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4-Formylphenyl 2,3,4,6-tetra-*O*-acetyl-β-D-galactopyranoside

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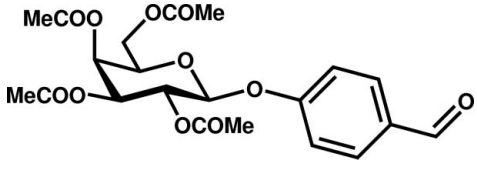
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.035; wR factor = 0.086; data-to-parameter ratio = 9.4.

The galactose ring in the title compound, $C_{21}H_{24}O_{11}$, has a chair conformation with the substituted benzene ring occupying an equatorial position. The crystal packing features C—H···O interactions that lead to the formation of supramolecular layers in the *ab* plane.

Related literature

For the synthesis, see: Benassi *et al.* (2007); Patil *et al.* (2008). For the biological activity of related structures, see: Zheng *et al.* (2010). For the structure of the isomeric allopyranoside and glucopyranoside derivatives, see: Ye *et al.* (2009); Heidelberg *et al.* (2011). For conformational analysis, see: Cremer & Pople (1975).



Experimental

Crystal data

$C_{21}H_{24}O_{11}$
M_r = 452.40
Monoclinic, $P2_1$
a = 11.8358 (4) Å
b = 5.6664 (2) Å
c = 17.5079 (6) Å
β = 109.616 (4)°
V = 1106.05 (7) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 100 K
0.25 × 0.20 × 0.05 mm

Data collection

Agilent Supernova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min} = 0.596, T_{max} = 1.000
10396 measured reflections
2768 independent reflections
2535 reflections with $I > 2\sigma(I)$
R_{int} = 0.051

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.086
S = 1.05
2768 reflections
293 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O9^{i}	1.00	2.39	3.199 (3)	137
C5—H5···O9^{i}	1.00	2.45	3.268 (3)	139
C10—H10b···O3^{ii}	0.98	2.46	3.307 (3)	145
C12—H12b···O5^{iii}	0.98	2.57	3.548 (3)	172
C14—H14c···O11^{iv}	0.98	2.50	3.415 (4)	155

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

Data collection: *CrysAlis PRO* (Agilent, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZ2236).

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4-Formylphenyl 2,3,4,6-tetra-*O*-acetyl-*β*-D-galactopyranoside

Rusnah Syahila Duali Hussen, Thorsten Heidelberg, Nasrul Zamani Mohd Rodzi, Seik Weng Ng and Edward R. T. Tiekink

S1. Comment

The title compound, 4-formyl-phenyl 2,3,4,6-tetra-*O*-acetyl-*β*-D-galactopyranoside, a known species (Benassi *et al.*, 2007; Patil *et al.*, 2008), was prepared as a precursor for the synthesis of galactosylated resveratrol, an anti-oxidizing agent with possible pharmaceutical potential (Zheng *et al.*, 2010).

The structure determination, Fig. 1, confirms the relative stereochemistry. The absolute structure, while not determined experimentally, is based on that of the acetobromogalactose reagent, *i.e. R*, *S*, *S*, *R* and *S* for C1–C5, respectively. The galactose ring has a chair conformation as seen in the puckering parameters (Cremer & Pople, 1975): puckering amplitude (Q) = 0.579 (2) Å, θ = 166.9 (2) °, and φ = 187.6 (10) °. Around the ring, the substituents at the C1, C4 and C5 atoms are equatorial, while at C2 the substituent is axial and that at the C3 atom is biaxial.

The crystal packing is dominated by C–H···O interactions, Table 1, involving all carbonyl atoms, except the O7 atom, as acceptors and either methine- or methyl-H as the donors. These lead to the formation of supramolecular layers that stack along the *c* axis, Fig. 2.

The present report complements the structures reported recently for the isomeric allopyranoside (Ye *et al.*, 2009) and glucopyranoside (Heidelberg *et al.*, 2011) derivatives.

