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(*E*)-3-[3,4-Bis(methoxymethoxy)phenyl]-1-(7-hydroxy-5-methoxy-2,2-dimethylchroman-8-yl)prop-2-en-1-one

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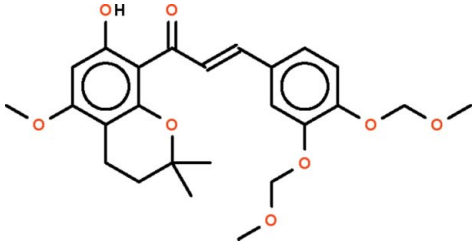
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.032; wR factor = 0.082; data-to-parameter ratio = 8.5.

The reaction of 5,6-(2,2-dimethylchromanyl)-2-hydroxy-4-methoxyacetophenone and 3,4-bis(methoxymethyloxy)-benzaldehyde affords the intense orange title chalcone derivative, $C_{25}H_{30}O_8$. The two benzene rings are connected through a −C(=O)−CH=CH− (propenone) unit, which is in an *E* conformation; the ring with the hydroxy substitutent is aligned at 19.5 (2)° with respect to this unit, whereas the ring with the methoxymethyloxy substituent is aligned at 9.3 (3)°. The dihedral angle between the rings is 19.38 (10)°. The hydroxy group engages in an intramolecular O−H···O hydrogen bond with the carbonyl O atom of the propenone unit, generating an *S*(5) ring.

Related literature

For background to chalcones, see: Avila *et al.* (2008); Narender *et al.* (2007); Reddy *et al.* (2010).



Experimental

Crystal data

$C_{25}H_{30}O_8$
M_r = 458.49
Monoclinic, *Pc*
a = 9.5990 (8) Å
b = 8.3294 (7) Å
c = 14.7444 (12) Å
β = 107.684 (1)°
V = 1123.17 (16) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 100 K
0.30 × 0.25 × 0.05 mm

Data collection

Bruker SMART APEX CCD diffractometer
10316 measured reflections
2576 independent reflections
2433 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.082
S = 1.06
2576 reflections
302 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O4	0.90 (4)	1.65 (4)	2.480 (2)	153 (3)

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

We thank the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6345).

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supplementary materials

Acta Cryst. (2011). E**67**, o2300 [**doi:10.1107/S160053681103131X**]

(*E*)-3-[3,4-Bis(methoxymethoxy)phenyl]-1-(7-hydroxy-5-methoxy-2,2-dimethylchroman-8-yl)prop-2-en-1-one

N. A. Hashim, F. Ahmad, N. Basar, K. Awang and S. W. Ng

Comment

Chalcones or 1,3-diphenyl-2-propen-1-one derivatives flavonoids consist of two aromatic rings that are linked by a three-carbon α, β-unsaturated carbonyl unit (Avila *et al.*, 2008; Reddy *et al.*, 2010), and key precursors for the synthesis of a various flavonoids, some of which are components in food (Narender *et al.*, 2007). We intend to use the intensely-orange title compound, (I), in the synthesis of other compounds. Its two benzene rings are connected through the –C(═ O)–CH═CH– unit, which is of an *E* configuration; the ring with the hydroxy substitutent is aligned at 19.5 (2) ° with this unit whereas the ring with the methoxymethyloxy substituents is aligned at 9.3 (3) °. The hydroxy group engages in intramolecular hydrogen bonding with the carbonyl O atom of the unit (Fig.1).

Experimental

A solution of 2-hydroxy-4-methoxy-5,6-(2,2-dimethylchromane)acetophenone (100 mg, 0.45 mmol) and 3,4-bis(methoxymethyloxy)benzaldehyde (100 mg, 0.45 mmol) in ethanol (10 ml) was treated with 50% potassium hydroxide (1 ml). The mixture was stirred for 48 h. The mixture was poured into iced water (30 ml); this was acidified with 10% hydrochloric acid. The mixture was extracted with dichloromethane (3 *x* 20 ml). The organic layer was washed with water (3 *x* 10 ml) and brine (3 *x* 5 ml) followed by drying over anhydrous magnesium sulfate. The solvent was evaporated under reduced pressure to yield a dark greenish syrup. The syrup was subjected to VLC for purification by using silica gel and eluting with a hexane:ethyl acetate solvent system (9:1) to give the title compound (520 mg, 30%) as orange prisms of (I), m.p. 363–368 K. The formulation was established by ^{1}H– and ^{13}C-NMR spectroscopy.

Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 to 0.98 Å) and were included in the refinement in the riding model approximation, with U(H) set to 1.2 to 1.5U_{eq}(C).

The hydroxy H-atom was located in a difference Fourier map, and was freely refined.

In the absence of heavy scatters, 2245 Friedel pairs were merged. Omitted from the refinement were (-3 3 - 8), (-2 8 - 1), (1 1 - 4), (-4 9 3) and (-3 0 16).

Figures

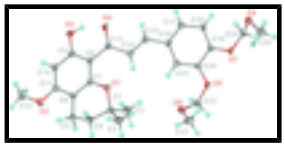


Fig. 1. The molecular structure of (I) at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

(*E*)-3-[3,4-Bis(methoxymethoxy)phenyl]-1-(7-hydroxy-5-methoxy- 2,2-dimethylchroman-8-yl)prop-2-en-1-one

Crystal data

$C_{25}H_{30}O_8$	$F(000) = 488$
$M_r = 458.49$	$D_x = 1.356$ Mg m^{-3}
Monoclinic, *Pc*	Mo *K*α radiation, λ = 0.71073 Å
Hall symbol: P -2yc	Cell parameters from 3857 reflections
$a = 9.5990$ (8) Å	θ = 2.2–28.2°
$b = 8.3294$ (7) Å	μ = 0.10 mm^{-1}
$c = 14.7444$ (12) Å	$T = 100$ K
β = 107.684 (1)°	Prism, orange
$V = 1123.17$ (16) Å^3	0.30 × 0.25 × 0.05 mm
$Z = 2$	

Data collection

Bruker SMART APEX CCD diffractometer	2433 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.037$
graphite	$\theta_{max} = 27.5°$, $\theta_{min} = 2.2°$
ω scans	$h = -12 \rightarrow 12$
10316 measured reflections	$k = -10 \rightarrow 10$
2576 independent reflections	$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.032$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.082$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.06$	$w = 1/[\sigma^2(F_o^2) + (0.0491P)^2 + 0.1273P]$ where $P = (F_o^2 + 2F_c^2)/3$
2576 reflections	$(\Delta/\sigma)_{max} = 0.001$
302 parameters	$\Delta\rho_{max} = 0.25$ e Å^{-3}
2 restraints	$\Delta\rho_{min} = -0.20$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.50254 (17)	0.07085 (17)	0.49935 (10)	0.0174 (3)
O2	0.69328 (17)	0.04190 (19)	0.83437 (11)	0.0204 (3)
O3	0.79441 (18)	0.4985 (2)	0.67354 (12)	0.0221 (3)
H3	0.777 (4)	0.526 (4)	0.612 (3)	0.042 (9)*

