Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

5-Hydroxy-6,7-dimethoxy-2-[(*E*)-1-(4-methoxyphenyl)-methylidene]-3,4-dihydro-2*H*-napthalen-1-one

Alan B. Turner and William T. A. Harrison*

Department of Chemistry, University of Aberdeen, Meston Walk, Aberdeen AB24 3UE, Scotland

Correspondence e-mail:
w.harrison@abdn.ac.uk

Received 10 November 2005
Accepted 8 December 2005
Online 23 December 2005

The title compound, $C_{20}H_{20}O_5$, possesses normal geometrical parameters. O—H···O bonds and possible C—H···O interactions are present.

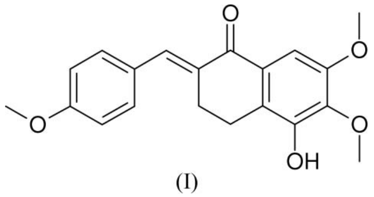
Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.045
wR factor = 0.114
Data-to-parameter ratio = 16.5

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

Comment

The title compound, commonly called 2(4-methoxybenzylidene)-5-hydroxy-6,7-dimethoxytetral-1-one, (I), prepared by the condensation of anisaldehyde with 5-hydroxy-6,7-dimethoxytetral-1-one, was found to be sensitive to aerial oxidation. Its crystal structure was determined in the hope of identifying structural features which might explain its ready oxidation.



(I)

All the geometrical parameters for (I) (Fig. 1 and Table 1) lie within their expected ranges (Allen *et al.*, 1995). The six-membered C9–C14 ring adopts an envelope configuration, with C9 and C11–C14 approximately coplanar [r.m.s. deviation = 0.054 Å, maximum deviation = 0.0846 (10) for atom C14] and C10 in the flap position, displaced by 0.637 (2) Å from the mean plane. The dihedral angle between the C3 and C15 benzene rings is 66.90 (4)°. The terminal methyl groups are displaced from their attached benzene ring C-atom mean planes by 0.066 (3), 0.079 (3), and −1.113 (3) Å for atoms C1, C19, and C20, respectively.

Various O—H···O and possible C—H···O interactions exist in the crystal structure of (I) (Table 2). The O3—H1 group forms a bifurcated intramolecular/intermolecular hydrogen bond. The intermolecular O—H···O connectivity results in chains of (I) propagating along [010]. Pairs of inversion-symmetry-related C3-benzene rings interact by π–π^{i} [symmetry code: (i) $\frac{3}{2} - x, \frac{1}{2} - y, 1 - z$] stacking with a centroid separation of 3.6516 (9) Å and an interplane separation of 3.451 Å. Overall, we cannot observe any unusual structural features in (I) that correlate with its sensitivity to oxidation.

Experimental

A stirred solution of 102 mg of 5-hydroxy-6,7-dimethoxytetral-1-one (Cooke & Robinson, 1970) and 100 mg of 4-methoxybenzaldehyde in ethanol (10 ml) was treated dropwise with concentrated sulfuric acid (1.2 ml) over a period of 10 min (slight exothermic reaction) and the pale-brown solution was left at room temperature for 5 d. The crystals that had formed were collected and washed with cold ethanol. Recrystallization from ethanol gave (I) as pale-yellow needles (124 mg, 80%; m.p. 412–413 K) UV-vis: λ_{max} 334 nm (ε = 16,100); ν_{max} (cm^{-1}) 3312, 1654, 1600, 1578, 1252, 1167, 1120, 1009, 928, 824; ^{13}C NMR (100 MHz): δ 21.27, 26.78, 55.33, 55.95, 61.06, 102.81, 113.92, 123.61, 128.51, 129.17, 131.66, 133.79, 136.33, 139.41, 145.70, 150.80, 159.85, and 187.28. The crystals were sensitive to oxidation, turning red on exposure to air. TLC investigations revealed a complex mixture of oxidation products. The compound was also oxidized by high-potential quinones in methanol or dioxane solution, again giving an intractable mixture of products.

