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{O})$ and in the refinement the d(O—H) and d(H..H) were restrained to retain the previously known geometry of the water molecule. The H502 is an hydrogen atom connected to a solvent water molecule where the H502 related by a 2 fold axis will be positioned at a much too close distance. The water molecule defined by OW5, H501 and H502 do not strictly fulfil the crystallographic symmetry of the rest of the structure, at least this is true for one of the H atoms for this very water molecule.

Computing details

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *PLATON* (Spek, 2009).

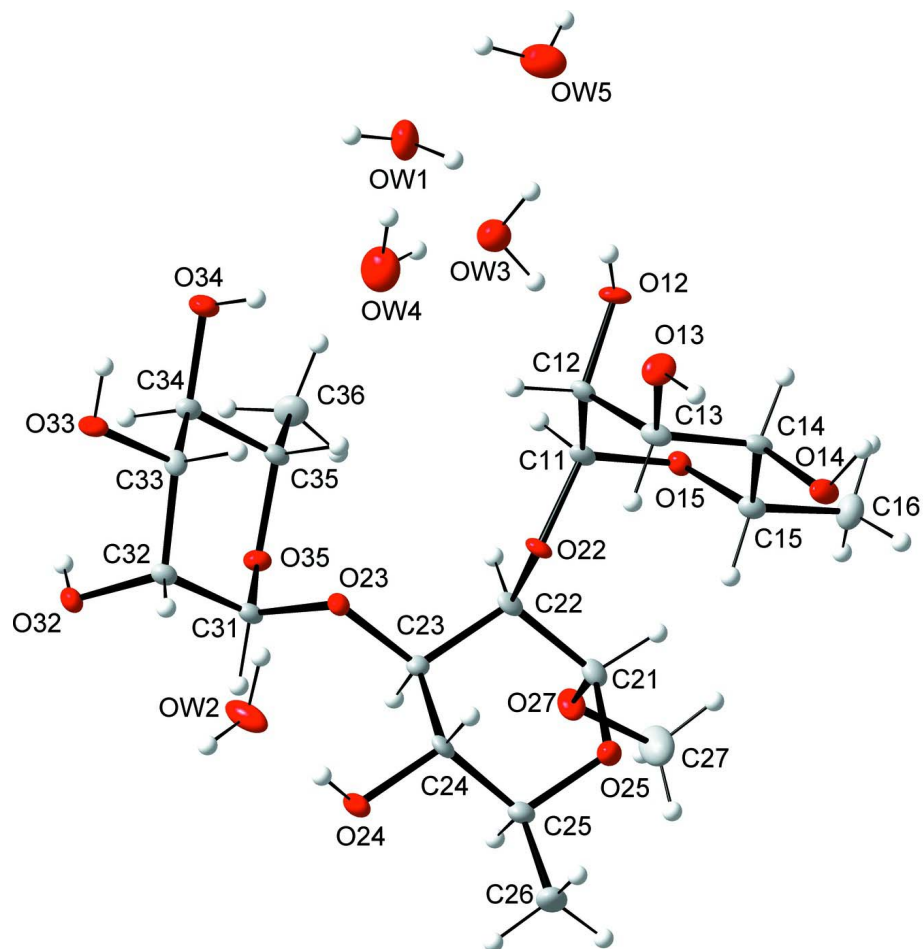
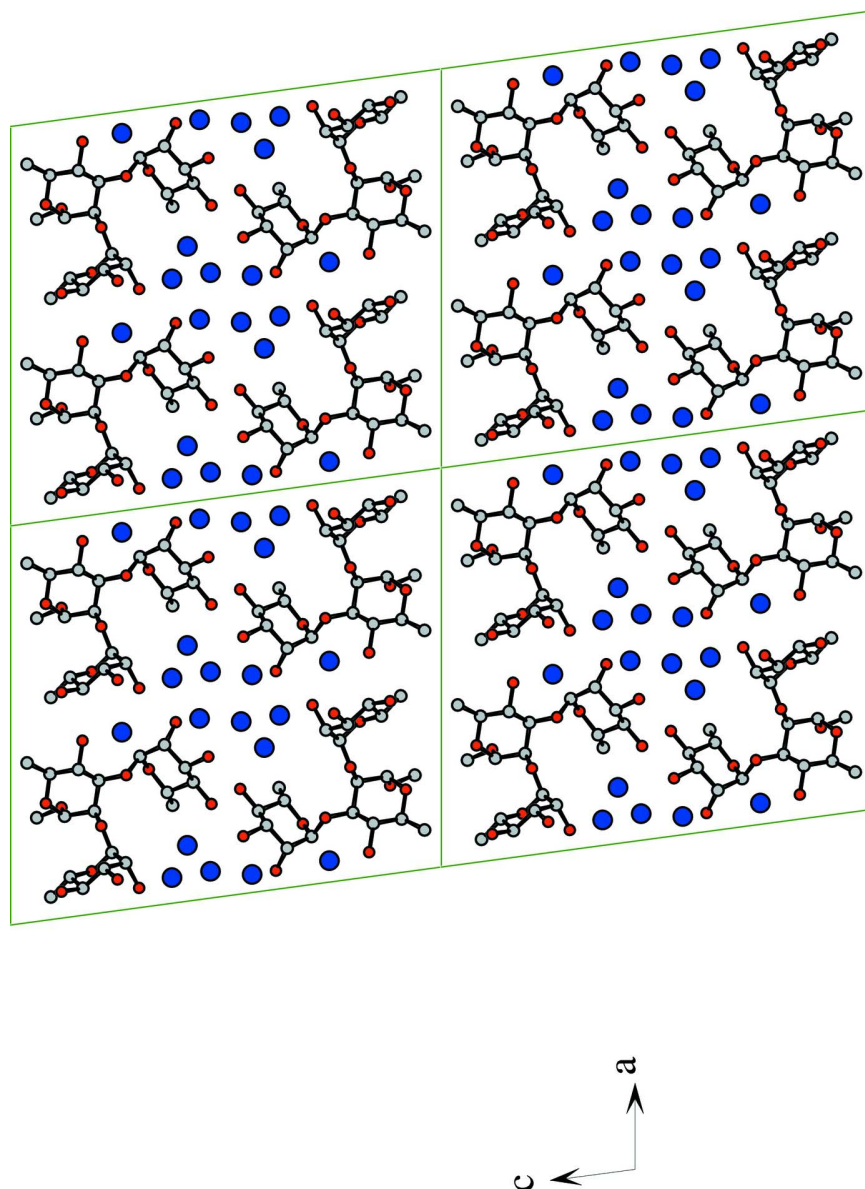
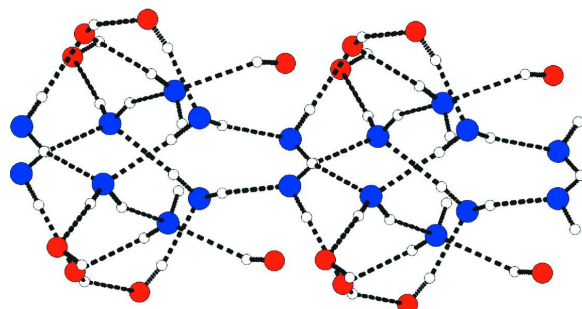
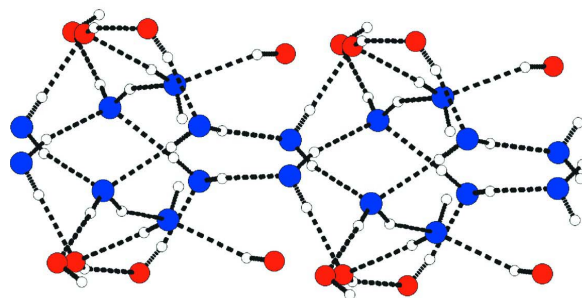


