Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Methyl 9*H*-xanthene-9-carboxylate

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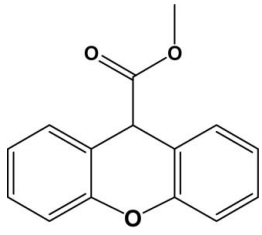
Received 20 February 2008; accepted 4 March 2008

Key indicators: single-crystal X-ray study; T = 123 K; mean σ(C–C) = 0.003 Å; R factor = 0.051; wR factor = 0.106; data-to-parameter ratio = 16.1.

The title compound, $C_{15}H_{12}O_3$, was obtained unintentionally as the by-product of an attempted recrystallization from methanol of propantheline bromide, an antimuscarinic drug. The xanthone unit is folded, with a dihedral angle of 24.81 (9)° between the benzene rings. The ester substituent adopts a *trans* staggered conformation, with a C—C—O—C torsion angle of 178.4 (1)°. The molecules pack in distinct layers, facilitated by C—H···π and weak π–π ring interactions. A weak C—H···O interaction also occurs; however, no classical hydrogen bonding is observed.

Related literature

For details of the first spectroscopic evidence of the transesterification of propantheline bromide by methanol to 9*H*-xanthene-9-carboxylic acid methyl ester, see: Avdovich *et al.* (1986). For a description of the comparative effectiveness of propantheline bromide for the treatment of neurogenic detrusor overactivity, see: George *et al.* (2007).



Experimental

Crystal data

$C_{15}H_{12}O_3$
M_r = 240.25
Monoclinic, $C2/c$
a = 25.6601 (16) Å
b = 5.7624 (3) Å
c = 15.7578 (9) Å
β = 92.933 (4)°
V = 2327.0 (2) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 123 (2) K
0.50 × 0.50 × 0.50 mm

Data collection

Bruker Kappa APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.932, T_{max} = 0.954
11906 measured reflections
2672 independent reflections
1985 reflections with $I > 2\sigma(I)$
R_{int} = 0.050

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.106
S = 1.05
2672 reflections
166 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 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
C15—H15*C*···O2^{i}	0.98	2.53	3.407 (3)	149
C3—H3···*Cg*2^{ii}	0.95	2.95	3.668 (2)	133
C11—H11···*Cg*1^{iii}	0.95	3.18	3.825 (2)	127
C15—H15*B*···*Cg*1^{iv}	0.98	3.06	3.432 (2)	104
C15—H15*C*···*Cg*1^{iv}	0.98	3.11	3.432 (2)	101

Symmetry codes: (i) $x, y-1, z$; (ii) $x, -y, z-\frac{1}{2}$; (iii) $-x+\frac{1}{2}, -y+\frac{1}{2}, -z+1$; (iv) $-x+1, -y, -z+1$. *Cg*1 is the centroid of ring C1–C6; *Cg*2 is the centroid of ring C8–C13.

Table 2
Geometrical parameters (Å, °) of the inter-ring π—π interactions.

CgI	*CgJ*	*Cg*···*Cg*	α	Symmetry position of *CgJ*
*Cg*1	*Cg*2	5.590 (1)	59.44	$x, 1-y, -\frac{1}{2}+z$
*Cg*1	*Cg*2	4.944 (1)	24.81	$\frac{1}{2}-x, \frac{1}{2}-y, 1-z$
*Cg*2	*Cg*1	4.863 (1)	59.44	$x, -y, \frac{1}{2}+z$
*Cg*2	*Cg*2	3.684 (1)	0.03	$\frac{1}{2}-x, \frac{1}{2}-y, 1-z$

α is the dihedral angle between planes *I* and *J*, *CgI* is the centroid of plane *I* and *CgJ* the centroid of plane *J*. *Cg*1 is the centroid of ring C1–C6; *Cg*2 is the centroid of ring C8–C13.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *POV-RAY for Windows* (Persistence of Vision, 1999); software used to prepare material for publication: *PLATON* (Spek, 2003).