Crystal data

$C_{20}H_{20}O_5$
M_r = 340.36
Monoclinic, $C2/c$
a = 18.3264 (5) Å
b = 13.3022 (5) Å
c = 15.7673 (6) Å
β = 119.1321 (16)°
V = 3357.5 (2) Å^3
Z = 8
D_x = 1.347 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3939 reflections
θ = 2.9–27.5°
μ = 0.10 mm^{-1}
T = 120 (2) K
Block (cut from needle), pale yellow
0.22 × 0.20 × 0.12 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)
T_{min} = 0.979, T_{max} = 0.989
20240 measured reflections
3856 independent reflections
3231 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 27.5°
$h = -23 \rightarrow 23$
$k = -17 \rightarrow 17$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.114
S = 1.07
3856 reflections
233 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0447P)^2 + 3.3509P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0030 (5)

Table 1
Selected geometric parameters (Å, °).

C5—C8	1.460 (2)	C13—C14	1.4814 (19)
C9—C14	1.484 (2)		
C9—C10—C11—C12	52.21 (16)	C12—C13—C14—C9	14.32 (19)
C10—C11—C12—C13	−27.58 (18)	C10—C9—C14—C13	13.43 (18)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H1···O2^i	0.86 (2)	2.02 (2)	2.8248 (15)	155 (2)
O3—H1···O5	0.86 (2)	2.29 (2)	2.7308 (15)	112 (2)
C6—H6···O5^{ii}	0.95	2.48	3.2668 (18)	140
C7—H7···O3^{ii}	0.95	2.59	3.4591 (18)	153

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

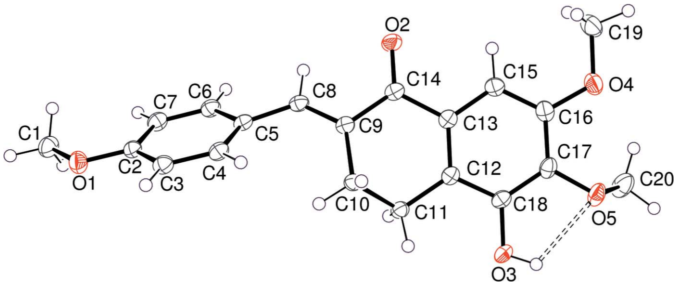


Figure 1
View of (I) (50% displacement ellipsoids; H atoms are drawn as small spheres of arbitrary radius). The dashed lines represent the O—H···O intramolecular interaction.

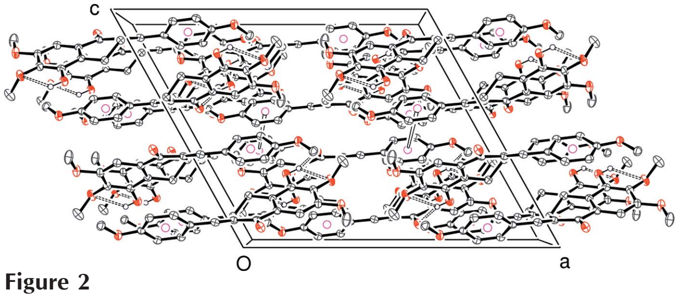


Figure 2
View showing crystal packing (all H atoms omitted except OH; pink circles used to represent the centroid of the C3 benzene ring).