Figure 1

A view of the molecule with displacement ellipsoids drawn at the 50% probability level.

**Figure 2**

Four unit cells viewed along the *b* axis with the water molecules symbolized by the large blue discs. The water molecules mediate intermolecular hydrogen bonds between the sugar molecules and along the *b* axis.

**Figure 3**

Stereoview of the hydrogen bonded water structure of approximately two unit-cell lengths along the *b* axis. The water O atoms are shown with blue color and the hydroxyl O atoms are shown with red color.

Methyl α -L-rhamnosyl-(1 \rightarrow 2)[α -L-rhamnosyl- (1 \rightarrow 3)]- α -L-rhamnoside pentahydrate

Crystal data

$C_{19}H_{34}O_{13} \cdot 5H_2O$

$M_r = 560.54$

Monoclinic, *C* 2y

Hall symbol: *C* 2y

$a = 19.345$ (3) Å

$b = 6.4870$ (13) Å

$c = 21.145$ (3) Å

$\beta = 97.617$ (14)°

$V = 2630.0$ (8) Å³

$Z = 4$

$F(000) = 1208$

$D_x = 1.416$ Mg m⁻³

Synchrotron radiation, $\lambda = 0.8970$ Å

Cell parameters from 963 reflections

$\theta = 2.5$ – 39.8 °

$\mu = 0.22$ mm⁻¹

$T = 100$ K
Plate, colourless

$0.20 \times 0.05 \times 0.01$ mm

Data collection

Bruker SMART 1K CCD
diffractometer
Radiation source: Beamline I711, Maxlab
Silicon monochromator
Detector resolution: 10 pixels mm⁻¹
 ω scan at different φ
Absorption correction: multi-scan
(*SADABS*; Sheldrick, 2002)
 $T_{\min} = 0.97$, $T_{\max} = 0.99$

17172 measured reflections
2906 independent reflections
2655 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.046$
 $\theta_{\text{max}} = 34.1^\circ$, $\theta_{\text{min}} = 2.5^\circ$
 $h = -23 \rightarrow 24$
 $k = -8 \rightarrow 8$
 $l = -23 \rightarrow 26$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.087$
 $S = 1.07$
2906 reflections
376 parameters
16 restraints
Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map
Hydrogen site location: inferred from
neighbouring sites
H atoms treated by a mixture of independent
and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0607P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.56 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.29 \text{ e } \text{\AA}^{-3}$

