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Crystal structure of 4-methyl-7-propoxy-2*H*-chromen-2-one

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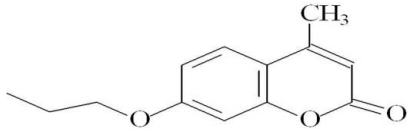
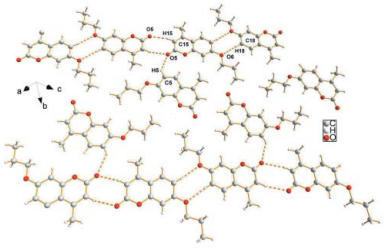
The asymmetric unit of the title compound, $C_{13}H_{14}O_3$, contains two independent molecules, *A* and *B*, that are interconnected through an offset π–π interaction [inter-centroid separation = 3.6087 (4) Å]. The fused benzene and pyran-2-one rings in each molecule are essentially coplanar, having dihedral angles of 1.22 (12) and 1.57 (12)° for molecules *A* and *B*, respectively. Similarly, the coumarin ring system and the 7-propoxy substituent are close to being coplanar [C—C—O—C torsion angles = 2.9 (2) and 1.4 (2)° for molecules *A* and *B*, respectively]. In the crystal, the molecules are connected by C—H···O hydrogen bonds, forming supramolecular tapes along [100] that are linked into a three-dimensional network by C—H···π interactions, as well as by the aforementioned π–π interactions.

1. Chemical context

Coumarin (2*H*-1-benzopyran-2-one) is a plant-derived natural product known for its pharmacological properties such as anti-inflammatory, anticoagulant, antibacterial, antifungal, antiviral, anticancer, antihypertensive, antitubercular, anticonvulsant, anti-adipogenic, antihyperglycemic, anti-oxidant and neuroprotective properties. Dietary exposure to benzopyrones is significant as these compounds are found in vegetables, fruits, seeds, nuts, coffee, tea and wine (Venugopala *et al.*, 2013). In order to assist our knowledge about the stereoelectronic requirements from these kinds of molecules to show anti-asthmatic or tracheal relaxant actions, we have synthesized (Sánchez-Recillas *et al.*, 2014) and determined the crystal structure of the title compound, (I). A related structure, 3-acetylcoumarin, has been reported on by Munshi *et al.* (2004).





2. Structural commentary

The asymmetric unit of (I) contains two independent molecules (*A* and *B*). Bond lengths between equivalent non-H atoms of each molecule are similar, with differences less than 3 s.u.

The fused aryl and pyran-2-one rings in each molecule are individually planar (r.m.s. deviations < 0.0064 for aryl rings and < 0.0141 Å for pyran-2-one rings) and form single planar

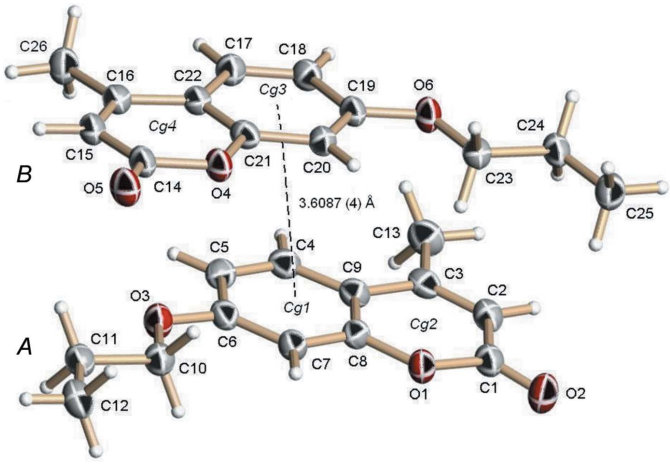


Figure 1
The molecular structure of (I), showing the atom-labelling scheme and the offset π–π interaction. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radius. The dashed line indicates the interaction between the benzene ring centroids *Cg*1 (C4—C9) and *Cg2* (C17—C22).

units [r.m.s. deviation = 0.0159 Å, dihedral angle between the two six-membered rings of 1.22 (12)° in molecule *A*; r.m.s. deviation = 0.0192 Å, dihedral angle between the two six-membered rings of 1.57 (12)° for *B*].