The O-bound H atom was located in a difference map. Its position was freely refined with the constraint $U_{iso}(H) = 1.2U_{eq}(O)$ applied. All the C-bound H atoms were placed in calculated positions (C—H = 0.95–0.99 Å) and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$(methyl C). The methyl groups were allowed to rotate to best fit the electron density.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997), and *SORTAV* (Blessing, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

We thank the EPSRC National Crystallography Service (University of Southampton) for data collection.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1995). *International Tables for Crystallography*, Vol. C, Section 9.5, pp. 685–706. Dordrecht: Kluwer Academic Publishers.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Bruker (2003). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cooke, R. G. & Robinson, J. B. (1970). *Aust. J. Chem.* **23**, 1695–1698.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

supporting information

Acta Cryst. (2006). E**62**, o386–o387 [doi:10.1107/S1600536805041097]

5-Hydroxy-6,7-dimethoxy-2-[(*E*)-1-(4-methoxyphenyl)methylidene]-3,4-dihydro-2*H*-napthalen-1-one

Alan B. Turner and William T. A. Harrison

S1. Comment

The title compound, commonly called 2(4-methoxybenzylidene)-5-hydroxy-6,7-dimethoxytetral-1-one, (I), prepared by the condensation of anisaldehyde with 5-hydroxy-6,7-dimethoxytetral-1-one, was found to be sensitive to aerial oxidation. Its crystal structure was determined in the hope of identifying structural features which might explain its ready oxidation.

All the geometrical parameters for (I) (Fig. 1 and Table 1) lie within their expected ranges (Allen *et al.*, 1995). The six-membered C9–C14 ring adopts an envelope configuration, with C9 and C11–C14 approximately co-planar [r.m.s. deviation = 0.054 Å, maximum deviation = −0.0846 (10) for atom C14] and C10 in the flap position, displaced by 0.637 (2) Å from the best plane. The dihedral angle between the C3 and C15 benzene rings is 66.90 (4)°. The terminal methyl groups are displaced from their attached benzene ring C-atom best planes by 0.066 (3), 0.079 (3), and −1.113 (3) Å for atoms C1, C19, and C20, respectively.

Various O—H···O and possible C—H···O interactions exist in the crystal structure of (I) (Table 2). The O3—H1 moiety forms a bifurcated intramolecular/intermolecular hydrogen bond. The intermolecular O—H···O connectivity results in chains of (I) propagating in [010]. Pairs of inversion-symmetry-generated C3-benzene rings interact by π–π^{i} [symmetry code: (i) $3/2 - x$, $1/2 - y$, $1 - z$] stacking with a centroid separation of 3.6516 (9) Å and an interplane separation of 3.451 Å. Overall, we cannot observe any unusual structural features in (I) that correlate with its sensitivity to oxidation.

S2. Experimental

A stirred solution of 102 mg of 5-hydroxy-6,7-dimethoxytetral-1-one (Cooke & Robinson, 1970) and 100 mg of 4-methoxybenzaldehyde in ethanol (10 ml) was treated dropwise with concentrated sulfuric acid (1.2 ml) over a period of 10 min (slight exothermic reaction) and the pale-brown solution was left at room temperature for 5 d. The crystals that had formed were collected and washed with cold ethanol. Recrystallization from ethanol gave (I) as pale-yellow needles (124 mg, 80%; m.p. 412–413 K) UV-vis: λ_{max} 334 nm (ε = 16,100); ν_{max} (cm^{-1}) 3312, 1654, 1600, 1578, 1252, 1167, 1120, 1009, 928, 824; ^{13}C NMR (100 MHz): δ 21.27, 26.78, 55.33, 55.95, 61.06, 102.81, 113.92, 123.61, 128.51, 129.17, 131.66, 133.79, 136.33, 139.41, 145.70, 150.80, 159.85, and 187.28. The crystals were sensitive to oxidation, turning red on exposure to air. TLC investigations revealed a complex mixture of oxidation products. The compound was also oxidized by high-potential quinones in methanol or dioxane solution, again giving an intractable mixture of products.

S3. Refinement

The O-bound H atom was located in a difference map. Its position was freely refined with the constraint U_{iso}(H) = $1.2U_{eq}$(O) applied. All the C-bound H atoms were placed in calculated positions (C—H = 0.95–0.99 Å) and refined as riding, with U_{iso}(H) = $1.2U_{eq}$(C) or $1.5U_{eq}$(methyl C). The methyl groups was allowed to rotate to best fit the electron density.