PMD is grateful to Monash University for the Monash Graduate Scholarship and Monash International Postgraduate Research Scholarship, and to Monash University, School of Chemistry for funding for JT.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WN2242).

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supporting information

Acta Cryst. (2008). E**64**, o854 [doi:10.1107/S1600536808005990]

Methyl 9*H*-xanthene-9-carboxylate

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S1. Comment

It was found that propantheline bromide (George *et al.*, 2007) undergoes facile transesterification by methanol to produce the by-product 9*H*-xanthene-9-carboxylic acid methyl ester (Avdovich *et al.*, 1986). Surprisingly, the structural elucidation of this analogue (Fig. 1) has not been reported in the literature until now. Now the structural determination and analysis is briefly described.

The xanthone unit is bent, with the aromatic planes oriented to each other by an interplanar angle of 24.81 (9)°. The ester substituent adopts a *trans* staggered conformation with a C7—C14—O3—C15 torsion angle of 178.4 (1)°. Additionally, as is typical of an ester, the O3—C14 distance is 1.326 (2) Å and the O3—C15 distance is 1.448 (2) Å, indicating the sp^2 hybridization of C14.

The overall packing is shown in Fig. 2. Molecules are related by centres of symmetry, resulting in a head-to-head arrangement, that packs in aromatic and non-aromatic layers lying parallel to the (100) plane. Fig. 2 displays the orientation of the molecules, facilitating the weak C—H···O hydrogen bonding between the methyl and carbonyl groups (distance: C15—H15C···O2^{i} (i = x,y - 1,z) 3.407 (2) Å - see Table 1) and the C—H···π and weak π···π ring interactions (Table 2). A short range contact, 2.683 (2) Å, also occurs between the aromatic C4—H4 and the carbonyl oxygen O2 (distance: C4—H4···O2^{ii} (ii = x,1 - y,-1/2 + z).

S2. Experimental

The title compound was obtained unintentionally as the product of an attempted recrystallization of propantheline bromide (50 mg) in methanol (2 ml) at room temperature. Crystals resulted after 6 days; these were coated with Paratone N oil (Exxon Chemical Co., TX, USA) immediately after isolation and cooled in a stream of nitrogen vapour on the diffractometer. Melting point: 360.7 K.

S3. Refinement

All H atoms were observed in difference syntheses and were then placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H distances in the range 0.95–1.00 Å. U_{iso}(H) = xU_{eq}(C), where x = 1.5 for methyl and 1.2 for all other C atoms.