O4	0.68542 (17)	0.51474 (19)	0.49826 (11)	0.0198 (3)
O5	0.01705 (16)	0.23344 (18)	0.10555 (10)	0.0178 (3)
O6	0.06369 (19)	0.0284 (2)	0.22016 (12)	0.0251 (4)
O7	0.05288 (16)	0.41671 (18)	−0.02881 (10)	0.0187 (3)
O8	0.05354 (17)	0.66810 (19)	−0.10124 (11)	0.0210 (3)
C1	0.4074 (2)	−0.0676 (2)	0.49859 (16)	0.0181 (4)
C2	0.3732 (3)	−0.1334 (3)	0.39786 (16)	0.0237 (5)
H2A	0.4635	−0.1720	0.3874	0.036*
H2B	0.3304	−0.0482	0.3520	0.036*
H2C	0.3036	−0.2224	0.3894	0.036*
C3	0.2692 (2)	−0.0069 (3)	0.51717 (18)	0.0228 (5)
H3A	0.2199	0.0701	0.4675	0.034*
H3B	0.2949	0.0460	0.5795	0.034*
H3C	0.2038	−0.0975	0.5165	0.034*
C4	0.4918 (2)	−0.1879 (3)	0.57238 (15)	0.0194 (4)
H4A	0.5793	−0.2241	0.5559	0.023*
H4B	0.4295	−0.2829	0.5718	0.023*
C5	0.5390 (2)	−0.1138 (3)	0.67197 (15)	0.0193 (4)
H5A	0.4542	−0.1096	0.6969	0.023*
H5B	0.6152	−0.1819	0.7153	0.023*
C6	0.5981 (2)	0.0529 (2)	0.66954 (15)	0.0160 (4)
C7	0.5763 (2)	0.1365 (3)	0.58478 (15)	0.0149 (4)
C8	0.6366 (2)	0.2927 (3)	0.58144 (15)	0.0148 (4)
C9	0.7257 (2)	0.3568 (3)	0.66940 (15)	0.0166 (4)
C10	0.7437 (2)	0.2780 (3)	0.75539 (15)	0.0174 (4)
H10	0.7988	0.3259	0.8137	0.021*
C11	0.6798 (2)	0.1281 (3)	0.75453 (15)	0.0161 (4)
C12	0.7820 (3)	0.1083 (3)	0.92334 (15)	0.0225 (5)
H12A	0.7826	0.0346	0.9753	0.034*
H12B	0.7416	0.2120	0.9342	0.034*
H12C	0.8822	0.1233	0.9211	0.034*
C13	0.6066 (2)	0.3942 (3)	0.49629 (14)	0.0150 (4)
C14	0.4826 (2)	0.3661 (3)	0.41004 (15)	0.0163 (4)
H14	0.4162	0.2807	0.4081	0.020*
C15	0.4642 (2)	0.4620 (3)	0.33424 (15)	0.0167 (4)
H15	0.5348	0.5446	0.3407	0.020*
C16	0.3495 (2)	0.4553 (3)	0.24339 (15)	0.0160 (4)
C17	0.3573 (2)	0.5633 (2)	0.17258 (15)	0.0178 (4)
H17	0.4318	0.6428	0.1869	0.021*
C18	0.2588 (2)	0.5565 (3)	0.08200 (15)	0.0174 (4)
H18	0.2660	0.6310	0.0349	0.021*
C19	0.1493 (2)	0.4408 (3)	0.05985 (14)	0.0163 (4)
C20	0.1330 (2)	0.3372 (2)	0.13222 (15)	0.0147 (4)
C21	0.2341 (2)	0.3429 (2)	0.22193 (15)	0.0159 (4)
H21	0.2257	0.2703	0.2697	0.019*
C22	−0.0247 (2)	0.1575 (3)	0.18001 (15)	0.0201 (4)
H22A	−0.0210	0.2375	0.2303	0.024*
H22B	−0.1270	0.1199	0.1543	0.024*
C23	0.0360 (3)	−0.1112 (3)	0.16131 (19)	0.0282 (5)