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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|-------------|--------------|----------------------------------|
| OW1 | 0.93266 (10) | -0.3863 (3) | 0.46456 (9) | 0.0236 (4) |
| H101 | 0.9098 (15) | -0.456 (4) | 0.4905 (12) | 0.035* |
| H102 | 0.9424 (17) | -0.474 (4) | 0.4352 (12) | 0.035* |
| OW2 | 0.55372 (9) | -0.3755 (3) | 0.25876 (10) | 0.0244 (4) |
| H201 | 0.5191 (11) | -0.458 (4) | 0.2626 (15) | 0.037* |
| H202 | 0.5911 (10) | -0.422 (5) | 0.2816 (14) | 0.037* |
| OW3 | 0.93523 (9) | 0.2971 (3) | 0.37476 (9) | 0.0213 (4) |
| H301 | 0.9740 (11) | 0.225 (5) | 0.3809 (13) | 0.032* |
| H302 | 0.9301 (15) | 0.339 (5) | 0.3350 (8) | 0.032* |
| OW4 | 0.85929 (11) | -0.0490 (3) | 0.41056 (10) | 0.0321 (5) |
| H401 | 0.8879 (15) | 0.052 (4) | 0.4032 (17) | 0.048* |
| H402 | 0.8891 (14) | -0.133 (4) | 0.4327 (16) | 0.048* |
| OW5 | 1.04589 (10) | 0.0556 (3) | 0.43863 (9) | 0.0271 (4) |

| | | | | |
|------|--------------|-------------|--------------|------------|
| H501 | 1.0791 (14) | 0.144 (5) | 0.4484 (14) | 0.041* |
| H502 | 1.0265 (15) | 0.022 (5) | 0.4722 (11) | 0.041* |
| C11 | 0.85217 (11) | -0.0103 (3) | 0.23565 (11) | 0.0109 (5) |
| H11 | 0.8527 | 0.0985 | 0.2692 | 0.013* |
| C12 | 0.88270 (11) | -0.2061 (3) | 0.26740 (11) | 0.0116 (5) |
| H12 | 0.8528 | -0.2573 | 0.2992 | 0.014* |
| C13 | 0.88938 (12) | -0.3693 (3) | 0.21708 (11) | 0.0126 (5) |
| H13 | 0.8419 | -0.4074 | 0.1957 | 0.015* |
| C14 | 0.93200 (12) | -0.2870 (3) | 0.16795 (11) | 0.0116 (5) |
| H14 | 0.9806 | -0.2587 | 0.1886 | 0.014* |
| C15 | 0.89936 (12) | -0.0874 (4) | 0.13897 (11) | 0.0121 (5) |
| H15 | 0.8521 | -0.1181 | 0.1157 | 0.014* |
| O15 | 0.89286 (8) | 0.0599 (2) | 0.18944 (7) | 0.0107 (3) |
| O12 | 0.94950 (8) | -0.1441 (2) | 0.29873 (8) | 0.0133 (4) |
| H12A | 0.9670 | -0.2405 | 0.3221 | 0.020* |
| O13 | 0.92191 (9) | -0.5475 (3) | 0.24839 (8) | 0.0174 (4) |
| H13A | 0.9233 | -0.6427 | 0.2217 | 0.026* |
| O14 | 0.93366 (8) | -0.4346 (3) | 0.11879 (8) | 0.0155 (4) |
| H14A | 0.9728 | -0.4926 | 0.1230 | 0.023* |
| C16 | 0.94325 (14) | 0.0139 (4) | 0.09399 (12) | 0.0192 (5) |
| H16A | 0.9205 | 0.1412 | 0.0771 | 0.029* |
| H16B | 0.9485 | -0.0801 | 0.0586 | 0.029* |
| H16C | 0.9893 | 0.0465 | 0.1169 | 0.029* |
| C21 | 0.74537 (12) | 0.1967 (4) | 0.12541 (11) | 0.0135 (5) |
| H21 | 0.7951 | 0.1923 | 0.1173 | 0.016* |
| C22 | 0.74289 (11) | 0.1369 (3) | 0.19460 (11) | 0.0109 (5) |
| H22 | 0.7628 | 0.2513 | 0.2231 | 0.013* |
| O22 | 0.78200 (8) | -0.0481 (2) | 0.21047 (7) | 0.0106 (3) |
| C23 | 0.66745 (11) | 0.0994 (3) | 0.20516 (11) | 0.0105 (5) |
| H23 | 0.6417 | 0.2329 | 0.1986 | 0.013* |
| C24 | 0.63267 (11) | -0.0554 (4) | 0.15733 (11) | 0.0120 (5) |
| H24 | 0.6584 | -0.1893 | 0.1615 | 0.014* |
| C25 | 0.63456 (12) | 0.0347 (4) | 0.09175 (11) | 0.0147 (5) |
| H25 | 0.6115 | 0.1730 | 0.0896 | 0.018* |
| O23 | 0.66298 (8) | 0.0302 (2) | 0.26878 (7) | 0.0111 (3) |
| O24 | 0.56178 (8) | -0.0859 (3) | 0.16682 (8) | 0.0151 (4) |
| H24A | 0.5600 | -0.1706 | 0.1966 | 0.023* |
| O25 | 0.70645 (8) | 0.0599 (3) | 0.08206 (8) | 0.0150 (4) |
| C26 | 0.59920 (13) | -0.0981 (5) | 0.03875 (12) | 0.0228 (6) |
| H26A | 0.6034 | -0.0334 | -0.0024 | 0.034* |
| H26B | 0.5498 | -0.1132 | 0.0436 | 0.034* |
| H26C | 0.6213 | -0.2342 | 0.0406 | 0.034* |
| O27 | 0.72203 (9) | 0.4015 (2) | 0.11823 (8) | 0.0160 (4) |
| C27 | 0.74020 (15) | 0.4932 (5) | 0.06129 (14) | 0.0277 (6) |
| H27A | 0.7895 | 0.4660 | 0.0581 | 0.042* |
| H27B | 0.7324 | 0.6423 | 0.0625 | 0.042* |
| H27C | 0.7112 | 0.4341 | 0.0242 | 0.042* |
| C31 | 0.62082 (12) | 0.1575 (4) | 0.30105 (11) | 0.0110 (5) |
| H31 | 0.5776 | 0.1923 | 0.2715 | 0.013* |