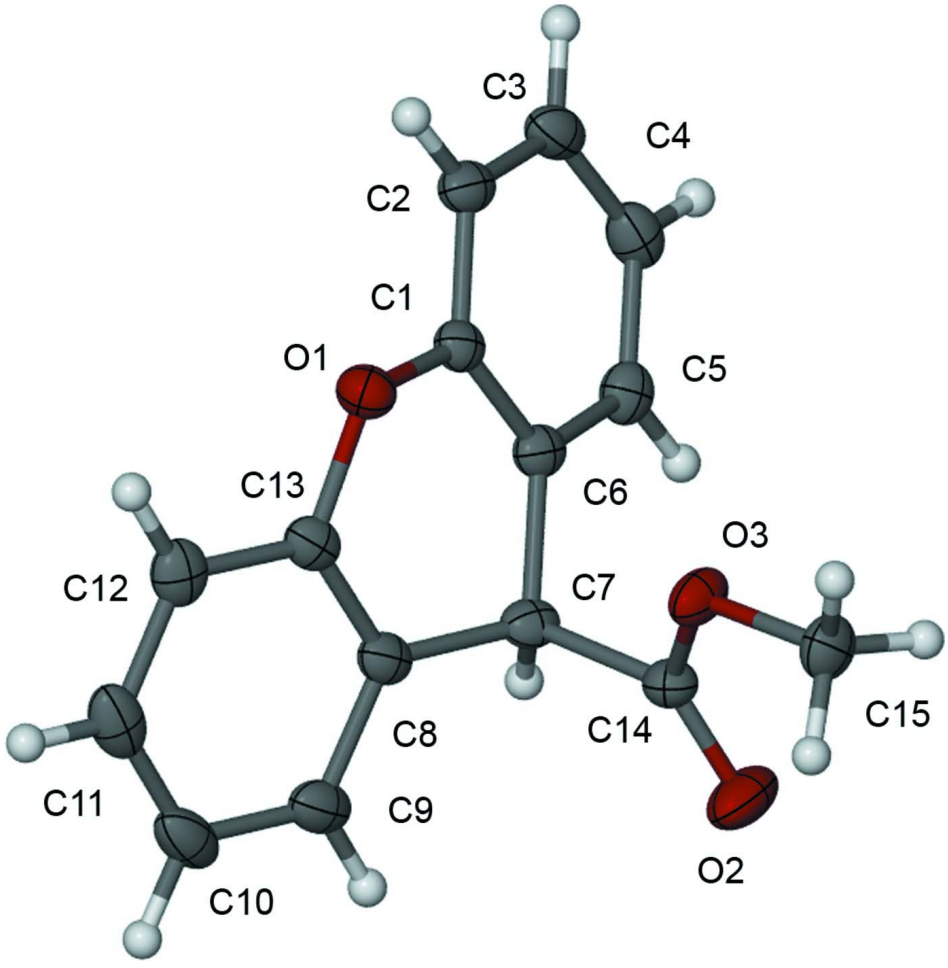

Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level and hydrogen atoms as spheres of arbitrary radius.

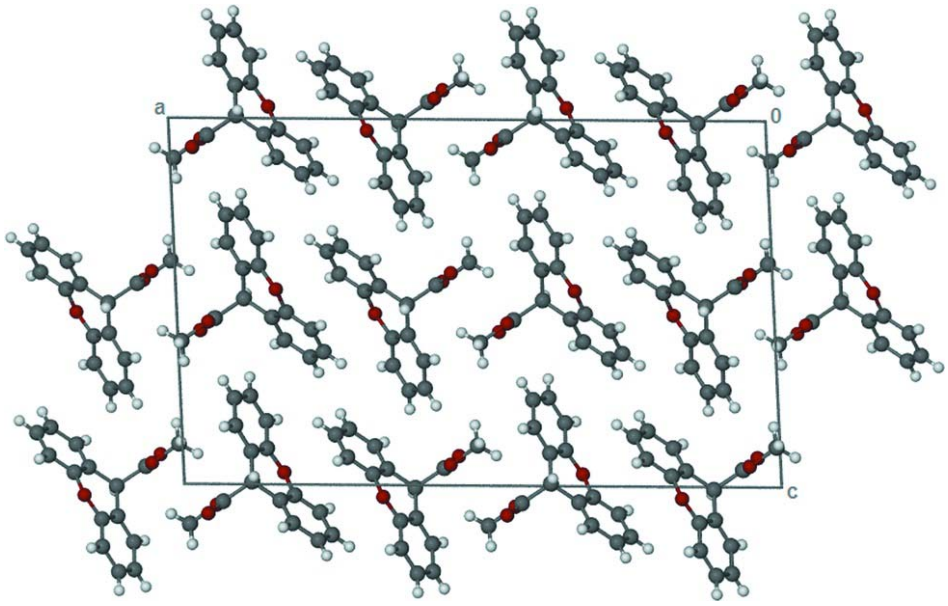

Figure 2

A ball-and-stick representation of the unit-cell contents, viewed down the *b* axis.