H23A	0.1017	−0.1979	0.1934	0.042*
H23B	0.0531	−0.0866	0.1006	0.042*
H23C	−0.0657	−0.1451	0.1496	0.042*
C24	0.0800 (2)	0.5048 (3)	−0.10506 (15)	0.0178 (4)
H24A	0.1831	0.4887	−0.1030	0.021*
H24B	0.0171	0.4616	−0.1664	0.021*
C25	−0.0952 (3)	0.7036 (3)	−0.10670 (18)	0.0271 (5)
H25A	−0.1074	0.8200	−0.1034	0.041*
H25B	−0.1188	0.6520	−0.0535	0.041*
H25C	−0.1607	0.6630	−0.1670	0.041*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0221 (8)	0.0131 (7)	0.0157 (7)	−0.0047 (6)	0.0039 (6)	−0.0013 (6)
O2	0.0239 (8)	0.0202 (8)	0.0156 (7)	−0.0001 (6)	0.0038 (6)	0.0013 (6)
O3	0.0244 (8)	0.0185 (8)	0.0178 (8)	−0.0070 (6)	−0.0018 (6)	0.0009 (6)
O4	0.0205 (8)	0.0183 (8)	0.0175 (7)	−0.0055 (6)	0.0013 (6)	0.0006 (6)
O5	0.0174 (7)	0.0182 (8)	0.0161 (7)	−0.0057 (6)	0.0026 (6)	0.0007 (6)
O6	0.0279 (9)	0.0205 (8)	0.0221 (8)	−0.0044 (7)	0.0004 (7)	0.0051 (6)
O7	0.0202 (8)	0.0189 (8)	0.0149 (7)	−0.0030 (6)	0.0023 (6)	0.0022 (6)
O8	0.0227 (8)	0.0182 (8)	0.0206 (8)	0.0002 (6)	0.0044 (7)	0.0026 (6)
C1	0.0199 (11)	0.0120 (10)	0.0202 (10)	−0.0048 (8)	0.0030 (8)	−0.0014 (8)
C2	0.0278 (12)	0.0180 (11)	0.0216 (11)	−0.0053 (9)	0.0019 (10)	−0.0033 (9)
C3	0.0165 (10)	0.0197 (10)	0.0313 (12)	0.0002 (8)	0.0061 (9)	0.0020 (9)
C4	0.0200 (11)	0.0128 (10)	0.0226 (11)	−0.0019 (8)	0.0024 (9)	0.0000 (8)
C5	0.0209 (11)	0.0150 (10)	0.0191 (11)	−0.0016 (8)	0.0020 (9)	0.0025 (8)
C6	0.0150 (10)	0.0137 (10)	0.0192 (10)	0.0007 (8)	0.0050 (8)	0.0002 (8)
C7	0.0116 (9)	0.0144 (10)	0.0179 (10)	0.0010 (7)	0.0032 (8)	−0.0017 (8)
C8	0.0133 (10)	0.0140 (10)	0.0165 (9)	0.0000 (8)	0.0035 (8)	−0.0017 (7)
C9	0.0146 (10)	0.0151 (10)	0.0194 (11)	0.0007 (8)	0.0039 (8)	−0.0004 (8)
C10	0.0149 (10)	0.0189 (10)	0.0161 (10)	0.0004 (8)	0.0010 (8)	−0.0016 (8)
C11	0.0134 (10)	0.0181 (10)	0.0167 (10)	0.0031 (8)	0.0044 (8)	0.0030 (8)
C12	0.0289 (12)	0.0225 (11)	0.0134 (10)	0.0044 (10)	0.0022 (9)	0.0010 (9)
C13	0.0143 (10)	0.0139 (10)	0.0167 (10)	0.0004 (8)	0.0046 (8)	−0.0009 (7)
C14	0.0162 (10)	0.0134 (10)	0.0179 (10)	−0.0016 (8)	0.0030 (8)	−0.0014 (8)
C15	0.0178 (10)	0.0138 (10)	0.0188 (10)	−0.0012 (8)	0.0058 (8)	−0.0023 (8)
C16	0.0167 (10)	0.0150 (10)	0.0169 (10)	−0.0001 (8)	0.0058 (8)	−0.0005 (8)
C17	0.0203 (11)	0.0139 (10)	0.0189 (10)	−0.0024 (8)	0.0055 (8)	−0.0005 (8)
C18	0.0190 (10)	0.0162 (11)	0.0172 (10)	−0.0007 (8)	0.0059 (8)	0.0018 (8)
C19	0.0189 (10)	0.0152 (10)	0.0140 (10)	0.0018 (8)	0.0037 (8)	−0.0006 (8)
C20	0.0156 (10)	0.0106 (9)	0.0189 (10)	−0.0005 (8)	0.0065 (8)	−0.0020 (8)
C21	0.0166 (10)	0.0143 (10)	0.0170 (10)	0.0006 (8)	0.0054 (8)	0.0016 (8)
C22	0.0206 (11)	0.0229 (11)	0.0169 (10)	−0.0047 (9)	0.0057 (9)	−0.0004 (8)
C23	0.0282 (13)	0.0222 (12)	0.0345 (14)	−0.0057 (10)	0.0099 (11)	−0.0002 (10)
C24	0.0186 (10)	0.0193 (11)	0.0149 (10)	0.0002 (8)	0.0043 (8)	0.0020 (8)
C25	0.0267 (13)	0.0280 (12)	0.0260 (12)	0.0072 (10)	0.0070 (10)	0.0006 (10)

