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(3*E*)-4-(3,4,5-Trimethoxyphenyl)but-3-en-2-one

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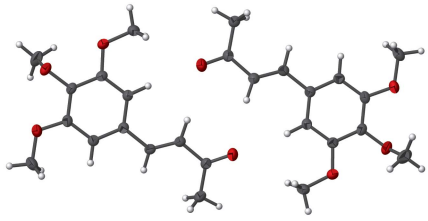
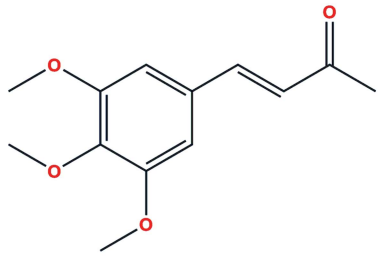
The asymmetric unit of the title compound, $C_{13}H_{16}O_4$, consists of two independent molecules, which are linked into dimers by four C—H···O hydrogen bonds. In both molecules, the C atoms of the *meta*-methoxy groups lie close to their respective ring planes, whereas the C atoms of *para*-methoxy groups are significantly displaced. Pairwise C—H···π(ring) interactions form stacks of dimers, which are further associated through additional C—H···O hydrogen bonds.

3D view



Chemical scheme



Structure description

α,β-Unsaturated carbonyl compounds have displayed diverse biological activities such as antitumor, antimicrobial and plant growth regulatory activity (El-Subbagh *et al.*, 2000; Amslinger, 2010; Tanaka *et al.*, 2003; Bag *et al.* 2009). As part of our studies in this area, the title compound was synthesized and its crystal structure is described here.

The asymmetric unit consists of two independent molecules, *A* (containing C1) and *B* (containing C14) with similar conformations (r.m.s. overlay fit = 0.119 Å). In both molecules, the carbon atoms of the *meta*-methoxy groups lie close to their respective ring planes [for molecule *A*, deviations for C11 and C13 = 0.156 (1) and 0.060 (1) Å, respectively; for molecule *B*, deviations for C24 and C26 = −0.027 (1) and 0.054 (1) Å, respectively], whereas the C atoms of the *para*-methoxy groups are significantly displaced [deviations of C12 (molecule *A*) and C25 (molecule *B*) = −1.213 (1) and 1.273 (1) Å, respectively]. The torsion angles C1—C2—C3—C4 [1.02 (16)] and C14—C15—C16—C17 [1.96 (16)°] indicate very similar, near planar, conformations for the but-3-en-2-one side chains.

In the crystal, the two independent molecules are linked by four C—H···O hydrogen bonds (Table 1 and Fig. 1). These dimeric units stack in an alternating fashion through

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

*Cg*1 and *Cg*2 are the centroids of the C5–C10 andC18–C23 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O5	0.992 (14)	2.409 (14)	3.3993 (13)	175.9 (11)
C10—H10···O5	0.945 (13)	2.531 (13)	3.4713 (12)	173.7 (11)
C16—H16···O1	0.988 (14)	2.442 (14)	3.4106 (13)	166.6 (11)
C19—H19···O1	0.961 (13)	2.471 (13)	3.4267 (13)	173.0 (10)
C23—H23···O7^{i}	0.968 (14)	2.573 (14)	3.4492 (12)	150.7 (10)
C1—H1*B*···*Cg*1^{ii}	0.988 (15)	2.655 (14)	3.411 (1)	133.6 (11)
C1—H1*C*···*Cg*2^{iii}	1.008 (15)	2.662 (14)	3.490 (1)	139.5 (11)

Symmetry codes: (i) $-x+2, y-\frac{1}{2}, -z+\frac{3}{2}$; (ii) $x-1, y, z$; (iii) $-x, -y+1, -z+1$.

pairwise C—H···π(ring) interactions (Figs. 2 and 3, Table 1) . The normals to the stacks are inclined to (001) by ±39.2 (6)° and the stacks are associated through C23—H23···O7 hydrogen bonds (Table 1 and Fig. 2).