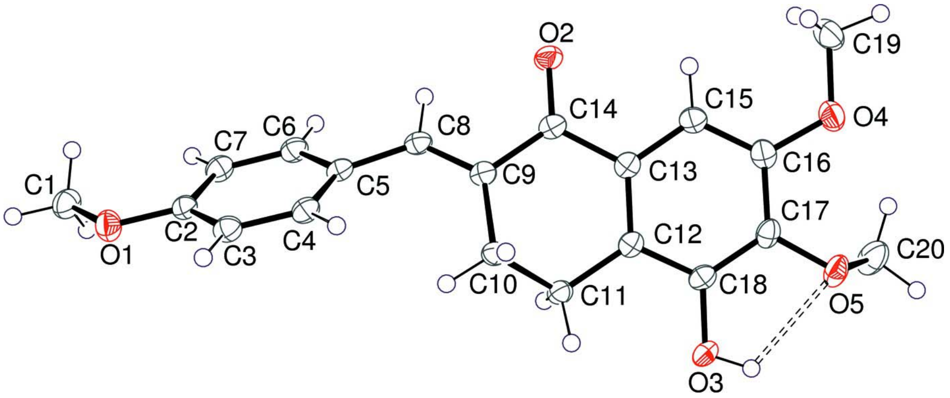


Figure 1

View of (I) (50% displacement ellipsoids; H atoms are drawn as small spheres of arbitrary radius). The dashed lines represent the O—H···O intramolecular interaction.

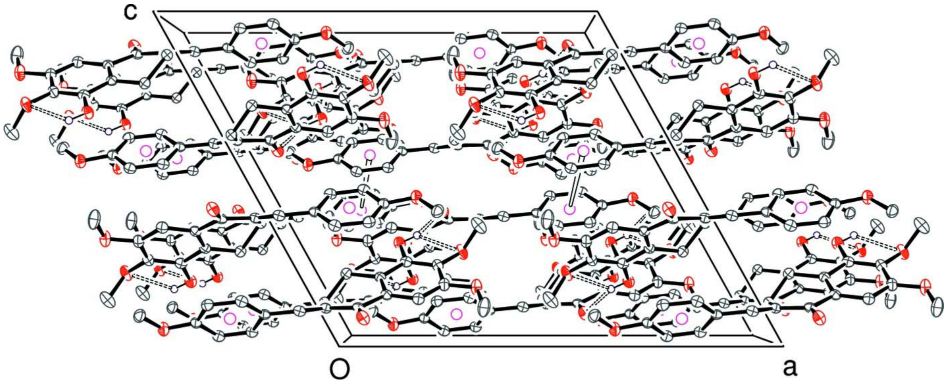


Figure 2

View showing crystal packing (all H atoms omitted except OH; pink circles used to represent the centroid of the C3 benzene ring).

5-Hydroxy-6,7-dimethoxy-2-[(*E*)-1-(4-methoxyphenyl)methylidene]-3,4-dihydro- 2*H*-napthalen-1-one 2-(4-methoxybenzylidene)-5-hydroxy-6,7-dimethoxytetral-1-one

Crystal data

$C_{20}H_{20}O_5$	$F(000) = 1440$
$M_r = 340.36$	$D_x = 1.347$ Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -C 2yc	Cell parameters from 3939 reflections
$a = 18.3264$ (5) Å	$\theta = 2.9$–$27.5°$
$b = 13.3022$ (5) Å	$\mu = 0.10$ mm^{-1}
$c = 15.7673$ (6) Å	$T = 120$ K
$\beta = 119.1321$ (16)°	Block, pale yellow
$V = 3357.5$ (2) Å^3	0.22 × 0.20 × 0.12 mm
$Z = 8$	