The torsion angles between the coumarins ring systems and the 7-propoxy substituents in *A* (C7—C6—O3—C10) and *B* (C20—C19—O6—C23) are 2.9 (2) and 1.4 (2)°, respectively. The two independent molecules are interconnected through an offset π–π interaction, with a distance between the centroids of the C4–C9 and C17–C22 benzene rings of 3.6087 (4) Å.

3. Supramolecular features

The packing is mainly through C—H···O and C—H···π hydrogen bonding (Table 1) as well as the π–π interaction mentioned above. Three *B* molecules are connected through two pairs of C—H···O hydrogen bonds, generating two

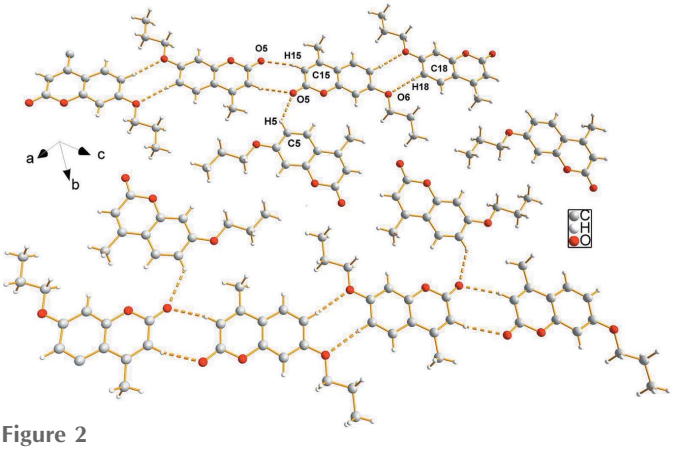


Figure 2
View of the supramolecular tape structure along [100] sustained by C—H···O hydrogen bonds (dashed lines).

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

*Cg*2, *Cg*3 and *Cg*4 are the centroids of the O1/C1–C3/C8/C9, C17–C22 and O4/C14–C16/C21/C22 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···O5^{i}	0.95	2.57	3.296 (2)	133
C15—H15···O5^{ii}	0.95	2.49	3.422 (2)	165
C18—H18···O6^{iii}	0.95	2.45	3.402 (2)	175
C11—H11*A*···*Cg*2^{iv}	0.99	2.76	3.6087 (4)	140
C24—H24*A*···*Cg*4^{v}	0.99	2.81	3.613 (2)	139
C23—H23*B*···*Cg*3^{v}	0.99	2.75	3.614 (2)	145

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

centrosymmetric $R_2^2(8)$ graph sets; the first involving atoms $(\cdots$H15–C15–C14–O5$\cdots)_2$ and the second involving atoms $(\cdots$H18–C18–C19–O6$\cdots)_2$. The $R_2^2(8)$ motifs are connected with *A* molecules through C—H···O contacts, generating a tape-like structure along [100] (Fig. 2). Additional, C—H···π interactions provide the links between neighboring tapes, resulting in a three-dimensional network (Fig. 3).

4. Synthesis and crystallization

The title compound was prepared by *SN*2 reaction between 7-hydroxy-4-methyl-2*H*-chromen-2-one and *n*-propyl bromide. 7-Hydroxy-4-methyl-2*H*-chromen-2-one (0.4 g, 2.27 mmol) and potassium carbonate (1.28 g, 9.30 mmol, 4.1 equiv) were dissolved in acetone (2.0 ml) and kept at room temperature. After 20 minutes, *n*-propylbromide (0.641 ml, 7.03 mmol) was added drop wise and the reaction mixture was heated to reflux (313 K) and monitored by TLC. After completion of the reaction (six days), the reaction mixture was filtered and the solid residue was washed off with cold water (10 ml). The total mother liquors were concentrated under reduced pressure and then poured into water and extracted with ethyl acetate (3 ×