S2. Experimental

2,3,4,6-Tetra-*O*-acetyl-*α*-D-galactopyranosyl bromide (2.0 g) and 4-hydroxybenzaldehyde (1.0 g) were dissolved in chloroform (10 ml) and the mixture was treated with an aqueous solution (5 ml) of sodium carbonate (0.9 g) and tetra-butylammonium bromide (0.3 g). The mixture was heated to reflux under vigorous stirring overnight, after which ethyl acetate was added and the organic layer was washed three times with sodium hydroxide solution (1 N) to remove remaining phenols. After drying the solution over magnesium sulfate and evaporation of the solvent, the target product (1.4 g, 60%) was obtained by crystallization from 2-propanol/hexane (2:1).

^{1}H-NMR (400 MHz, $CDCl_3$): δ 9.93 (s; CHO), 7.85 & 7.11 (AB syst; aromatic 4 H), 5.52 (dd; H2), 5.48 (bd; H4), 5.17 (d; H1), 5.12 (dd; H3), 4.23 (dd; H6a), 4.19–4.10 (m, 2 H; H5, H6b), 2.19–2.02 (3 s, 12 H; Ac); $^3J_{4,5}$ = 10.0 Hz, $^3J_{5,6a}$ = 5.0 Hz, $^3J_{5,6\,b}$ = 2.5 Hz, $^2J_6$ = 12.0 Hz.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 to 1.00 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2 to 1.5U_{equiv}(C). In the absence of significant anomalous scattering effects, 1977 Friedel pairs were averaged in the final refinement. The absolute structure was assigned on the basis of that for the acetobromogalactose reagent.

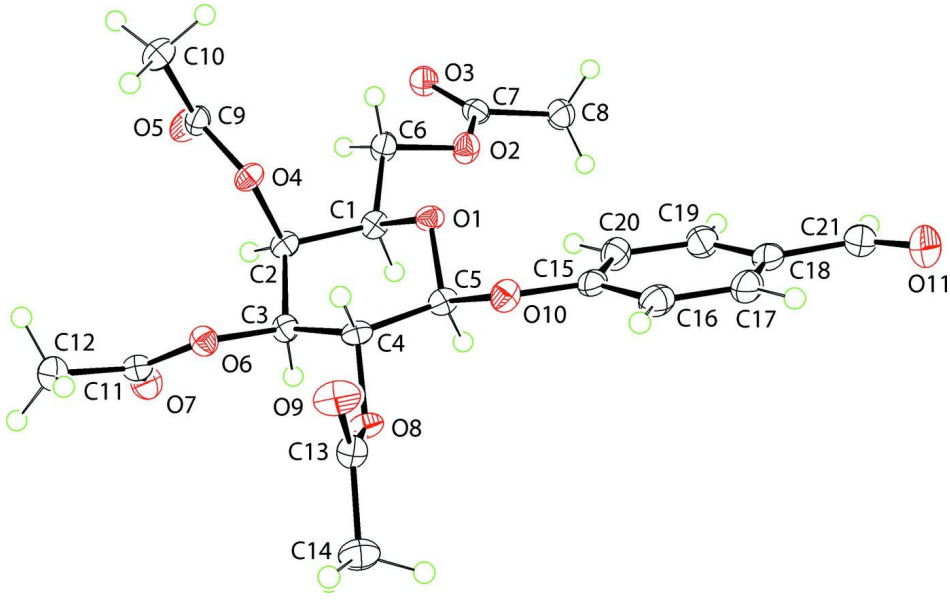

Figure 1

Molecular structure, showing the atom-labelling scheme and displacement ellipsoids at the 50% probability level.