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)
$T_{\min} = 0.979$, $T_{\max} = 0.989$
20240 measured reflections
3856 independent reflections
3231 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.042$
$\theta_{\max} = 27.5°$, $\theta_{\min} = 3.1°$
$h = -23 \rightarrow 23$
$k = -17 \rightarrow 17$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.114$
$S = 1.07$
3856 reflections
233 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difmap (O-H) and geom (C-H)
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0447P)^2 + 3.3509P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.28$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}
Extinction correction: *SHELXL97*, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0030 (5)

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	$U_{\rm iso}$*/$U_{\rm eq}$
C1	0.89149 (10)	0.29139 (12)	0.40275 (12)	0.0293 (4)
H1A	0.9509	0.3029	0.4249	0.044*
H1B	0.8599	0.2957	0.3318	0.044*
H1C	0.8841	0.2245	0.4234	0.044*
C2	0.78079 (9)	0.35687 (11)	0.42500 (10)	0.0215 (3)
C3	0.75285 (9)	0.42983 (11)	0.46647 (11)	0.0232 (3)
H3	0.7889	0.4831	0.5033	0.028*
C4	0.67302 (9)	0.42464 (11)	0.45401 (10)	0.0215 (3)
H4	0.6545	0.4747	0.4822	0.026*
C5	0.61872 (9)	0.34639 (10)	0.40022 (10)	0.0200 (3)
C6	0.64855 (9)	0.27408 (11)	0.36052 (11)	0.0228 (3)
H6	0.6133	0.2197	0.3251	0.027*
C7	0.72791 (9)	0.27932 (11)	0.37116 (11)	0.0236 (3)
H7	0.7461	0.2302	0.3418	0.028*
C8	0.53592 (9)	0.33069 (11)	0.38987 (10)	0.0206 (3)

H8	0.5207	0.2621	0.3878	0.025*
C9	0.47747 (9)	0.39649 (10)	0.38278 (10)	0.0198 (3)
C10	0.47752 (9)	0.50872 (10)	0.36893 (10)	0.0201 (3)
H10A	0.5238	0.5272	0.3565	0.024*
H10B	0.4865	0.5435	0.4288	0.024*
C11	0.39419 (9)	0.54235 (11)	0.28324 (10)	0.0208 (3)
H11A	0.3924	0.6167	0.2799	0.025*
H11B	0.3899	0.5167	0.2220	0.025*
C12	0.32103 (8)	0.50453 (10)	0.29296 (10)	0.0191 (3)
C13	0.32654 (9)	0.41647 (10)	0.34387 (10)	0.0197 (3)
C14	0.40214 (9)	0.35263 (10)	0.38071 (10)	0.0205 (3)
C15	0.25951 (9)	0.38177 (11)	0.35495 (11)	0.0220 (3)
H15	0.2650	0.3219	0.3904	0.026*
C16	0.18553 (9)	0.43493 (11)	0.31411 (11)	0.0231 (3)
C17	0.17730 (9)	0.52133 (11)	0.25892 (11)	0.0225 (3)
C18	0.24411 (9)	0.55464 (11)	0.24764 (10)	0.0206 (3)
C19	0.12370 (12)	0.32182 (13)	0.37813 (16)	0.0405 (4)
H19A	0.0704	0.3096	0.3769	0.061*
H19B	0.1677	0.3323	0.4453	0.061*
H19C	0.1378	0.2636	0.3508	0.061*
C20	0.03432 (10)	0.53371 (13)	0.13886 (14)	0.0375 (4)
H20A	−0.0113	0.5828	0.1106	0.056*
H20B	0.0167	0.4755	0.1625	0.056*
H20C	0.0485	0.5119	0.0893	0.056*
O1	0.86164 (6)	0.36594 (8)	0.44389 (8)	0.0279 (3)
O2	0.40156 (6)	0.26562 (8)	0.40761 (8)	0.0273 (3)
O3	0.23724 (7)	0.63713 (8)	0.19300 (8)	0.0249 (3)
H1	0.1879 (12)	0.6628 (14)	0.1686 (13)	0.030*
O4	0.11660 (7)	0.40919 (8)	0.32184 (9)	0.0307 (3)
O5	0.10613 (6)	0.57905 (8)	0.21820 (8)	0.0282 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0298 (8)	0.0329 (9)	0.0298 (8)	0.0080 (7)	0.0181 (7)	0.0051 (7)
C2	0.0208 (7)	0.0228 (7)	0.0202 (7)	0.0030 (5)	0.0095 (6)	0.0049 (6)
C3	0.0245 (7)	0.0198 (7)	0.0218 (7)	−0.0007 (6)	0.0085 (6)	−0.0006 (6)
C4	0.0241 (7)	0.0183 (7)	0.0201 (7)	0.0025 (5)	0.0091 (6)	−0.0008 (5)
C5	0.0219 (7)	0.0176 (7)	0.0173 (7)	0.0025 (5)	0.0071 (6)	0.0027 (5)
C6	0.0249 (7)	0.0175 (7)	0.0230 (7)	0.0005 (6)	0.0093 (6)	−0.0009 (6)
C7	0.0275 (7)	0.0209 (7)	0.0227 (7)	0.0038 (6)	0.0126 (6)	−0.0006 (6)
C8	0.0230 (7)	0.0157 (7)	0.0194 (7)	−0.0007 (5)	0.0073 (6)	0.0004 (5)
C9	0.0204 (7)	0.0183 (7)	0.0176 (7)	−0.0009 (5)	0.0067 (6)	−0.0005 (5)
C10	0.0207 (7)	0.0170 (7)	0.0218 (7)	−0.0005 (5)	0.0098 (6)	0.0003 (5)
C11	0.0229 (7)	0.0177 (7)	0.0227 (7)	0.0025 (5)	0.0119 (6)	0.0017 (5)
C12	0.0206 (7)	0.0172 (7)	0.0180 (7)	0.0003 (5)	0.0083 (6)	−0.0039 (5)
C13	0.0203 (7)	0.0176 (7)	0.0187 (7)	−0.0012 (5)	0.0074 (6)	−0.0041 (5)
C14	0.0219 (7)	0.0174 (7)	0.0179 (7)	−0.0005 (5)	0.0063 (6)	−0.0013 (5)