Methyl 9*H*-xanthene-9-carboxylate

Crystal data

$C_{15}H_{12}O_3$
$M_r = 240.25$
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
$a = 25.6601\ (16)$ Å
$b = 5.7624\ (3)$ Å
$c = 15.7578\ (9)$ Å
$\beta = 92.933\ (4)°$
$V = 2327.0\ (2)$ Å^3
$Z = 8$
$F(000) = 1008$
$D_x = 1.372$ Mg m^{-3}
Melting point: 360.7 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1829 reflections
$\theta = 2.6–25.8°$
$\mu = 0.10$ mm^{-1}
$T = 123$ K
Prismatic, colourless
0.50 × 0.50 × 0.50 mm

Data collection

Bruker KappaAPEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
0.5° frames in φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.932$, $T_{max} = 0.954$
11906 measured reflections
2672 independent reflections
1985 reflections with $I > 2\sigma(I)$
$R_{int} = 0.050$
$\theta_{max} = 27.5°$, $\theta_{min} = 1.6°$
$h = -33\rightarrow33$
$k = -7\rightarrow7$
$l = -20\rightarrow20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.106$
$S = 1.06$
2672 reflections
166 parameters
0 restraints
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

$w = 1/[\sigma^2(F_o^2) + (0.0212P)^2 + 2.7981P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.33199 (5)	−0.0433 (2)	0.46758 (8)	0.0291 (4)
O3	0.44741 (5)	0.1331 (2)	0.55663 (8)	0.0317 (4)
C1	0.35919 (6)	0.0509 (3)	0.40209 (11)	0.0246 (4)
O2	0.46182 (5)	0.5104 (2)	0.57999 (10)	0.0460 (5)
C5	0.41061 (7)	0.3418 (3)	0.33969 (11)	0.0296 (5)
H5	0.4285	0.4860	0.3432	0.036*
C8	0.34065 (6)	0.3209 (3)	0.54356 (11)	0.0270 (5)
C14	0.43644 (7)	0.3568 (3)	0.54706 (11)	0.0269 (4)
C9	0.32151 (7)	0.4651 (4)	0.60572 (11)	0.0340 (5)
H9	0.3374	0.6117	0.6166	0.041*
C4	0.40994 (7)	0.2167 (4)	0.26464 (12)	0.0344 (5)
H4	0.4275	0.2744	0.2174	0.041*
C2	0.35812 (7)	−0.0765 (3)	0.32741 (11)	0.0296 (5)
H2	0.3400	−0.2202	0.3236	0.036*
C12	0.27490 (7)	0.0372 (4)	0.57561 (11)	0.0320 (5)
H12	0.2591	−0.1099	0.5653	0.038*
C10	0.27969 (7)	0.3977 (4)	0.65182 (12)	0.0404 (6)
H10	0.2668	0.4983	0.6936	0.048*
C11	0.25674 (7)	0.1837 (4)	0.63695 (12)	0.0383 (6)
H11	0.2282	0.1369	0.6691	0.046*
C3	0.38359 (7)	0.0071 (4)	0.25876 (12)	0.0336 (5)
H3	0.3831	−0.0792	0.2074	0.040*
C6	0.38552 (6)	0.2600 (3)	0.41010 (11)	0.0255 (4)
C7	0.38629 (7)	0.3930 (3)	0.49288 (11)	0.0265 (5)
H7	0.3828	0.5622	0.4796	0.032*
C13	0.31655 (6)	0.1086 (3)	0.52950 (11)	0.0266 (4)
C15	0.49380 (7)	0.0797 (4)	0.60921 (12)	0.0349 (5)
H15A	0.4911	0.1505	0.6654	0.052*
H15B	0.5245	0.1415	0.5825	0.052*
H15C	0.4972	−0.0889	0.6154	0.052*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0329 (7)	0.0252 (7)	0.0294 (7)	0.0015 (5)	0.0027 (5)	−0.0008 (5)
O3	0.0293 (7)	0.0257 (7)	0.0385 (8)	0.0029 (6)	−0.0121 (5)	−0.0011 (6)
C1	0.0234 (8)	0.0252 (10)	0.0251 (9)	0.0051 (7)	−0.0011 (7)	−0.0002 (7)
O2	0.0419 (8)	0.0328 (8)	0.0611 (10)	−0.0017 (7)	−0.0166 (7)	−0.0116 (7)
C5	0.0264 (9)	0.0298 (10)	0.0324 (10)	0.0028 (8)	−0.0027 (7)	0.0057 (8)
C8	0.0260 (9)	0.0296 (10)	0.0248 (9)	0.0088 (7)	−0.0044 (7)	−0.0016 (7)
C14	0.0281 (9)	0.0249 (10)	0.0276 (9)	0.0000 (8)	0.0010 (7)	−0.0043 (8)
C9	0.0329 (10)	0.0396 (12)	0.0284 (10)	0.0133 (9)	−0.0089 (8)	−0.0074 (8)
C4	0.0309 (10)	0.0457 (13)	0.0267 (10)	0.0086 (9)	0.0019 (7)	0.0050 (9)
C2	0.0288 (9)	0.0280 (10)	0.0314 (10)	0.0060 (8)	−0.0048 (7)	−0.0042 (8)
C12	0.0286 (9)	0.0384 (11)	0.0285 (10)	0.0052 (8)	−0.0038 (7)	0.0087 (8)
C10	0.0360 (10)	0.0603 (15)	0.0245 (10)	0.0203 (10)	−0.0028 (8)	−0.0072 (9)
C11	0.0298 (10)	0.0604 (15)	0.0246 (10)	0.0124 (10)	0.0005 (7)	0.0091 (9)
C3	0.0321 (10)	0.0426 (12)	0.0254 (10)	0.0111 (9)	−0.0044 (7)	−0.0061 (8)
C6	0.0248 (8)	0.0244 (10)	0.0267 (9)	0.0050 (7)	−0.0040 (7)	0.0002 (7)
C7	0.0310 (9)	0.0204 (9)	0.0277 (9)	0.0051 (7)	−0.0037 (7)	−0.0015 (7)
C13	0.0279 (9)	0.0303 (10)	0.0210 (9)	0.0082 (8)	−0.0030 (7)	0.0017 (7)
C15	0.0261 (9)	0.0417 (12)	0.0361 (11)	0.0036 (8)	−0.0071 (8)	0.0010 (9)