| | | | | |
|------|--------------|-------------|--------------|------------|
| C32 | 0.60030 (11) | 0.0409 (4) | 0.35762 (10) | 0.0110 (5) |
| H32 | 0.5808 | -0.0956 | 0.3422 | 0.013* |
| C33 | 0.66440 (12) | 0.0023 (3) | 0.40588 (11) | 0.0106 (5) |
| H33 | 0.6966 | -0.0894 | 0.3854 | 0.013* |
| C34 | 0.70281 (12) | 0.2040 (3) | 0.42416 (11) | 0.0108 (5) |
| H34 | 0.6719 | 0.2960 | 0.4460 | 0.013* |
| C35 | 0.72015 (12) | 0.3104 (3) | 0.36380 (11) | 0.0111 (5) |
| H35 | 0.7515 | 0.2194 | 0.3422 | 0.013* |
| O35 | 0.65577 (8) | 0.3437 (2) | 0.32144 (7) | 0.0117 (3) |
| O32 | 0.54699 (8) | 0.1518 (3) | 0.38395 (8) | 0.0126 (3) |
| H32A | 0.5645 | 0.2558 | 0.4037 | 0.019* |
| O33 | 0.64458 (8) | -0.1032 (2) | 0.45946 (8) | 0.0133 (4) |
| H33A | 0.6805 | -0.1388 | 0.4838 | 0.020* |
| O34 | 0.76396 (8) | 0.1594 (3) | 0.46677 (8) | 0.0157 (4) |
| H34A | 0.7898 | 0.0805 | 0.4488 | 0.024* |
| C36 | 0.75423 (13) | 0.5176 (4) | 0.37637 (12) | 0.0187 (5) |
| H36A | 0.7546 | 0.5910 | 0.3359 | 0.028* |
| H36B | 0.8023 | 0.4985 | 0.3969 | 0.028* |
| H36C | 0.7281 | 0.5980 | 0.4044 | 0.028* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| OW1 | 0.0248 (10) | 0.0236 (10) | 0.0246 (10) | 0.0013 (8) | 0.0114 (8) | -0.0030 (8) |
| OW2 | 0.0130 (9) | 0.0193 (9) | 0.0394 (12) | -0.0022 (8) | -0.0019 (8) | 0.0144 (8) |
| OW3 | 0.0211 (10) | 0.0208 (9) | 0.0218 (10) | -0.0004 (8) | 0.0019 (8) | -0.0010 (8) |
| OW4 | 0.0354 (12) | 0.0274 (11) | 0.0349 (12) | -0.0050 (10) | 0.0102 (10) | 0.0050 (9) |
| OW5 | 0.0270 (11) | 0.0176 (9) | 0.0343 (11) | -0.0025 (8) | -0.0046 (9) | 0.0022 (9) |
| C11 | 0.0078 (11) | 0.0104 (10) | 0.0138 (11) | -0.0012 (9) | -0.0008 (9) | -0.0032 (9) |
| C12 | 0.0066 (11) | 0.0135 (11) | 0.0143 (12) | -0.0022 (9) | 0.0000 (9) | -0.0007 (9) |
| C13 | 0.0112 (11) | 0.0085 (10) | 0.0171 (12) | -0.0003 (9) | -0.0018 (9) | 0.0011 (9) |
| C14 | 0.0086 (11) | 0.0113 (10) | 0.0139 (11) | 0.0015 (9) | -0.0025 (9) | -0.0015 (9) |
| C15 | 0.0108 (11) | 0.0108 (10) | 0.0140 (12) | -0.0002 (9) | -0.0006 (9) | -0.0005 (9) |
| O15 | 0.0098 (8) | 0.0089 (7) | 0.0133 (8) | 0.0001 (6) | 0.0020 (6) | -0.0003 (7) |
| O12 | 0.0097 (8) | 0.0133 (8) | 0.0149 (9) | 0.0010 (7) | -0.0055 (6) | 0.0006 (7) |
| O13 | 0.0251 (9) | 0.0082 (8) | 0.0185 (9) | 0.0040 (7) | 0.0017 (7) | 0.0012 (7) |
| O14 | 0.0131 (8) | 0.0163 (8) | 0.0164 (9) | 0.0063 (7) | -0.0006 (7) | -0.0047 (7) |
| C16 | 0.0241 (14) | 0.0176 (12) | 0.0172 (13) | 0.0002 (10) | 0.0075 (10) | 0.0026 (10) |
| C21 | 0.0104 (12) | 0.0126 (11) | 0.0179 (12) | 0.0031 (9) | 0.0032 (9) | 0.0022 (10) |
| C22 | 0.0080 (11) | 0.0083 (10) | 0.0161 (12) | 0.0034 (9) | 0.0009 (9) | -0.0015 (9) |
| O22 | 0.0055 (8) | 0.0099 (7) | 0.0156 (8) | 0.0017 (6) | -0.0012 (6) | 0.0010 (6) |
| C23 | 0.0092 (11) | 0.0120 (11) | 0.0100 (11) | 0.0023 (9) | 0.0003 (8) | 0.0011 (9) |
| C24 | 0.0054 (10) | 0.0154 (11) | 0.0154 (11) | 0.0028 (9) | 0.0014 (9) | -0.0010 (10) |
| C25 | 0.0100 (11) | 0.0196 (12) | 0.0139 (12) | 0.0027 (10) | -0.0010 (9) | 0.0000 (10) |
| O23 | 0.0088 (8) | 0.0131 (8) | 0.0113 (8) | 0.0038 (6) | 0.0016 (6) | 0.0013 (7) |
| O24 | 0.0084 (8) | 0.0199 (9) | 0.0167 (9) | -0.0017 (7) | 0.0007 (6) | 0.0043 (7) |
| O25 | 0.0124 (8) | 0.0204 (9) | 0.0123 (8) | 0.0013 (7) | 0.0018 (6) | -0.0015 (7) |
| C26 | 0.0146 (13) | 0.0365 (15) | 0.0163 (13) | -0.0014 (11) | -0.0014 (10) | -0.0060 (12) |
| O27 | 0.0161 (9) | 0.0126 (8) | 0.0199 (9) | 0.0052 (7) | 0.0050 (7) | 0.0070 (7) |
| C27 | 0.0283 (15) | 0.0270 (14) | 0.0291 (15) | 0.0056 (12) | 0.0082 (12) | 0.0162 (12) |