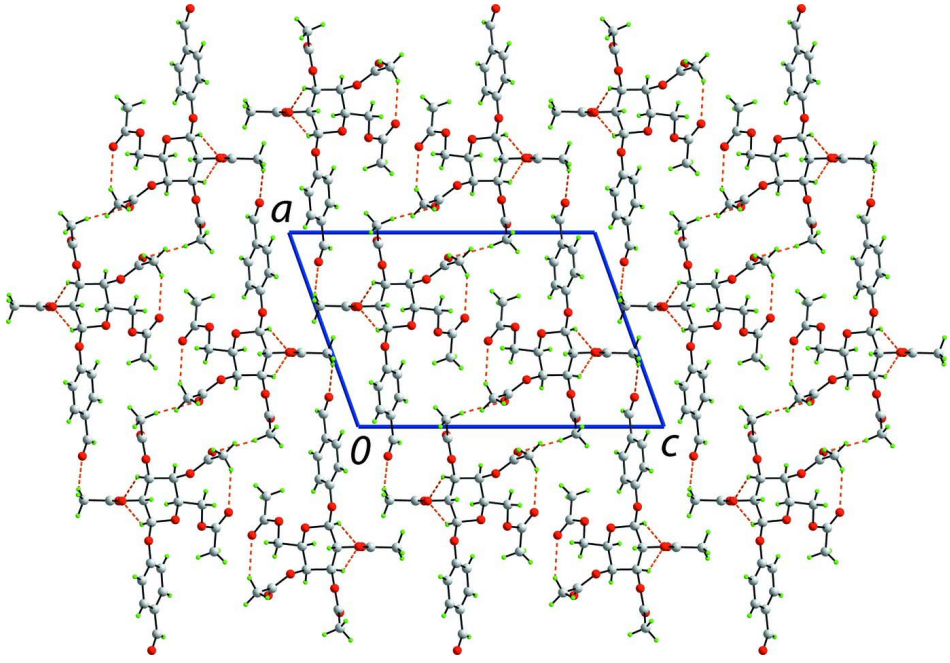

Figure 2

A view in projection down the *b* axis of the unit-cell contents highlighting the stacking of layers. The C—H···O interactions are shown as orange dashed lines.

4-Formylphenyl 2,3,4,6-tetra-*O*-acetyl-*β*-D-galactopyranoside

Crystal data

$C_{21}H_{24}O_{11}$
M_r = 452.40

Monoclinic, $P2_1$
Hall symbol: P 2yb

a = 11.8358 (4) Å
b = 5.6664 (2) Å
c = 17.5079 (6) Å
β = 109.616 (4)°
V = 1106.05 (7) Å^3
Z = 2
F(000) = 476
D_x = 1.358 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5487 reflections
θ = 2.5–29.3°
μ = 0.11 mm^{-1}
T = 100 K
Prism, colourless
0.25 × 0.20 × 0.05 mm

Data collection

Agilent Supernova Dual diffractometer with an Atlas detector
Radiation source: SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.4041 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)

T_{min} = 0.596, T_{max} = 1.000
10396 measured reflections
2768 independent reflections
2535 reflections with $I > 2\sigma(I)$
R_{int} = 0.051
θ_{max} = 27.5°, θ_{min} = 2.5°
h = −15→15
k = −7→6
l = −22→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.086
S = 1.05
2768 reflections
293 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
$w = 1/[\sigma^2(F_o^2) + (0.0424P)^2 + 0.1621P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	0.52081 (12)	0.4982 (3)	0.29215 (8)	0.0188 (3)
O2	0.48738 (13)	0.0896 (3)	0.37091 (9)	0.0240 (4)
O3	0.55891 (13)	−0.1643 (3)	0.47497 (9)	0.0243 (4)
O4	0.75837 (12)	0.6298 (3)	0.39447 (8)	0.0184 (3)
O5	0.86847 (14)	0.3841 (3)	0.49186 (9)	0.0283 (4)
O6	0.83224 (12)	0.7065 (3)	0.26663 (8)	0.0202 (3)
O7	0.95682 (14)	0.3988 (3)	0.27801 (9)	0.0271 (4)
O8	0.61785 (14)	0.7131 (3)	0.12877 (9)	0.0202 (3)