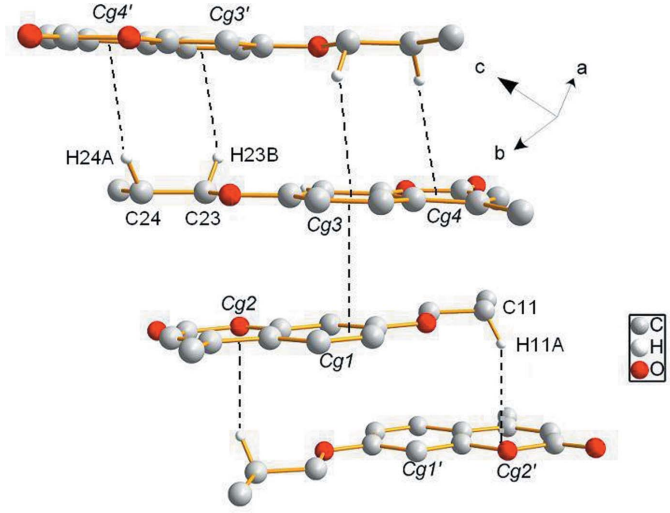


Figure 3
View of the C—H···π hydrogen bonds (dashed lines) between neighbouring tapes. *Cg*2′, *Cg*3′ [symmetry code: (′) $-x+1, -y+2, -z$] and *Cg*4′ [symmetry code: (′) $-x+1, -y+1, -z+1$].

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_{14}O_3$
M_r	218.24
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	100
a, b, c (Å)	7.2418 (9), 11.5459 (14), 14.5301 (17)
α, β, γ (°)	69.014 (2), 76.086 (2), 84.124 (2)
V (Å^3)	1100.9 (2)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.25 × 0.25 × 0.21
Data collection	
Diffractometer	Bruker *SMART APEX* CCD area detector
Absorption correction	Multi-scan (*SADABS*; Sheldrick, 2003)
T_{min}, T_{max}	0.977, 0.981
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	9469, 3872, 3420
R_{int}	0.030
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.047, 0.122, 1.12
No. of reflections	3872
No. of parameters	293
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.22, −0.25

Computer programs: *SMART* (Bruker, 2000), *SAINT-Plus* (Bruker, 2001), *DIAMOND* (Brandenburg, 1997), *SHELXTL-NT* (Sheldrick, 2008) and *publCIF* (Westrip, 2010).

15 ml). The organic layer was dried over anhydrous Na_2SO_4 and evaporated under reduced pressure to give a white-coloured solid (m.p. 347.55–348.35 K). Single crystals were obtained from methanol. ^{1}H NMR data (400 MHz; $CDCl_3$: Me_4Si) *d*: 1.02 (3*H*, *t*, CH_3), 1.82 (2*H*, *m*, CH_2), 2.36 (3*H*, *s*, CH_3), 3.94 (2*H*, *t*, CH_2-O), 6.08 (*s*, 1H, H-3), 6.76 (1*H*, *d* H, *J* =2.4 Hz, H-8), 6.82 (1*H*, *dd*, CH, *J* = 8.8 Hz, *J* = 2.4 Hz, H-6), 7.45 (1*H*, *d*, CH, *J* = 8.8 Hz, H-5).

4.1. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. H atoms were positioned geometrically and constrained using the riding-model approximation [$C-H_{aryl}$ = 0.95 Å, $U_{iso}(H_{aryl})$ = 1.2 $U_{eq}(C)$; $C-H_{methylene}$ = 0.99 Å, $U_{iso}(H_{methylene})$ = 1.2 $U_{eq}(C)$; $C-H_{methyl}$ = 0.98 Å, $U_{iso}(H_{methyl})$ = 1.5 $U_{eq}(C)$].

Acknowledgements

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Crystal structure of 4-methyl-7-propoxy-2*H*-chromen-2-one

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Computing details

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus* (Bruker, 2001); program(s) used to solve structure: *SHELXTL-NT* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL-NT* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 1997); software used to prepare material for publication: *SHELXTL-NT* (Sheldrick, 2008) and *publCIF* (Westrip, 2010).