| | | | | | | |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C31 | 0.0073 (10) | 0.0126 (10) | 0.0128 (11) | 0.0009 (9) | -0.0004 (8) | -0.0017 (9) |
| C32 | 0.0092 (11) | 0.0102 (10) | 0.0131 (11) | -0.0006 (9) | -0.0004 (9) | -0.0008 (9) |
| C33 | 0.0118 (11) | 0.0079 (10) | 0.0124 (11) | 0.0010 (9) | 0.0030 (9) | 0.0017 (9) |
| C34 | 0.0101 (11) | 0.0097 (10) | 0.0123 (11) | 0.0004 (9) | -0.0003 (9) | -0.0017 (9) |
| C35 | 0.0074 (11) | 0.0118 (11) | 0.0137 (12) | 0.0013 (9) | -0.0005 (9) | -0.0012 (9) |
| O35 | 0.0111 (8) | 0.0106 (7) | 0.0125 (8) | 0.0002 (6) | -0.0015 (6) | 0.0001 (6) |
| O32 | 0.0087 (8) | 0.0153 (8) | 0.0142 (8) | 0.0009 (7) | 0.0029 (6) | 0.0009 (7) |
| O33 | 0.0105 (8) | 0.0141 (8) | 0.0142 (8) | 0.0013 (7) | -0.0024 (6) | 0.0036 (7) |
| O34 | 0.0103 (8) | 0.0193 (9) | 0.0165 (9) | 0.0017 (7) | -0.0022 (7) | 0.0008 (7) |
| C36 | 0.0204 (13) | 0.0152 (12) | 0.0201 (13) | -0.0065 (10) | 0.0006 (10) | -0.0002 (10) |

Geometric parameters (Å, °)