Synthesis and crystallization

A mixture of 2,3,4-trimethoxybenzaldehyde (1.00 g, 5.10 mmol) and sodium hydroxide (5 mmol) in absolute acetone was refluxed for 6 h. After cooling to room temperature, the reaction mixture was concentrated under vacuum; the residue obtained was diluted with CH_2Cl_2, washed successively with H_2O and a solution of sodium bisulfite. The separated organic layer was dried over anhydrous $MgSO_4$ and evaporated under vacuum. The crude product was purified by column chromatography on silica gel using hexane/ethyl acetate (9:1 to 4:5) as an eluent to give (*E*)-5-(3,4,5-trimethoxyphenyl)-pent-4-en-2-one as a white solid (0.82 g, yield: 69%), m.p. 120–122°C. Colourless blocks were recrystallized from hexane/ethyl acetate (9:1) solution.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_{16}O_4$
M_r	236.26
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	150
a, *b*, *c* (Å)	8.0082 (2), 12.9854 (3), 24.0205 (5)
β (°)	92.643 (1)
V (Å^3)	2495.23 (10)
Z	8
Radiation type	Cu *K*α
μ (mm^{-1})	0.77
Crystal size (mm)	0.21 × 0.21 × 0.17
Data collection	
Diffractometer	Bruker D8 VENTURE PHOTON 100 CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.82, 0.88
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	41918, 4902, 4505
R_{int}	0.034
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.617
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.034, 0.093, 1.03
No. of reflections	4902
No. of parameters	436
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.25, −0.17

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2016* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Sheldrick, 2008).

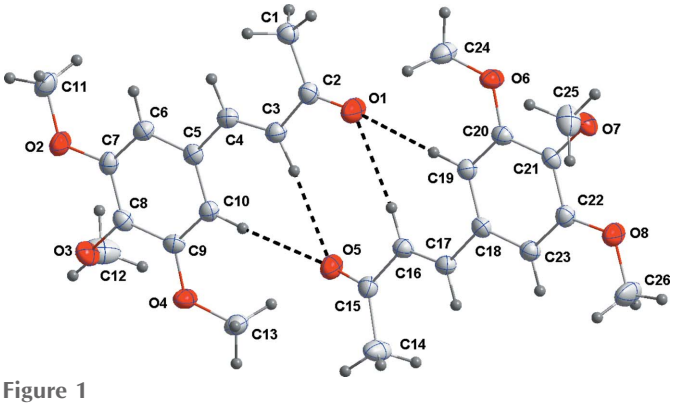


Figure 1
The asymmetric unit showing 50% probability ellipsoids. C—H···O hydrogen bonds are shown by dashed lines

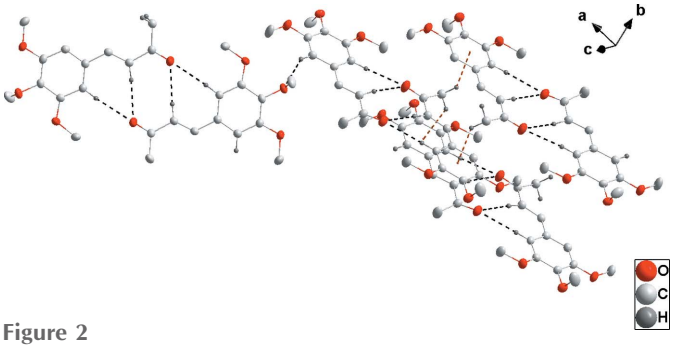


Figure 2
Detail of the C—H···O hydrogen bonds (black dashed lines) and C—H···π(ring) interactions (orange dashed lines).