4-Methyl-7-propoxy-2*H*-chromen-2-one

Crystal data

$C_{13}H_{14}O_3$
$M_r = 218.24$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.2418\ (9)$ Å
$b = 11.5459\ (14)$ Å
$c = 14.5301\ (17)$ Å
$\alpha = 69.014\ (2)°$
$\beta = 76.086\ (2)°$
$\gamma = 84.124\ (2)°$
$V = 1100.9\ (2)$ Å^3
$Z = 4$
$F(000) = 464$
$D_x = 1.317$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7718 reflections
$\theta = 2.8–28.2°$
$\mu = 0.09$ mm^{-1}
$T = 100$ K
Block, colourless
0.25 × 0.25 × 0.21 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.3 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.977$, $T_{max} = 0.981$
9469 measured reflections
3872 independent reflections
3420 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 25.0°$, $\theta_{min} = 1.5°$
$h = -8\rightarrow8$
$k = -13\rightarrow13$
$l = -17\rightarrow17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.122$
$S = 1.12$
3872 reflections
293 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
$w = 1/[\sigma^2(F_o^2) + (0.0454P)^2 + 0.5831P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ 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}
C1	0.2312 (2)	1.02587 (16)	0.34492 (13)	0.0272 (4)
C2	0.0602 (2)	0.95556 (16)	0.37805 (13)	0.0274 (4)
H2	−0.0354	0.9666	0.4320	0.033*
C3	0.0295 (2)	0.87428 (16)	0.33576 (13)	0.0269 (4)
C4	0.1609 (3)	0.77466 (16)	0.20345 (13)	0.0269 (4)
H4	0.0473	0.7297	0.2212	0.032*
C5	0.3071 (2)	0.75950 (16)	0.12847 (13)	0.0265 (4)
H5	0.2937	0.7048	0.0948	0.032*
C6	0.4758 (2)	0.82451 (15)	0.10153 (12)	0.0253 (4)
C7	0.4952 (2)	0.90553 (15)	0.14979 (12)	0.0248 (4)
H7	0.6091	0.9504	0.1317	0.030*
C8	0.3444 (2)	0.91946 (15)	0.22501 (12)	0.0235 (4)
C9	0.1753 (2)	0.85505 (15)	0.25466 (12)	0.0240 (4)
C10	0.7927 (2)	0.86097 (16)	−0.00035 (13)	0.0281 (4)
H10A	0.8528	0.8348	0.0586	0.034*
H10B	0.7742	0.9522	−0.0230	0.034*
C11	0.9176 (3)	0.82336 (17)	−0.08476 (13)	0.0295 (4)
H11A	0.8568	0.8502	−0.1434	0.035*
H11B	0.9329	0.7319	−0.0620	0.035*
C12	1.1119 (3)	0.88242 (19)	−0.11598 (14)	0.0347 (4)
H12A	1.0966	0.9729	−0.1394	0.052*
H12B	1.1919	0.8573	−0.1707	0.052*
H12C	1.1724	0.8550	−0.0580	0.052*
C13	−0.1524 (3)	0.80431 (18)	0.37212 (14)	0.0346 (4)
H13A	−0.2305	0.8233	0.4303	0.052*
H13B	−0.1237	0.7151	0.3921	0.052*
H13C	−0.2219	0.8288	0.3177	0.052*
C14	0.8545 (2)	0.54134 (16)	0.14392 (12)	0.0250 (4)
C15	0.7477 (2)	0.46641 (15)	0.11512 (12)	0.0253 (4)
H15	0.8064	0.4400	0.0599	0.030*
C16	0.5668 (2)	0.43214 (15)	0.16371 (12)	0.0252 (4)
C17	0.2919 (2)	0.44277 (16)	0.30600 (13)	0.0255 (4)
H17	0.2194	0.3891	0.2924	0.031*
C18	0.2136 (2)	0.48829 (15)	0.38246 (13)	0.0254 (4)
H18	0.0881	0.4665	0.4208	0.030*