O9	0.61716 (16)	1.1080 (3)	0.13951 (10)	0.0304 (4)
O10	0.41232 (13)	0.7100 (3)	0.18039 (8)	0.0213 (3)
O11	−0.15194 (15)	0.5245 (5)	0.06218 (12)	0.0490 (6)
C1	0.61385 (17)	0.3269 (4)	0.32349 (12)	0.0180 (4)
H1	0.6016	0.1990	0.2821	0.022*
C2	0.73547 (18)	0.4392 (4)	0.33610 (12)	0.0178 (4)
H2	0.8001	0.3174	0.3549	0.021*
C3	0.73421 (17)	0.5437 (4)	0.25518 (12)	0.0177 (4)
H3	0.7421	0.4124	0.2191	0.021*
C4	0.62145 (17)	0.6840 (4)	0.21135 (11)	0.0177 (4)
H4	0.6253	0.8420	0.2376	0.021*
C5	0.50894 (18)	0.5541 (4)	0.21107 (12)	0.0182 (4)
H5	0.4966	0.4081	0.1772	0.022*
C6	0.59907 (18)	0.2203 (4)	0.39814 (12)	0.0216 (5)
H6A	0.6670	0.1138	0.4253	0.026*
H6B	0.5958	0.3454	0.4368	0.026*
C7	0.47923 (18)	−0.0968 (4)	0.41586 (12)	0.0205 (4)
C8	0.35738 (19)	−0.2058 (5)	0.38251 (13)	0.0272 (5)
H8A	0.3622	−0.3731	0.3974	0.041*
H8B	0.3277	−0.1909	0.3233	0.041*
H8C	0.3024	−0.1247	0.4049	0.041*
C9	0.83088 (18)	0.5785 (4)	0.47134 (12)	0.0195 (4)
C10	0.8534 (2)	0.7915 (4)	0.52407 (13)	0.0246 (5)
H10A	0.9152	0.7559	0.5762	0.037*
H10B	0.7791	0.8375	0.5332	0.037*
H10C	0.8807	0.9212	0.4976	0.037*
C11	0.94103 (19)	0.6087 (5)	0.27887 (12)	0.0221 (5)
C12	1.0356 (2)	0.7935 (5)	0.29539 (14)	0.0301 (6)
H12A	1.0988	0.7413	0.2746	0.045*
H12B	1.0701	0.8202	0.3540	0.045*
H12C	1.0003	0.9407	0.2684	0.045*
C13	0.61949 (19)	0.9346 (4)	0.10083 (13)	0.0208 (5)
C14	0.6223 (2)	0.9316 (5)	0.01601 (13)	0.0284 (5)
H14A	0.6497	1.0852	0.0034	0.043*
H14B	0.5416	0.8993	−0.0219	0.043*
H14C	0.6774	0.8081	0.0111	0.043*
C15	0.29775 (18)	0.6182 (5)	0.16131 (12)	0.0214 (5)
C16	0.2083 (2)	0.7649 (5)	0.11247 (13)	0.0269 (5)
H16	0.2281	0.9110	0.0935	0.032*
C17	0.0891 (2)	0.6936 (5)	0.09193 (14)	0.0310 (6)
H17	0.0271	0.7924	0.0588	0.037*
C18	0.0601 (2)	0.4804 (5)	0.11914 (13)	0.0282 (5)
C19	0.1514 (2)	0.3361 (5)	0.16730 (13)	0.0275 (5)
H19	0.1317	0.1889	0.1856	0.033*
C20	0.27087 (19)	0.4037 (4)	0.18905 (13)	0.0246 (5)
H20	0.3328	0.3050	0.2222	0.030*
C21	−0.0667 (2)	0.4052 (6)	0.09904 (14)	0.0369 (6)
H21	−0.0816	0.2530	0.1163	0.044*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0204 (7)	0.0207 (8)	0.0147 (7)	0.0044 (6)	0.0053 (6)	0.0017 (6)
O2	0.0208 (7)	0.0267 (9)	0.0219 (7)	−0.0005 (7)	0.0037 (6)	0.0064 (7)