C15	0.0244 (7)	0.0173 (7)	0.0237 (7)	−0.0026 (5)	0.0112 (6)	−0.0037 (6)
C16	0.0225 (7)	0.0199 (7)	0.0283 (8)	−0.0044 (6)	0.0135 (6)	−0.0083 (6)
C17	0.0199 (7)	0.0186 (7)	0.0269 (8)	0.0009 (5)	0.0097 (6)	−0.0067 (6)
C18	0.0241 (7)	0.0153 (7)	0.0203 (7)	0.0007 (5)	0.0091 (6)	−0.0031 (5)
C19	0.0414 (10)	0.0286 (9)	0.0667 (13)	−0.0028 (7)	0.0382 (10)	0.0037 (8)
C20	0.0239 (8)	0.0273 (9)	0.0457 (11)	0.0008 (6)	0.0047 (8)	−0.0037 (7)
O1	0.0229 (5)	0.0297 (6)	0.0331 (6)	0.0009 (4)	0.0151 (5)	−0.0015 (5)
O2	0.0232 (5)	0.0178 (5)	0.0358 (6)	−0.0010 (4)	0.0105 (5)	0.0046 (4)
O3	0.0227 (5)	0.0195 (5)	0.0307 (6)	0.0063 (4)	0.0115 (5)	0.0049 (4)
O4	0.0259 (6)	0.0259 (6)	0.0465 (7)	−0.0022 (4)	0.0223 (5)	−0.0010 (5)
O5	0.0196 (5)	0.0199 (5)	0.0402 (7)	0.0022 (4)	0.0106 (5)	−0.0068 (5)

Geometric parameters (Å, º)