C19	0.3192 (2)	0.56707 (15)	0.40370 (12)	0.0232 (4)
C20	0.5028 (2)	0.59811 (15)	0.34929 (12)	0.0230 (4)
H20	0.5755	0.6507	0.3639	0.028*
C21	0.5781 (2)	0.55004 (15)	0.27239 (12)	0.0224 (4)
C22	0.4766 (2)	0.47361 (14)	0.24749 (12)	0.0223 (4)
C23	0.3305 (2)	0.69312 (16)	0.50284 (13)	0.0250 (4)
H23A	0.3647	0.7685	0.4426	0.030*
H23B	0.4491	0.6526	0.5226	0.030*
C24	0.2037 (3)	0.72763 (16)	0.58868 (13)	0.0280 (4)
H24A	0.1709	0.6519	0.6489	0.034*
H24B	0.0841	0.7664	0.5690	0.034*
C25	0.3047 (3)	0.81783 (18)	0.61425 (14)	0.0357 (4)
H25A	0.4244	0.7799	0.6322	0.054*
H25B	0.2232	0.8373	0.6714	0.054*
H25C	0.3315	0.8943	0.5555	0.054*
C26	0.4575 (3)	0.35469 (17)	0.13210 (14)	0.0324 (4)
H26A	0.5353	0.3388	0.0725	0.049*
H26B	0.3402	0.3991	0.1158	0.049*
H26C	0.4255	0.2757	0.1874	0.049*
O1	0.37241 (17)	1.00201 (11)	0.26973 (9)	0.0264 (3)
O2	0.26525 (19)	1.10374 (12)	0.37645 (10)	0.0359 (3)
O3	0.61273 (17)	0.80147 (11)	0.02699 (9)	0.0291 (3)
O4	0.76161 (16)	0.58432 (11)	0.22148 (8)	0.0243 (3)
O5	1.01862 (17)	0.57246 (12)	0.10667 (9)	0.0315 (3)
O6	0.22868 (16)	0.60943 (11)	0.48005 (9)	0.0266 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0281 (9)	0.0300 (9)	0.0229 (8)	0.0023 (7)	−0.0038 (7)	−0.0103 (7)
C2	0.0273 (9)	0.0319 (9)	0.0217 (8)	0.0009 (7)	−0.0034 (7)	−0.0093 (7)
C3	0.0267 (9)	0.0288 (9)	0.0214 (8)	0.0006 (7)	−0.0061 (7)	−0.0039 (7)
C4	0.0294 (9)	0.0264 (9)	0.0245 (9)	−0.0029 (7)	−0.0089 (7)	−0.0058 (7)
C5	0.0315 (9)	0.0253 (8)	0.0250 (9)	−0.0007 (7)	−0.0107 (7)	−0.0086 (7)
C6	0.0286 (9)	0.0270 (9)	0.0192 (8)	0.0025 (7)	−0.0052 (7)	−0.0074 (7)
C7	0.0263 (9)	0.0271 (9)	0.0211 (8)	−0.0010 (7)	−0.0046 (7)	−0.0088 (7)
C8	0.0286 (9)	0.0234 (8)	0.0203 (8)	0.0007 (7)	−0.0083 (7)	−0.0080 (7)
C9	0.0252 (8)	0.0248 (8)	0.0209 (8)	−0.0009 (7)	−0.0070 (7)	−0.0052 (7)
C10	0.0288 (9)	0.0300 (9)	0.0256 (9)	0.0003 (7)	−0.0048 (7)	−0.0108 (7)
C11	0.0329 (10)	0.0323 (9)	0.0222 (9)	0.0026 (7)	−0.0047 (7)	−0.0100 (7)
C12	0.0331 (10)	0.0464 (11)	0.0256 (9)	0.0030 (8)	−0.0063 (8)	−0.0147 (8)
C13	0.0295 (10)	0.0400 (11)	0.0313 (10)	−0.0058 (8)	−0.0032 (8)	−0.0095 (8)
C14	0.0265 (9)	0.0283 (9)	0.0181 (8)	0.0021 (7)	−0.0005 (7)	−0.0089 (7)
C15	0.0284 (9)	0.0276 (9)	0.0201 (8)	0.0010 (7)	−0.0023 (7)	−0.0106 (7)
C16	0.0285 (9)	0.0250 (8)	0.0210 (8)	0.0001 (7)	−0.0038 (7)	−0.0079 (7)
C17	0.0252 (9)	0.0266 (8)	0.0251 (9)	−0.0019 (7)	−0.0040 (7)	−0.0101 (7)
C18	0.0232 (8)	0.0270 (9)	0.0236 (8)	−0.0028 (7)	0.0002 (7)	−0.0088 (7)
C19	0.0242 (8)	0.0253 (8)	0.0180 (8)	0.0017 (6)	−0.0002 (6)	−0.0086 (6)