Geometric parameters (Å, °)

O1—C7	1.358 (3)	C8—C9	1.423 (3)
O1—C1	1.469 (2)	C8—C13	1.467 (3)
O2—C11	1.351 (3)	C9—C10	1.391 (3)
O2—C12	1.440 (3)	C10—C11	1.390 (3)
O3—C9	1.344 (3)	C10—H10	0.9500
O3—H3	0.90 (4)	C12—H12A	0.9800
O4—C13	1.253 (3)	C12—H12B	0.9800
O5—C20	1.369 (2)	C12—H12C	0.9800
O5—C22	1.426 (2)	C13—C14	1.472 (3)
O6—C22	1.387 (3)	C14—C15	1.341 (3)
O6—C23	1.427 (3)	C14—H14	0.9500
O7—C19	1.368 (3)	C15—C16	1.454 (3)
O7—C24	1.431 (2)	C15—H15	0.9500
O8—C24	1.388 (3)	C16—C17	1.397 (3)
O8—C25	1.436 (3)	C16—C21	1.410 (3)
C1—C4	1.519 (3)	C17—C18	1.384 (3)
C1—C3	1.520 (3)	C17—H17	0.9500
C1—C2	1.523 (3)	C18—C19	1.389 (3)
C2—H2A	0.9800	C18—H18	0.9500
C2—H2B	0.9800	C19—C20	1.418 (3)
C2—H2C	0.9800	C20—C21	1.383 (3)
C3—H3A	0.9800	C21—H21	0.9500
C3—H3B	0.9800	C22—H22A	0.9900
C3—H3C	0.9800	C22—H22B	0.9900
C4—C5	1.529 (3)	C23—H23A	0.9800
C4—H4A	0.9900	C23—H23B	0.9800
C4—H4B	0.9900	C23—H23C	0.9800
C5—C6	1.504 (3)	C24—H24A	0.9900
C5—H5A	0.9900	C24—H24B	0.9900
C5—H5B	0.9900	C25—H25A	0.9800
C6—C7	1.390 (3)	C25—H25B	0.9800
C6—C11	1.407 (3)	C25—H25C	0.9800
C7—C8	1.431 (3)		
C7—O1—C1	118.07 (16)	O2—C12—H12B	109.5
C11—O2—C12	117.60 (17)	H12A—C12—H12B	109.5
C9—O3—H3	104 (2)	O2—C12—H12C	109.5
C20—O5—C22	116.93 (16)	H12A—C12—H12C	109.5
C22—O6—C23	113.33 (18)	H12B—C12—H12C	109.5
C19—O7—C24	116.34 (16)	O4—C13—C8	118.78 (18)
C24—O8—C25	113.00 (18)	O4—C13—C14	118.10 (18)
O1—C1—C4	108.51 (17)	C8—C13—C14	123.03 (18)
O1—C1—C3	108.19 (17)	C15—C14—C13	119.54 (19)
C4—C1—C3	113.25 (18)	C15—C14—H14	120.2
O1—C1—C2	103.86 (16)	C13—C14—H14	120.2
C4—C1—C2	111.78 (18)	C14—C15—C16	128.0 (2)
C3—C1—C2	110.74 (19)	C14—C15—H15	116.0