| | | | |
|----------|------------|----------|-----------|
| OW1—H101 | 0.875 (15) | C23—O23 | 1.432 (3) |
| OW1—H102 | 0.879 (15) | C23—C24 | 1.517 (3) |
| OW2—H201 | 0.871 (15) | C23—H23 | 1.0000 |
| OW2—H202 | 0.869 (15) | C24—O24 | 1.426 (3) |
| OW3—H301 | 0.878 (14) | C24—C25 | 1.510 (3) |
| OW3—H302 | 0.877 (14) | C24—H24 | 1.0000 |
| OW4—H401 | 0.883 (15) | C25—O25 | 1.442 (3) |
| OW4—H402 | 0.883 (15) | C25—C26 | 1.504 (3) |
| OW5—H501 | 0.867 (15) | C25—H25 | 1.0000 |
| OW5—H502 | 0.873 (14) | O23—C31 | 1.400 (3) |
| C11—O15 | 1.409 (3) | O24—H24A | 0.8400 |
| C11—O22 | 1.412 (3) | C26—H26A | 0.9800 |
| C11—C12 | 1.519 (3) | C26—H26B | 0.9800 |
| C11—H11 | 1.0000 | C26—H26C | 0.9800 |
| C12—O12 | 1.429 (3) | O27—C27 | 1.428 (3) |
| C12—C13 | 1.519 (3) | C27—H27A | 0.9800 |
| C12—H12 | 1.0000 | C27—H27B | 0.9800 |
| C13—O13 | 1.435 (3) | C27—H27C | 0.9800 |
| C13—C14 | 1.507 (3) | C31—O35 | 1.423 (3) |
| C13—H13 | 1.0000 | C31—C32 | 1.512 (3) |
| C14—O14 | 1.417 (3) | C31—H31 | 1.0000 |
| C14—C15 | 1.532 (3) | C32—O32 | 1.429 (3) |
| C14—H14 | 1.0000 | C32—C33 | 1.519 (3) |
| C15—O15 | 1.451 (3) | C32—H32 | 1.0000 |
| C15—C16 | 1.507 (3) | C33—O33 | 1.419 (3) |
| C15—H15 | 1.0000 | C33—C34 | 1.529 (3) |
| O12—H12A | 0.8400 | C33—H33 | 1.0000 |
| O13—H13A | 0.8400 | C34—O34 | 1.419 (3) |
| O14—H14A | 0.8400 | C34—C35 | 1.527 (3) |
| C16—H16A | 0.9800 | C34—H34 | 1.0000 |
| C16—H16B | 0.9800 | C35—O35 | 1.451 (3) |
| C16—H16C | 0.9800 | C35—C36 | 1.505 (3) |
| C21—O27 | 1.405 (3) | C35—H35 | 1.0000 |
| C21—O25 | 1.419 (3) | O32—H32A | 0.8400 |
| C21—C22 | 1.521 (3) | O33—H33A | 0.8400 |
| C21—H21 | 1.0000 | O34—H34A | 0.8400 |
| C22—O22 | 1.434 (3) | C36—H36A | 0.9800 |

| | | | |
|---------------|-------------|---------------|-------------|
| C22—C23 | 1.525 (3) | C36—H36B | 0.9800 |
| C22—H22 | 1.0000 | C36—H36C | 0.9800 |
| H101—OW1—H102 | 106 (2) | C25—C24—C23 | 107.07 (19) |
| H201—OW2—H202 | 109 (2) | O24—C24—H24 | 110.2 |
| H301—OW3—H302 | 107 (2) | C25—C24—H24 | 110.2 |
| H401—OW4—H402 | 100 (2) | C23—C24—H24 | 110.2 |
| H501—OW5—H502 | 111 (2) | O25—C25—C26 | 108.12 (19) |
| O15—C11—O22 | 113.08 (18) | O25—C25—C24 | 108.40 (17) |
| O15—C11—C12 | 110.89 (18) | C26—C25—C24 | 113.5 (2) |
| O22—C11—C12 | 108.63 (17) | O25—C25—H25 | 108.9 |
| O15—C11—H11 | 108.0 | C26—C25—H25 | 108.9 |
| O22—C11—H11 | 108.0 | C24—C25—H25 | 108.9 |
| C12—C11—H11 | 108.0 | C31—O23—C23 | 112.64 (17) |
| O12—C12—C13 | 111.41 (18) | C24—O24—H24A | 109.5 |
| O12—C12—C11 | 104.15 (17) | C21—O25—C25 | 114.75 (17) |
| C13—C12—C11 | 109.73 (18) | C25—C26—H26A | 109.5 |
| O12—C12—H12 | 110.5 | C25—C26—H26B | 109.5 |
| C13—C12—H12 | 110.5 | H26A—C26—H26B | 109.5 |
| C11—C12—H12 | 110.5 | C25—C26—H26C | 109.5 |
| O13—C13—C14 | 110.90 (19) | H26A—C26—H26C | 109.5 |
| O13—C13—C12 | 108.14 (18) | H26B—C26—H26C | 109.5 |
| C14—C13—C12 | 109.94 (19) | C21—O27—C27 | 111.92 (19) |
| O13—C13—H13 | 109.3 | O27—C27—H27A | 109.5 |
| C14—C13—H13 | 109.3 | O27—C27—H27B | 109.5 |
| C12—C13—H13 | 109.3 | H27A—C27—H27B | 109.5 |
| O14—C14—C13 | 109.53 (18) | O27—C27—H27C | 109.5 |
| O14—C14—C15 | 109.00 (18) | H27A—C27—H27C | 109.5 |
| C13—C14—C15 | 109.94 (19) | H27B—C27—H27C | 109.5 |
| O14—C14—H14 | 109.5 | O23—C31—O35 | 111.35 (18) |
| C13—C14—H14 | 109.5 | O23—C31—C32 | 108.77 (18) |
| C15—C14—H14 | 109.5 | O35—C31—C32 | 110.42 (18) |
| O15—C15—C16 | 106.72 (19) | O23—C31—H31 | 108.7 |
| O15—C15—C14 | 109.51 (17) | O35—C31—H31 | 108.7 |
| C16—C15—C14 | 112.6 (2) | C32—C31—H31 | 108.7 |
| O15—C15—H15 | 109.3 | O32—C32—C31 | 109.59 (18) |
| C16—C15—H15 | 109.3 | O32—C32—C33 | 112.85 (18) |
| C14—C15—H15 | 109.3 | C31—C32—C33 | 109.72 (18) |
| C11—O15—C15 | 114.07 (17) | O32—C32—H32 | 108.2 |
| C12—O12—H12A | 109.5 | C31—C32—H32 | 108.2 |
| C13—O13—H13A | 109.5 | C33—C32—H32 | 108.2 |
| C14—O14—H14A | 109.5 | O33—C33—C32 | 109.45 (18) |
| C15—C16—H16A | 109.5 | O33—C33—C34 | 112.62 (18) |
| C15—C16—H16B | 109.5 | C32—C33—C34 | 110.77 (18) |
| H16A—C16—H16B | 109.5 | O33—C33—H33 | 107.9 |
| C15—C16—H16C | 109.5 | C32—C33—H33 | 107.9 |
| H16A—C16—H16C | 109.5 | C34—C33—H33 | 107.9 |
| H16B—C16—H16C | 109.5 | O34—C34—C35 | 111.48 (18) |
| O27—C21—O25 | 112.73 (18) | O34—C34—C33 | 108.79 (18) |