C20	0.0241 (8)	0.0239 (8)	0.0206 (8)	−0.0009 (7)	−0.0022 (7)	−0.0087 (7)
C21	0.0197 (8)	0.0237 (8)	0.0193 (8)	0.0001 (6)	−0.0006 (6)	−0.0047 (6)
C22	0.0234 (8)	0.0224 (8)	0.0201 (8)	0.0002 (6)	−0.0029 (7)	−0.0076 (7)
C23	0.0251 (8)	0.0279 (9)	0.0229 (8)	−0.0001 (7)	−0.0038 (7)	−0.0108 (7)
C24	0.0298 (9)	0.0329 (9)	0.0212 (8)	0.0052 (7)	−0.0038 (7)	−0.0120 (7)
C25	0.0441 (11)	0.0377 (10)	0.0306 (10)	0.0074 (9)	−0.0113 (8)	−0.0182 (8)
C26	0.0341 (10)	0.0368 (10)	0.0293 (10)	−0.0038 (8)	−0.0030 (8)	−0.0169 (8)
O1	0.0283 (6)	0.0305 (6)	0.0229 (6)	−0.0031 (5)	−0.0023 (5)	−0.0136 (5)
O2	0.0382 (7)	0.0401 (7)	0.0359 (7)	−0.0036 (6)	−0.0031 (6)	−0.0232 (6)
O3	0.0304 (7)	0.0333 (7)	0.0250 (6)	−0.0018 (5)	−0.0018 (5)	−0.0142 (5)
O4	0.0210 (6)	0.0302 (6)	0.0208 (6)	−0.0024 (5)	0.0024 (5)	−0.0118 (5)
O5	0.0252 (7)	0.0418 (7)	0.0267 (7)	−0.0050 (5)	0.0036 (5)	−0.0158 (6)
O6	0.0242 (6)	0.0336 (7)	0.0238 (6)	−0.0030 (5)	0.0018 (5)	−0.0158 (5)

Geometric parameters (Å, º)

C1—O2	1.214 (2)	C14—O5	1.214 (2)
C1—O1	1.391 (2)	C14—O4	1.3916 (19)
C1—C2	1.437 (2)	C14—C15	1.440 (2)
C2—C3	1.353 (3)	C15—C16	1.353 (2)
C2—H2	0.9500	C15—H15	0.9500
C3—C9	1.447 (2)	C16—C22	1.452 (2)
C3—C13	1.498 (2)	C16—C26	1.501 (2)
C4—C5	1.372 (2)	C17—C18	1.373 (2)
C4—C9	1.406 (2)	C17—C22	1.405 (2)
C4—H4	0.9500	C17—H17	0.9500
C5—C6	1.399 (2)	C18—C19	1.402 (2)
C5—H5	0.9500	C18—H18	0.9500
C6—O3	1.365 (2)	C19—O6	1.3685 (19)
C6—C7	1.390 (2)	C19—C20	1.382 (2)
C7—C8	1.389 (2)	C20—C21	1.394 (2)
C7—H7	0.9500	C20—H20	0.9500
C8—O1	1.385 (2)	C21—O4	1.3769 (19)
C8—C9	1.394 (2)	C21—C22	1.392 (2)
C10—O3	1.438 (2)	C23—O6	1.442 (2)
C10—C11	1.510 (2)	C23—C24	1.513 (2)
C10—H10A	0.9900	C23—H23A	0.9900
C10—H10B	0.9900	C23—H23B	0.9900
C11—C12	1.523 (3)	C24—C25	1.524 (3)
C11—H11A	0.9900	C24—H24A	0.9900
C11—H11B	0.9900	C24—H24B	0.9900
C12—H12A	0.9800	C25—H25A	0.9800
C12—H12B	0.9800	C25—H25B	0.9800
C12—H12C	0.9800	C25—H25C	0.9800
C13—H13A	0.9800	C26—H26A	0.9800
C13—H13B	0.9800	C26—H26B	0.9800
C13—H13C	0.9800	C26—H26C	0.9800