O3	0.0241 (7)	0.0252 (9)	0.0233 (7)	−0.0009 (7)	0.0075 (6)	0.0057 (7)
O4	0.0216 (7)	0.0151 (7)	0.0159 (6)	0.0016 (6)	0.0025 (6)	−0.0009 (6)
O5	0.0353 (9)	0.0198 (9)	0.0226 (7)	0.0033 (7)	0.0000 (7)	0.0025 (7)
O6	0.0205 (7)	0.0175 (8)	0.0231 (7)	−0.0019 (6)	0.0081 (6)	−0.0008 (6)
O7	0.0248 (8)	0.0258 (10)	0.0309 (8)	0.0023 (7)	0.0095 (7)	0.0016 (8)
O8	0.0305 (8)	0.0153 (8)	0.0149 (7)	0.0001 (7)	0.0078 (6)	−0.0004 (6)
O9	0.0498 (10)	0.0161 (8)	0.0268 (8)	−0.0001 (8)	0.0148 (8)	0.0009 (8)
O10	0.0211 (7)	0.0181 (8)	0.0220 (7)	0.0049 (6)	0.0035 (6)	0.0020 (7)
O11	0.0240 (9)	0.0789 (17)	0.0400 (11)	0.0101 (10)	0.0052 (8)	−0.0055 (11)
C1	0.0201 (9)	0.0133 (10)	0.0193 (9)	0.0013 (9)	0.0048 (8)	0.0014 (8)
C2	0.0214 (10)	0.0131 (11)	0.0181 (9)	0.0008 (8)	0.0054 (8)	−0.0015 (8)
C3	0.0189 (10)	0.0142 (11)	0.0200 (10)	−0.0020 (8)	0.0064 (8)	−0.0014 (8)
C4	0.0238 (10)	0.0151 (11)	0.0144 (9)	0.0020 (9)	0.0065 (8)	0.0005 (8)
C5	0.0218 (10)	0.0146 (11)	0.0168 (9)	0.0034 (8)	0.0048 (8)	0.0008 (8)
C6	0.0197 (10)	0.0224 (12)	0.0204 (10)	−0.0029 (9)	0.0039 (8)	0.0039 (9)
C7	0.0219 (10)	0.0206 (11)	0.0217 (10)	−0.0018 (9)	0.0107 (9)	−0.0013 (9)
C8	0.0241 (11)	0.0309 (14)	0.0266 (11)	−0.0053 (10)	0.0087 (9)	0.0010 (11)
C9	0.0172 (9)	0.0210 (12)	0.0180 (9)	−0.0019 (9)	0.0027 (8)	0.0022 (9)
C10	0.0272 (11)	0.0224 (12)	0.0200 (10)	0.0011 (10)	0.0024 (9)	−0.0004 (10)
C11	0.0230 (10)	0.0270 (13)	0.0167 (9)	−0.0005 (10)	0.0072 (8)	−0.0005 (10)
C12	0.0255 (11)	0.0358 (15)	0.0290 (11)	−0.0071 (11)	0.0092 (10)	−0.0055 (11)
C13	0.0214 (10)	0.0174 (12)	0.0222 (10)	0.0005 (9)	0.0056 (9)	0.0034 (9)
C14	0.0380 (12)	0.0267 (14)	0.0215 (10)	0.0039 (11)	0.0113 (10)	0.0041 (10)
C15	0.0213 (10)	0.0248 (12)	0.0170 (9)	0.0027 (9)	0.0050 (8)	−0.0051 (9)
C16	0.0297 (12)	0.0280 (13)	0.0216 (10)	0.0069 (10)	0.0067 (10)	0.0003 (10)
C17	0.0243 (11)	0.0433 (16)	0.0222 (10)	0.0113 (11)	0.0036 (9)	−0.0008 (11)
C18	0.0252 (11)	0.0389 (15)	0.0201 (10)	0.0029 (11)	0.0070 (9)	−0.0085 (11)
C19	0.0269 (11)	0.0276 (13)	0.0279 (11)	−0.0027 (10)	0.0091 (10)	−0.0070 (10)
C20	0.0227 (10)	0.0235 (12)	0.0250 (10)	0.0036 (10)	0.0046 (9)	−0.0003 (10)
C21	0.0257 (12)	0.0567 (19)	0.0284 (12)	0.0004 (13)	0.0092 (10)	−0.0121 (13)