| | | | |
|-----------------|--------------|-----------------|--------------|
| O27—C21—C22 | 107.14 (19) | C35—C34—C33 | 109.19 (17) |
| O25—C21—C22 | 112.34 (19) | O34—C34—H34 | 109.1 |
| O27—C21—H21 | 108.2 | C35—C34—H34 | 109.1 |
| O25—C21—H21 | 108.2 | C33—C34—H34 | 109.1 |
| C22—C21—H21 | 108.2 | O35—C35—C36 | 107.24 (18) |
| O22—C22—C21 | 110.84 (18) | O35—C35—C34 | 108.53 (18) |
| O22—C22—C23 | 108.53 (17) | C36—C35—C34 | 113.29 (19) |
| C21—C22—C23 | 109.54 (18) | O35—C35—H35 | 109.2 |
| O22—C22—H22 | 109.3 | C36—C35—H35 | 109.2 |
| C21—C22—H22 | 109.3 | C34—C35—H35 | 109.2 |
| C23—C22—H22 | 109.3 | C31—O35—C35 | 113.29 (16) |
| C11—O22—C22 | 113.20 (16) | C32—O32—H32A | 109.5 |
| O23—C23—C24 | 110.01 (18) | C33—O33—H33A | 109.5 |
| O23—C23—C22 | 111.45 (17) | C34—O34—H34A | 109.5 |
| C24—C23—C22 | 110.88 (18) | C35—C36—H36A | 109.5 |
| O23—C23—H23 | 108.1 | C35—C36—H36B | 109.5 |
| C24—C23—H23 | 108.1 | H36A—C36—H36B | 109.5 |
| C22—C23—H23 | 108.1 | C35—C36—H36C | 109.5 |
| O24—C24—C25 | 108.90 (17) | H36A—C36—H36C | 109.5 |
| O24—C24—C23 | 110.37 (18) | H36B—C36—H36C | 109.5 |
| O15—C11—C12—O12 | 63.1 (2) | C22—C23—C24—C25 | -60.4 (2) |
| O22—C11—C12—O12 | -172.08 (17) | O24—C24—C25—O25 | -178.52 (18) |
| O15—C11—C12—C13 | -56.3 (2) | C23—C24—C25—O25 | 62.1 (2) |
| O22—C11—C12—C13 | 68.6 (2) | O24—C24—C25—C26 | -58.4 (3) |
| O12—C12—C13—O13 | 62.0 (2) | C23—C24—C25—C26 | -177.72 (19) |
| C11—C12—C13—O13 | 176.82 (17) | C24—C23—O23—C31 | -113.6 (2) |
| O12—C12—C13—C14 | -59.2 (2) | C22—C23—O23—C31 | 122.95 (19) |
| C11—C12—C13—C14 | 55.6 (2) | O27—C21—O25—C25 | -65.9 (2) |
| O13—C13—C14—O14 | 64.7 (2) | C22—C21—O25—C25 | 55.2 (2) |
| C12—C13—C14—O14 | -175.75 (17) | C26—C25—O25—C21 | 174.83 (19) |
| O13—C13—C14—C15 | -175.54 (17) | C24—C25—O25—C21 | -61.7 (2) |
| C12—C13—C14—C15 | -56.0 (2) | O25—C21—O27—C27 | -72.3 (2) |
| O14—C14—C15—O15 | 176.01 (17) | C22—C21—O27—C27 | 163.6 (2) |
| C13—C14—C15—O15 | 55.9 (2) | C23—O23—C31—O35 | -76.1 (2) |
| O14—C14—C15—C16 | -65.4 (2) | C23—O23—C31—C32 | 162.03 (16) |
| C13—C14—C15—C16 | 174.51 (19) | O23—C31—C32—O32 | -169.04 (17) |
| O22—C11—O15—C15 | -63.2 (2) | O35—C31—C32—O32 | 68.5 (2) |
| C12—C11—O15—C15 | 59.1 (2) | O23—C31—C32—C33 | 66.5 (2) |
| C16—C15—O15—C11 | 179.25 (18) | O35—C31—C32—C33 | -55.9 (2) |
| C14—C15—O15—C11 | -58.6 (2) | O32—C32—C33—O33 | 56.2 (2) |
| O27—C21—C22—O22 | -165.21 (16) | C31—C32—C33—O33 | 178.75 (18) |
| O25—C21—C22—O22 | 70.5 (2) | O32—C32—C33—C34 | -68.5 (2) |
| O27—C21—C22—C23 | 75.1 (2) | C31—C32—C33—C34 | 54.0 (2) |
| O25—C21—C22—C23 | -49.3 (2) | O33—C33—C34—O34 | 59.9 (2) |
| O15—C11—O22—C22 | -72.0 (2) | C32—C33—C34—O34 | -177.17 (18) |
| C12—C11—O22—C22 | 164.44 (18) | O33—C33—C34—C35 | -178.26 (18) |
| C21—C22—O22—C11 | 91.8 (2) | C32—C33—C34—C35 | -55.3 (2) |
| C23—C22—O22—C11 | -147.87 (18) | O34—C34—C35—O35 | 177.68 (17) |