O2—C1—O1	116.67 (16)	O4—C14—C15	117.51 (14)
O2—C1—C2	126.41 (16)	C16—C15—C14	122.54 (15)
O1—C1—C2	116.92 (15)	C16—C15—H15	118.7
C3—C2—C1	122.82 (16)	C14—C15—H15	118.7
C3—C2—H2	118.6	C15—C16—C22	118.65 (16)
C1—C2—H2	118.6	C15—C16—C26	121.73 (15)
C2—C3—C9	119.15 (16)	C22—C16—C26	119.61 (15)
C2—C3—C13	120.81 (16)	C18—C17—C22	121.56 (16)
C9—C3—C13	120.03 (16)	C18—C17—H17	119.2
C5—C4—C9	121.63 (16)	C22—C17—H17	119.2
C5—C4—H4	119.2	C17—C18—C19	119.83 (15)
C9—C4—H4	119.2	C17—C18—H18	120.1
C4—C5—C6	119.99 (16)	C19—C18—H18	120.1
C4—C5—H5	120.0	O6—C19—C20	123.65 (15)
C6—C5—H5	120.0	O6—C19—C18	115.75 (14)
O3—C6—C7	124.58 (16)	C20—C19—C18	120.60 (15)
O3—C6—C5	115.22 (15)	C19—C20—C21	118.08 (16)
C7—C6—C5	120.20 (16)	C19—C20—H20	121.0
C8—C7—C6	118.48 (16)	C21—C20—H20	121.0
C8—C7—H7	120.8	O4—C21—C22	121.81 (14)
C6—C7—H7	120.8	O4—C21—C20	115.06 (15)
O1—C8—C7	115.64 (15)	C22—C21—C20	123.13 (15)
O1—C8—C9	121.49 (15)	C21—C22—C17	116.77 (15)
C7—C8—C9	122.87 (16)	C21—C22—C16	118.50 (15)
C8—C9—C4	116.82 (16)	C17—C22—C16	124.73 (15)
C8—C9—C3	118.18 (15)	O6—C23—C24	108.36 (13)
C4—C9—C3	125.00 (16)	O6—C23—H23A	110.0
O3—C10—C11	107.78 (14)	C24—C23—H23A	110.0
O3—C10—H10A	110.2	O6—C23—H23B	110.0
C11—C10—H10A	110.2	C24—C23—H23B	110.0
O3—C10—H10B	110.2	H23A—C23—H23B	108.4
C11—C10—H10B	110.2	C23—C24—C25	110.15 (15)
H10A—C10—H10B	108.5	C23—C24—H24A	109.6
C10—C11—C12	110.13 (15)	C25—C24—H24A	109.6
C10—C11—H11A	109.6	C23—C24—H24B	109.6
C12—C11—H11A	109.6	C25—C24—H24B	109.6
C10—C11—H11B	109.6	H24A—C24—H24B	108.1
C12—C11—H11B	109.6	C24—C25—H25A	109.5
H11A—C11—H11B	108.1	C24—C25—H25B	109.5
C11—C12—H12A	109.5	H25A—C25—H25B	109.5
C11—C12—H12B	109.5	C24—C25—H25C	109.5
H12A—C12—H12B	109.5	H25A—C25—H25C	109.5
C11—C12—H12C	109.5	H25B—C25—H25C	109.5
H12A—C12—H12C	109.5	C16—C26—H26A	109.5
H12B—C12—H12C	109.5	C16—C26—H26B	109.5
C3—C13—H13A	109.5	H26A—C26—H26B	109.5
C3—C13—H13B	109.5	C16—C26—H26C	109.5
H13A—C13—H13B	109.5	H26A—C26—H26C	109.5