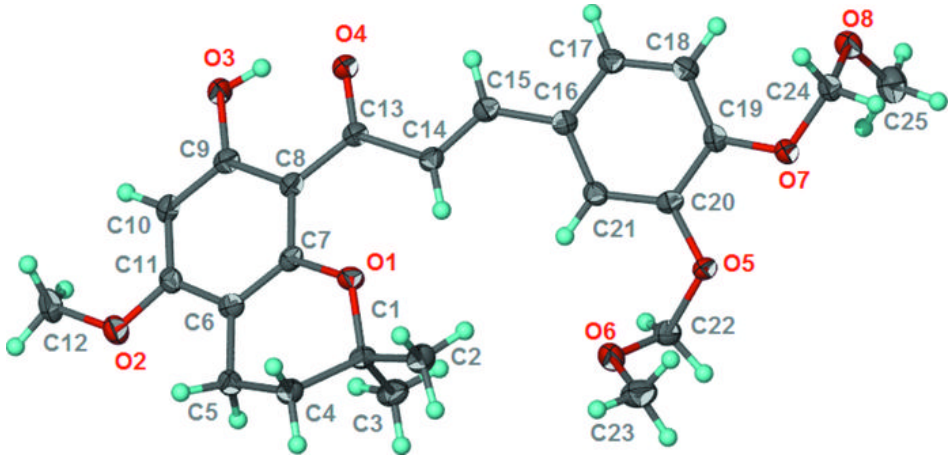
C1—C2—H2A	109.5	C16—C15—H15	116.0
C1—C2—H2B	109.5	C17—C16—C21	118.45 (19)
H2A—C2—H2B	109.5	C17—C16—C15	117.75 (19)
C1—C2—H2C	109.5	C21—C16—C15	123.77 (18)
H2A—C2—H2C	109.5	C18—C17—C16	121.24 (19)
H2B—C2—H2C	109.5	C18—C17—H17	119.4
C1—C3—H3A	109.5	C16—C17—H17	119.4
C1—C3—H3B	109.5	C17—C18—C19	120.12 (19)
H3A—C3—H3B	109.5	C17—C18—H18	119.9
C1—C3—H3C	109.5	C19—C18—H18	119.9
H3A—C3—H3C	109.5	O7—C19—C18	124.78 (18)
H3B—C3—H3C	109.5	O7—C19—C20	115.63 (18)
C1—C4—C5	111.09 (18)	C18—C19—C20	119.59 (19)
C1—C4—H4A	109.4	O5—C20—C21	124.72 (18)
C5—C4—H4A	109.4	O5—C20—C19	115.73 (18)
C1—C4—H4B	109.4	C21—C20—C19	119.53 (18)
C5—C4—H4B	109.4	C20—C21—C16	120.83 (18)
H4A—C4—H4B	108.0	C20—C21—H21	119.6
C6—C5—C4	110.70 (17)	C16—C21—H21	119.6
C6—C5—H5A	109.5	O6—C22—O5	113.22 (18)
C4—C5—H5A	109.5	O6—C22—H22A	108.9
C6—C5—H5B	109.5	O5—C22—H22A	108.9
C4—C5—H5B	109.5	O6—C22—H22B	108.9
H5A—C5—H5B	108.1	O5—C22—H22B	108.9
C7—C6—C11	117.93 (19)	H22A—C22—H22B	107.7
C7—C6—C5	121.93 (19)	O6—C23—H23A	109.5
C11—C6—C5	120.13 (18)	O6—C23—H23B	109.5
O1—C7—C6	121.74 (18)	H23A—C23—H23B	109.5
O1—C7—C8	115.87 (17)	O6—C23—H23C	109.5
C6—C7—C8	122.31 (19)	H23A—C23—H23C	109.5
C9—C8—C7	116.57 (18)	H23B—C23—H23C	109.5
C9—C8—C13	118.11 (18)	O8—C24—O7	113.05 (17)
C7—C8—C13	125.21 (18)	O8—C24—H24A	109.0
O3—C9—C10	116.71 (19)	O7—C24—H24A	109.0
O3—C9—C8	121.45 (19)	O8—C24—H24B	109.0
C10—C9—C8	121.84 (19)	O7—C24—H24B	109.0
C9—C10—C11	118.90 (19)	H24A—C24—H24B	107.8
C9—C10—H10	120.5	O8—C25—H25A	109.5
C11—C10—H10	120.5	O8—C25—H25B	109.5
O2—C11—C10	122.98 (19)	H25A—C25—H25B	109.5
O2—C11—C6	114.77 (18)	O8—C25—H25C	109.5
C10—C11—C6	122.24 (19)	H25A—C25—H25C	109.5
O2—C12—H12A	109.5	H25B—C25—H25C	109.5
C7—O1—C1—C4	−47.8 (2)	C5—C6—C11—C10	176.19 (19)
C7—O1—C1—C3	75.5 (2)	C9—C8—C13—O4	16.7 (3)
C7—O1—C1—C2	−166.83 (18)	C7—C8—C13—O4	−167.3 (2)
O1—C1—C4—C5	60.1 (2)	C9—C8—C13—C14	−159.84 (19)
C3—C1—C4—C5	−60.0 (2)	C7—C8—C13—C14	16.2 (3)
C2—C1—C4—C5	174.09 (18)	O4—C13—C14—C15	4.9 (3)