| | | | |
|-----------------|--------------|-----------------|-------------|
| O22—C22—C23—O23 | 55.3 (2) | C33—C34—C35—O35 | 57.5 (2) |
| C21—C22—C23—O23 | 176.42 (18) | O34—C34—C35—C36 | -63.3 (2) |
| O22—C22—C23—C24 | -67.6 (2) | C33—C34—C35—C36 | 176.44 (19) |
| C21—C22—C23—C24 | 53.5 (2) | O23—C31—O35—C35 | -59.2 (2) |
| O23—C23—C24—O24 | 57.5 (2) | C32—C31—O35—C35 | 61.8 (2) |
| C22—C23—C24—O24 | -178.80 (17) | C36—C35—O35—C31 | 174.92 (19) |
| O23—C23—C24—C25 | 175.84 (17) | C34—C35—O35—C31 | -62.4 (2) |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H... <i>A</i> | <i>D</i> —H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> —H... <i>A</i> |
|---|-------------|---------------|-----------------------|-------------------------|
| OW1—H101...O33 ⁱ | 0.88 (3) | 1.85 (3) | 2.726 (2) | 176 (2) |
| OW1—H102...OW3 ⁱⁱ | 0.88 (3) | 1.95 (3) | 2.802 (2) | 162 (2) |
| OW1—H102...O32 ⁱⁱⁱ | 0.88 (3) | 2.55 (3) | 2.976 (2) | 110 (2) |
| OW2—H201...O12 ^{iv} | 0.88 (3) | 2.03 (3) | 2.875 (2) | 163 (2) |
| OW2—H202...O35 ⁱⁱ | 0.87 (3) | 2.08 (3) | 2.877 (2) | 153 (2) |
| OW3—H301...OW5 | 0.88 (3) | 2.04 (3) | 2.845 (2) | 151 (2) |
| OW3—H302...O13 ^v | 0.88 (3) | 1.96 (3) | 2.836 (2) | 176 (2) |
| OW4—H401...OW3 | 0.88 (3) | 1.97 (3) | 2.840 (2) | 168 (2) |
| OW4—H402...OW1 | 0.88 (3) | 1.92 (3) | 2.771 (2) | 160 (2) |
| OW5—H501...O33 ^{vi} | 0.87 (3) | 2.07 (3) | 2.918 (2) | 168 (2) |
| OW5—H502...OW5 ^{vii} | 0.87 (3) | 2.50 (3) | 3.333 (2) | 159 (2) |
| O12—H12 <i>A</i> ...O32 ⁱⁱⁱ | 0.84 | 2.01 | 2.767 (2) | 149 |
| O13—H13 <i>A</i> ...O15 ⁱⁱ | 0.84 | 2.10 | 2.858 (2) | 149 |
| O14—H14 <i>A</i> ...O24 ⁱⁱⁱ | 0.84 | 1.95 | 2.733 (2) | 157 |
| O24—H24 <i>A</i> ...OW2 | 0.84 | 1.88 | 2.722 (2) | 176 |
| O32—H32 <i>A</i> ...OW5 ^{viii} | 0.84 | 2.13 | 2.864 (2) | 146 |
| O33—H33 <i>A</i> ...O34 ⁱ | 0.84 | 1.91 | 2.684 (2) | 152 |
| O34—H34 <i>A</i> ...OW4 | 0.84 | 1.86 | 2.687 (2) | 168 |

Symmetry codes: (i) $-x+3/2, y-1/2, -z+1$; (ii) $x, y-1, z$; (iii) $x+1/2, y-1/2, z$; (iv) $x-1/2, y-1/2, z$; (v) $x, y+1, z$; (vi) $x+1/2, y+1/2, z$; (vii) $-x+2, y, -z+1$; (viii) $x-1/2, y+1/2, z$.