C1—O1	1.4314 (19)	C11—H11A	0.9900
C1—H1A	0.9800	C11—H11B	0.9900
C1—H1B	0.9800	C12—C13	1.396 (2)
C1—H1C	0.9800	C12—C18	1.4006 (19)
C2—O1	1.3670 (17)	C13—C15	1.401 (2)
C2—C7	1.387 (2)	C13—C14	1.4814 (19)
C2—C3	1.399 (2)	C14—O2	1.2345 (17)
C3—C4	1.380 (2)	C15—C16	1.379 (2)
C3—H3	0.9500	C15—H15	0.9500
C4—C5	1.404 (2)	C16—O4	1.3703 (17)
C4—H4	0.9500	C16—C17	1.405 (2)
C5—C6	1.396 (2)	C17—O5	1.3739 (17)
C5—C8	1.460 (2)	C17—C18	1.393 (2)
C6—C7	1.382 (2)	C18—O3	1.3625 (18)
C6—H6	0.9500	C19—O4	1.429 (2)
C7—H7	0.9500	C19—H19A	0.9800
C8—C9	1.345 (2)	C19—H19B	0.9800
C8—H8	0.9500	C19—H19C	0.9800
C9—C14	1.484 (2)	C20—O5	1.4344 (19)
C9—C10	1.5089 (19)	C20—H20A	0.9800
C10—C11	1.5326 (19)	C20—H20B	0.9800
C10—H10A	0.9900	C20—H20C	0.9800
C10—H10B	0.9900	O3—H1	0.863 (19)
C11—C12	1.5079 (19)		

O1—C1—H1A	109.5	C10—C11—H11B	109.3
O1—C1—H1B	109.5	H11A—C11—H11B	108.0
H1A—C1—H1B	109.5	C13—C12—C18	117.77 (13)
O1—C1—H1C	109.5	C13—C12—C11	121.30 (12)
H1A—C1—H1C	109.5	C18—C12—C11	120.88 (13)
H1B—C1—H1C	109.5	C12—C13—C15	121.75 (13)
O1—C2—C7	124.01 (13)	C12—C13—C14	119.91 (12)
O1—C2—C3	116.18 (13)	C15—C13—C14	118.15 (13)
C7—C2—C3	119.77 (13)	O2—C14—C13	120.63 (13)

C4—C3—C2	120.17 (14)	O2—C14—C9	121.63 (13)
C4—C3—H3	119.9	C13—C14—C9	117.74 (12)
C2—C3—H3	119.9	C16—C15—C13	119.69 (14)
C3—C4—C5	120.91 (13)	C16—C15—H15	120.2
C3—C4—H4	119.5	C13—C15—H15	120.2
C5—C4—H4	119.5	O4—C16—C15	124.72 (14)
C6—C5—C4	117.68 (13)	O4—C16—C17	115.72 (13)
C6—C5—C8	117.64 (13)	C15—C16—C17	119.55 (13)
C4—C5—C8	124.46 (13)	O5—C17—C18	117.14 (13)
C7—C6—C5	121.96 (14)	O5—C17—C16	122.55 (13)
C7—C6—H6	119.0	C18—C17—C16	120.24 (13)
C5—C6—H6	119.0	O3—C18—C17	121.38 (13)
C6—C7—C2	119.49 (13)	O3—C18—C12	117.78 (13)
C6—C7—H7	120.3	C17—C18—C12	120.84 (13)
C2—C7—H7	120.3	O4—C19—H19A	109.5
C9—C8—C5	131.19 (13)	O4—C19—H19B	109.5
C9—C8—H8	114.4	H19A—C19—H19B	109.5
C5—C8—H8	114.4	O4—C19—H19C	109.5
C8—C9—C14	116.14 (13)	H19A—C19—H19C	109.5
C8—C9—C10	126.81 (13)	H19B—C19—H19C	109.5
C14—C9—C10	116.86 (12)	O5—C20—H20A	109.5
C9—C10—C11	110.23 (12)	O5—C20—H20B	109.5
C9—C10—H10A	109.6	H20A—C20—H20B	109.5
C11—C10—H10A	109.6	O5—C20—H20C	109.5
C9—C10—H10B	109.6	H20A—C20—H20C	109.5
C11—C10—H10B	109.6	H20B—C20—H20C	109.5
H10A—C10—H10B	108.1	C2—O1—C1	116.93 (12)
C12—C11—C10	111.52 (11)	C18—O3—H1	110.8 (12)
C12—C11—H11A	109.3	C16—O4—C19	116.69 (12)
C10—C11—H11A	109.3	C17—O5—C20	115.58 (11)
C12—C11—H11B	109.3		