Geometric parameters (Å, º)

O1—C13	1.384 (2)	C4—C3	1.385 (3)
O1—C1	1.386 (2)	C4—H4	0.9500
O3—C14	1.326 (2)	C2—C3	1.379 (3)
O3—C15	1.448 (2)	C2—H2	0.9500
C1—C6	1.384 (2)	C12—C11	1.382 (3)
C1—C2	1.386 (2)	C12—C13	1.385 (2)
O2—C14	1.201 (2)	C12—H12	0.9500
C5—C4	1.384 (3)	C10—C11	1.381 (3)
C5—C6	1.393 (2)	C10—H10	0.9500
C5—H5	0.9500	C11—H11	0.9500
C8—C13	1.384 (3)	C3—H3	0.9500
C8—C9	1.393 (2)	C6—C7	1.512 (2)
C8—C7	1.509 (2)	C7—H7	1.0000
C14—C7	1.522 (2)	C15—H15A	0.9800
C9—C10	1.382 (3)	C15—H15B	0.9800
C9—H9	0.9500	C15—H15C	0.9800
C13—O1—C1	116.79 (14)	C11—C10—H10	120.1
C14—O3—C15	115.78 (14)	C9—C10—H10	120.1
C6—C1—O1	122.37 (15)	C10—C11—C12	120.52 (18)
C6—C1—C2	121.80 (16)	C10—C11—H11	119.7
O1—C1—C2	115.83 (16)	C12—C11—H11	119.7
C4—C5—C6	121.21 (18)	C2—C3—C4	120.11 (17)
C4—C5—H5	119.4	C2—C3—H3	119.9