C1—C4—C5—C6	−43.8 (2)	C8—C13—C14—C15	−178.5 (2)
C4—C5—C6—C7	14.6 (3)	C13—C14—C15—C16	−179.6 (2)
C4—C5—C6—C11	−164.52 (19)	C14—C15—C16—C17	−176.5 (2)
C1—O1—C7—C6	18.7 (3)	C14—C15—C16—C21	1.3 (3)
C1—O1—C7—C8	−164.55 (17)	C21—C16—C17—C18	−2.9 (3)
C11—C6—C7—O1	177.99 (18)	C15—C16—C17—C18	175.06 (19)
C5—C6—C7—O1	−1.2 (3)	C16—C17—C18—C19	0.0 (3)
C11—C6—C7—C8	1.5 (3)	C24—O7—C19—C18	7.9 (3)
C5—C6—C7—C8	−177.66 (19)	C24—O7—C19—C20	−171.75 (17)
O1—C7—C8—C9	−174.29 (17)	C17—C18—C19—O7	−175.3 (2)
C6—C7—C8—C9	2.4 (3)	C17—C18—C19—C20	4.3 (3)
O1—C7—C8—C13	9.6 (3)	C22—O5—C20—C21	17.4 (3)
C6—C7—C8—C13	−173.71 (19)	C22—O5—C20—C19	−164.53 (18)
C7—C8—C9—O3	176.20 (19)	O7—C19—C20—O5	−4.1 (3)
C13—C8—C9—O3	−7.4 (3)	C18—C19—C20—O5	176.19 (18)
C7—C8—C9—C10	−5.1 (3)	O7—C19—C20—C21	174.01 (18)
C13—C8—C9—C10	171.26 (19)	C18—C19—C20—C21	−5.7 (3)
O3—C9—C10—C11	−177.43 (19)	O5—C20—C21—C16	−179.29 (19)
C8—C9—C10—C11	3.8 (3)	C19—C20—C21—C16	2.7 (3)
C12—O2—C11—C10	−1.9 (3)	C17—C16—C21—C20	1.5 (3)
C12—O2—C11—C6	176.82 (18)	C15—C16—C21—C20	−176.33 (19)
C9—C10—C11—O2	179.01 (19)	C23—O6—C22—O5	−75.0 (2)
C9—C10—C11—C6	0.4 (3)	C20—O5—C22—O6	−79.4 (2)
C7—C6—C11—O2	178.28 (17)	C25—O8—C24—O7	−60.4 (2)
C5—C6—C11—O2	−2.6 (3)	C19—O7—C24—O8	−70.3 (2)
C7—C6—C11—C10	−3.0 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O4	0.90 (4)	1.65 (4)	2.480 (2)	153 (3)

Fig. 1


O8
O4
O3
C17
C18
C15
C16
C24
C19
C25
C13
C14
C9
C8
O7
C10
C20
C7
C21
C11
O1
O5
C1
C6
O6
C22
C12
O2
C2
C5
C4
C3
C23