O1—C2—C3—C4	−177.79 (13)	C8—C9—C14—O2	17.3 (2)
C7—C2—C3—C4	0.1 (2)	C10—C9—C14—O2	−167.35 (13)
C2—C3—C4—C5	0.3 (2)	C8—C9—C14—C13	−161.95 (13)
C3—C4—C5—C6	0.4 (2)	C10—C9—C14—C13	13.43 (18)
C3—C4—C5—C8	174.93 (13)	C12—C13—C15—C16	0.6 (2)
C4—C5—C6—C7	−1.4 (2)	C14—C13—C15—C16	−174.39 (13)
C8—C5—C6—C7	−176.37 (13)	C13—C15—C16—O4	−178.69 (13)
C5—C6—C7—C2	1.8 (2)	C13—C15—C16—C17	2.3 (2)
O1—C2—C7—C6	176.59 (13)	O4—C16—C17—O5	2.5 (2)
C3—C2—C7—C6	−1.1 (2)	C15—C16—C17—O5	−178.41 (13)
C6—C5—C8—C9	−149.65 (16)	O4—C16—C17—C18	179.17 (13)
C4—C5—C8—C9	35.8 (2)	C15—C16—C17—C18	−1.7 (2)
C5—C8—C9—C14	−175.38 (14)	O5—C17—C18—O3	−4.4 (2)
C5—C8—C9—C10	9.8 (3)	C16—C17—C18—O3	178.77 (13)
C8—C9—C10—C11	128.49 (15)	O5—C17—C18—C12	175.17 (13)
C14—C9—C10—C11	−46.34 (16)	C16—C17—C18—C12	−1.7 (2)

C9—C10—C11—C12	52.21 (16)	C13—C12—C18—O3	−176.06 (12)
C10—C11—C12—C13	−27.58 (18)	C11—C12—C18—O3	1.4 (2)
C10—C11—C12—C18	155.07 (13)	C13—C12—C18—C17	4.4 (2)
C18—C12—C13—C15	−3.9 (2)	C11—C12—C18—C17	−178.16 (13)
C11—C12—C13—C15	178.71 (13)	C7—C2—O1—C1	1.7 (2)
C18—C12—C13—C14	170.99 (12)	C3—C2—O1—C1	179.45 (13)
C11—C12—C13—C14	−6.4 (2)	C15—C16—O4—C19	1.3 (2)
C12—C13—C14—O2	−164.91 (14)	C17—C16—O4—C19	−179.67 (14)
C15—C13—C14—O2	10.1 (2)	C18—C17—O5—C20	113.18 (16)
C12—C13—C14—C9	14.32 (19)	C16—C17—O5—C20	−70.03 (19)
C15—C13—C14—C9	−170.65 (13)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H1···O2^{i}	0.863 (19)	2.02 (2)	2.8248 (15)	154.8 (17)
O3—H1···O5	0.863 (19)	2.289 (18)	2.7308 (15)	111.9 (15)
C6—H6···O5^{ii}	0.95	2.48	3.2668 (18)	140
C7—H7···O3^{ii}	0.95	2.59	3.4591 (18)	153

Symmetry codes: (i) −*x*+1/2, *y*+1/2, −*z*+1/2; (ii) *x*+1/2, *y*−1/2, *z*.