C3—C13—H13C	109.5	H26B—C26—H26C	109.5
H13A—C13—H13C	109.5	C8—O1—C1	121.32 (13)
H13B—C13—H13C	109.5	C6—O3—C10	117.68 (13)
O5—C14—O4	115.87 (15)	C21—O4—C14	120.86 (13)
O5—C14—C15	126.62 (15)	C19—O6—C23	117.62 (13)
O2—C1—C2—C3	176.86 (18)	O6—C19—C20—C21	−179.18 (14)
O1—C1—C2—C3	−3.6 (3)	C18—C19—C20—C21	0.7 (2)
C1—C2—C3—C9	1.0 (3)	C19—C20—C21—O4	−179.80 (14)
C1—C2—C3—C13	−179.20 (16)	C19—C20—C21—C22	0.6 (3)
C9—C4—C5—C6	0.2 (3)	O4—C21—C22—C17	178.73 (14)
C4—C5—C6—O3	179.21 (15)	C20—C21—C22—C17	−1.7 (2)
C4—C5—C6—C7	−0.7 (3)	O4—C21—C22—C16	−1.6 (2)
O3—C6—C7—C8	−179.56 (14)	C20—C21—C22—C16	177.96 (15)
C5—C6—C7—C8	0.3 (2)	C18—C17—C22—C21	1.5 (2)
C6—C7—C8—O1	−179.66 (14)	C18—C17—C22—C16	−178.13 (16)
C6—C7—C8—C9	0.5 (3)	C15—C16—C22—C21	1.9 (2)
O1—C8—C9—C4	179.26 (14)	C26—C16—C22—C21	−177.11 (15)
C7—C8—C9—C4	−0.9 (2)	C15—C16—C22—C17	−178.45 (16)
O1—C8—C9—C3	−1.4 (2)	C26—C16—C22—C17	2.6 (3)
C7—C8—C9—C3	178.37 (15)	O6—C23—C24—C25	−179.12 (14)
C5—C4—C9—C8	0.5 (2)	C7—C8—O1—C1	178.93 (14)
C5—C4—C9—C3	−178.70 (16)	C9—C8—O1—C1	−1.2 (2)
C2—C3—C9—C8	1.5 (2)	O2—C1—O1—C8	−176.75 (15)
C13—C3—C9—C8	−178.27 (16)	C2—C1—O1—C8	3.6 (2)
C2—C3—C9—C4	−179.23 (16)	C7—C6—O3—C10	2.9 (2)
C13—C3—C9—C4	1.0 (3)	C5—C6—O3—C10	−176.98 (14)
O3—C10—C11—C12	−179.21 (14)	C11—C10—O3—C6	−178.95 (13)
O5—C14—C15—C16	177.33 (17)	C22—C21—O4—C14	−1.3 (2)
O4—C14—C15—C16	−3.4 (2)	C20—C21—O4—C14	179.11 (14)
C14—C15—C16—C22	0.6 (3)	O5—C14—O4—C21	−176.94 (14)
C14—C15—C16—C26	179.60 (16)	C15—C14—O4—C21	3.7 (2)
C22—C17—C18—C19	−0.3 (3)	C20—C19—O6—C23	1.4 (2)
C17—C18—C19—O6	179.01 (14)	C18—C19—O6—C23	−178.55 (14)
C17—C18—C19—C20	−0.9 (3)	C24—C23—O6—C19	179.83 (13)

Hydrogen-bond geometry (Å, º)

Cg2, Cg3 and Cg4 are the centroids of the O1/C1–C3/C8/C9, C17–C22 and O4/C14–C16/C21/C22 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···O5^{i}	0.95	2.57	3.296 (2)	133
C15—H15···O5^{ii}	0.95	2.49	3.422 (2)	165
C18—H18···O6^{iii}	0.95	2.45	3.402 (2)	175
C11—H11*A*···*Cg*2^{iv}	0.99	2.76	3.6087 (4)	140
C24—H24*A*···*Cg*4^{v}	0.99	2.81	3.613 (2)	139
C23—H23*B*···*Cg*3^{v}	0.99	2.75	3.614 (2)	145

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