Geometric parameters (Å, º)

O1—C5	1.415 (2)	C7—C8	1.496 (3)
O1—C1	1.432 (2)	C8—H8A	0.9800
O2—C7	1.340 (3)	C8—H8B	0.9800
O2—C6	1.449 (3)	C8—H8C	0.9800
O3—C7	1.205 (3)	C9—C10	1.488 (3)
O4—C9	1.362 (2)	C10—H10A	0.9800
O4—C2	1.448 (2)	C10—H10B	0.9800
O5—C9	1.197 (3)	C10—H10C	0.9800
O6—C11	1.351 (3)	C11—C12	1.489 (3)
O6—C3	1.443 (2)	C12—H12A	0.9800

O7—C11	1.205 (3)	C12—H12B	0.9800
O8—C13	1.350 (3)	C12—H12C	0.9800
O8—C4	1.441 (2)	C13—C14	1.497 (3)
O9—C13	1.199 (3)	C14—H14A	0.9800
O10—C15	1.384 (3)	C14—H14B	0.9800
O10—C5	1.401 (2)	C14—H14C	0.9800
O11—C21	1.206 (3)	C15—C20	1.385 (3)
C1—C6	1.502 (3)	C15—C16	1.391 (3)
C1—C2	1.521 (3)	C16—C17	1.393 (3)
C1—H1	1.0000	C16—H16	0.9500
C2—C3	1.531 (3)	C17—C18	1.384 (4)
C2—H2	1.0000	C17—H17	0.9500
C3—C4	1.521 (3)	C18—C19	1.391 (3)
C3—H3	1.0000	C18—C21	1.484 (3)
C4—C5	1.520 (3)	C19—C20	1.389 (3)
C4—H4	1.0000	C19—H19	0.9500
C5—H5	1.0000	C20—H20	0.9500
C6—H6A	0.9900	C21—H21	0.9500
C6—H6B	0.9900		

C5—O1—C1	109.93 (14)	H8B—C8—H8C	109.5
C7—O2—C6	116.57 (16)	O5—C9—O4	122.7 (2)
C9—O4—C2	116.39 (17)	O5—C9—C10	126.07 (19)
C11—O6—C3	116.03 (17)	O4—C9—C10	111.22 (19)
C13—O8—C4	118.07 (17)	C9—C10—H10A	109.5
C15—O10—C5	117.57 (17)	C9—C10—H10B	109.5
O1—C1—C6	107.86 (16)	H10A—C10—H10B	109.5
O1—C1—C2	109.91 (17)	C9—C10—H10C	109.5
C6—C1—C2	115.02 (17)	H10A—C10—H10C	109.5
O1—C1—H1	107.9	H10B—C10—H10C	109.5
C6—C1—H1	107.9	O7—C11—O6	123.1 (2)
C2—C1—H1	107.9	O7—C11—C12	125.9 (2)
O4—C2—C1	111.00 (16)	O6—C11—C12	110.9 (2)
O4—C2—C3	107.82 (17)	C11—C12—H12A	109.5
C1—C2—C3	108.23 (16)	C11—C12—H12B	109.5
O4—C2—H2	109.9	H12A—C12—H12B	109.5
C1—C2—H2	109.9	C11—C12—H12C	109.5
C3—C2—H2	109.9	H12A—C12—H12C	109.5
O6—C3—C4	105.32 (16)	H12B—C12—H12C	109.5
O6—C3—C2	111.18 (16)	O9—C13—O8	123.49 (19)
C4—C3—C2	113.79 (16)	O9—C13—C14	125.6 (2)
O6—C3—H3	108.8	O8—C13—C14	110.91 (19)
C4—C3—H3	108.8	C13—C14—H14A	109.5
C2—C3—H3	108.8	C13—C14—H14B	109.5
O8—C4—C5	108.73 (16)	H14A—C14—H14B	109.5
O8—C4—C3	106.93 (15)	C13—C14—H14C	109.5
C5—C4—C3	111.57 (17)	H14A—C14—H14C	109.5
O8—C4—H4	109.9	H14B—C14—H14C	109.5