C6—C5—H5	119.4	C4—C3—H3	119.9
C13—C8—C9	117.98 (17)	C1—C6—C5	117.73 (16)
C13—C8—C7	120.76 (15)	C1—C6—C7	120.31 (16)
C9—C8—C7	121.26 (17)	C5—C6—C7	121.95 (16)
O2—C14—O3	124.06 (17)	C8—C7—C6	109.91 (15)
O2—C14—C7	124.42 (16)	C8—C7—C14	108.79 (14)
O3—C14—C7	111.45 (15)	C6—C7—C14	112.80 (14)
C10—C9—C8	120.9 (2)	C8—C7—H7	108.4
C10—C9—H9	119.6	C6—C7—H7	108.4
C8—C9—H9	119.6	C14—C7—H7	108.4
C5—C4—C3	119.72 (18)	C8—C13—O1	122.03 (15)
C5—C4—H4	120.1	C8—C13—C12	121.96 (17)
C3—C4—H4	120.1	O1—C13—C12	116.00 (17)
C3—C2—C1	119.42 (18)	O3—C15—H15A	109.5
C3—C2—H2	120.3	O3—C15—H15B	109.5
C1—C2—H2	120.3	H15A—C15—H15B	109.5
C11—C12—C13	118.81 (19)	O3—C15—H15C	109.5
C11—C12—H12	120.6	H15A—C15—H15C	109.5
C13—C12—H12	120.6	H15B—C15—H15C	109.5
C11—C10—C9	119.83 (18)		

C13—O1—C1—C6	21.8 (2)	C13—C8—C7—C6	22.5 (2)
C13—O1—C1—C2	−157.71 (15)	C9—C8—C7—C6	−157.52 (16)
C15—O3—C14—O2	−1.3 (3)	C13—C8—C7—C14	−101.48 (18)
C15—O3—C14—C7	−178.43 (14)	C9—C8—C7—C14	78.5 (2)
C13—C8—C9—C10	0.0 (3)	C1—C6—C7—C8	−22.1 (2)
C7—C8—C9—C10	179.99 (16)	C5—C6—C7—C8	157.52 (16)
C6—C5—C4—C3	0.5 (3)	C1—C6—C7—C14	99.48 (19)
C6—C1—C2—C3	−0.5 (3)	C5—C6—C7—C14	−80.9 (2)
O1—C1—C2—C3	179.03 (15)	O2—C14—C7—C8	−105.6 (2)
C8—C9—C10—C11	0.6 (3)	O3—C14—C7—C8	71.48 (18)
C9—C10—C11—C12	−0.7 (3)	O2—C14—C7—C6	132.14 (19)
C13—C12—C11—C10	0.1 (3)	O3—C14—C7—C6	−50.7 (2)
C1—C2—C3—C4	0.0 (3)	C9—C8—C13—O1	178.26 (15)
C5—C4—C3—C2	0.0 (3)	C7—C8—C13—O1	−1.7 (2)
O1—C1—C6—C5	−178.53 (15)	C9—C8—C13—C12	−0.6 (3)
C2—C1—C6—C5	1.0 (2)	C7—C8—C13—C12	179.41 (16)
O1—C1—C6—C7	1.1 (2)	C1—O1—C13—C8	−21.5 (2)
C2—C1—C6—C7	−179.37 (15)	C1—O1—C13—C12	157.43 (15)
C4—C5—C6—C1	−1.0 (2)	C11—C12—C13—C8	0.5 (3)
C4—C5—C6—C7	179.39 (16)	C11—C12—C13—O1	−178.37 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C15—H15*C*···O2^{i}	0.98	2.53	3.407 (3)	149
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C11—H11···*Cg*1^{iii}	0.95	3.18	3.825 (2)	127

C15—H15*B*···*Cg*1^{iv}	0.98	3.06	3.432 (2)	104
C15—H15*C*···*Cg*1^{iv}	0.98	3.11	3.432 (2)	101

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