C5—C4—H4	109.9	O10—C15—C20	124.53 (19)
C3—C4—H4	109.9	O10—C15—C16	113.9 (2)
O10—C5—O1	108.61 (15)	C20—C15—C16	121.5 (2)
O10—C5—C4	107.29 (17)	C15—C16—C17	118.7 (2)
O1—C5—C4	108.26 (16)	C15—C16—H16	120.6
O10—C5—H5	110.9	C17—C16—H16	120.6
O1—C5—H5	110.9	C18—C17—C16	120.8 (2)
C4—C5—H5	110.9	C18—C17—H17	119.6
O2—C6—C1	106.20 (16)	C16—C17—H17	119.6
O2—C6—H6A	110.5	C17—C18—C19	119.3 (2)
C1—C6—H6A	110.5	C17—C18—C21	121.1 (2)
O2—C6—H6B	110.5	C19—C18—C21	119.5 (3)
C1—C6—H6B	110.5	C20—C19—C18	121.1 (2)
H6A—C6—H6B	108.7	C20—C19—H19	119.5
O3—C7—O2	124.39 (19)	C18—C19—H19	119.5
O3—C7—C8	125.4 (2)	C15—C20—C19	118.6 (2)
O2—C7—C8	110.25 (18)	C15—C20—H20	120.7
C7—C8—H8A	109.5	C19—C20—H20	120.7
C7—C8—H8B	109.5	O11—C21—C18	124.3 (3)
H8A—C8—H8B	109.5	O11—C21—H21	117.8
C7—C8—H8C	109.5	C18—C21—H21	117.8
H8A—C8—H8C	109.5		

C5—O1—C1—C6	−163.31 (17)	C3—C4—C5—O1	54.5 (2)
C5—O1—C1—C2	70.6 (2)	C7—O2—C6—C1	151.59 (18)
C9—O4—C2—C1	100.1 (2)	O1—C1—C6—O2	66.5 (2)
C9—O4—C2—C3	−141.53 (17)	C2—C1—C6—O2	−170.44 (17)
O1—C1—C2—O4	61.5 (2)	C6—O2—C7—O3	−2.2 (3)
C6—C1—C2—O4	−60.5 (2)	C6—O2—C7—C8	177.27 (18)
O1—C1—C2—C3	−56.7 (2)	C2—O4—C9—O5	−4.5 (3)
C6—C1—C2—C3	−178.60 (18)	C2—O4—C9—C10	176.56 (17)
C11—O6—C3—C4	−158.49 (16)	C3—O6—C11—O7	2.3 (3)
C11—O6—C3—C2	77.8 (2)	C3—O6—C11—C12	−176.14 (16)
O4—C2—C3—O6	44.2 (2)	C4—O8—C13—O9	−4.2 (3)
C1—C2—C3—O6	164.33 (16)	C4—O8—C13—C14	176.78 (17)
O4—C2—C3—C4	−74.5 (2)	C5—O10—C15—C20	17.7 (3)
C1—C2—C3—C4	45.6 (2)	C5—O10—C15—C16	−164.09 (18)
C13—O8—C4—C5	120.0 (2)	O10—C15—C16—C17	−177.76 (19)
C13—O8—C4—C3	−119.44 (19)	C20—C15—C16—C17	0.5 (3)
O6—C3—C4—O8	73.70 (19)	C15—C16—C17—C18	−0.3 (3)
C2—C3—C4—O8	−164.29 (17)	C16—C17—C18—C19	−0.2 (3)
O6—C3—C4—C5	−167.54 (16)	C16—C17—C18—C21	178.7 (2)
C2—C3—C4—C5	−45.5 (2)	C17—C18—C19—C20	0.6 (3)
C15—O10—C5—O1	−74.1 (2)	C21—C18—C19—C20	−178.3 (2)
C15—O10—C5—C4	169.11 (16)	O10—C15—C20—C19	177.9 (2)
C1—O1—C5—O10	176.18 (16)	C16—C15—C20—C19	−0.2 (3)
C1—O1—C5—C4	−67.6 (2)	C18—C19—C20—C15	−0.4 (3)
O8—C4—C5—O10	−70.80 (19)	C17—C18—C21—O11	−3.8 (4)

C3—C4—C5—O10	171.51 (15)	C19—C18—C21—O11	175.1 (2)
O8—C4—C5—O1	172.16 (16)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O9^{i}	1.00	2.39	3.199 (3)	137
C5—H5···O9^{i}	1.00	2.45	3.268 (3)	139
C10—H10b···O3^{ii}	0.98	2.46	3.307 (3)	145
C12—H12b···O5^{iii}	0.98	2.57	3.548 (3)	172
C14—H14c···O11^{iv}	0.98	2.50	3.415 (4)	155

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