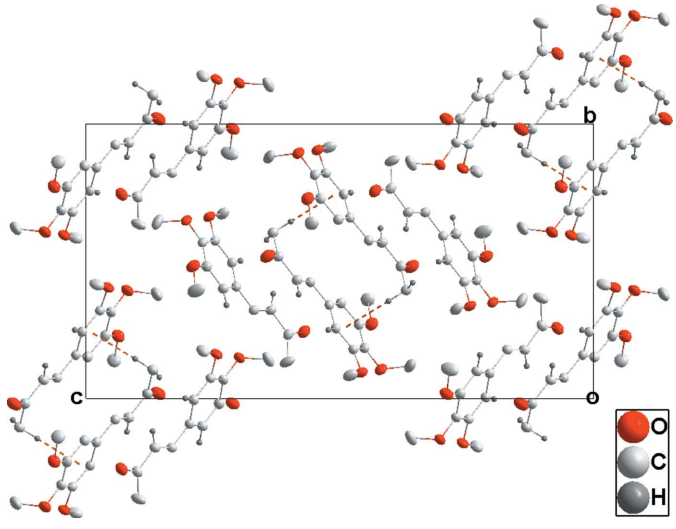


Figure 3
Packing viewed along the *a*-axis direction.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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full crystallographic data

IUCrData (2017). **2**, x171531 [https://doi.org/10.1107/S2414314617015310]

(3*E*)-4-(3,4,5-Trimethoxyphenyl)but-3-en-2-one

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Crystal data

$C_{13}H_{16}O_4$
M_r = 236.26
Monoclinic, $P2_1/c$
a = 8.0082 (2) Å
b = 12.9854 (3) Å
c = 24.0205 (5) Å
β = 92.643 (1)°
V = 2495.23 (10) Å^3
Z = 8
$F(000)$ = 1008
D_x = 1.258 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 9930 reflections
θ = 3.4–72.0°
μ = 0.77 mm^{-1}
T = 150 K
Block, colourless
0.21 × 0.21 × 0.17 mm

Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer
Radiation source: INCOATEC IμS micro-focus source
Mirror monochromator
Detector resolution: 10.4167 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2016)
T_{min} = 0.82, T_{max} = 0.88
41918 measured reflections
4902 independent reflections
4505 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 72.1°, θ_{min} = 3.7°
h = −9→9
k = −16→15
l = −29→29

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.093
S = 1.03
4902 reflections
436 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0493P)^2 + 0.6003P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: *SHELXL2016* (Sheldrick, 2015*b*), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00194 (18)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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	0.29813 (10)	0.51899 (7)	0.64195 (4)	0.0425 (2)
O2	−0.48758 (9)	0.27182 (6)	0.43679 (3)	0.03424 (19)
O3	−0.29964 (10)	0.10633 (6)	0.41871 (3)	0.03521 (19)
O4	0.01776 (9)	0.09503 (6)	0.45696 (3)	0.03171 (18)
C1	0.03774 (14)	0.60440 (8)	0.63148 (4)	0.0279 (2)
H1A	0.0858 (18)	0.6492 (11)	0.6602 (6)	0.042 (4)*
H1B	−0.0698 (18)	0.5781 (11)	0.6442 (6)	0.039 (3)*
H1C	0.0111 (18)	0.6448 (11)	0.5964 (6)	0.042 (4)*
C2	0.15675 (13)	0.51798 (8)	0.62076 (4)	0.0268 (2)
C3	0.10454 (13)	0.43165 (8)	0.58393 (4)	0.0264 (2)
H3	0.1938 (17)	0.3794 (10)	0.5805 (5)	0.036 (3)*
C4	−0.04518 (12)	0.42417 (8)	0.55755 (4)	0.0241 (2)
H4	−0.1295 (16)	0.4788 (10)	0.5631 (5)	0.035 (3)*
C5	−0.10489 (12)	0.34091 (7)	0.52037 (4)	0.0231 (2)
C6	−0.26746 (13)	0.34810 (8)	0.49727 (4)	0.0253 (2)
H6	−0.3351 (16)	0.4072 (10)	0.5063 (5)	0.034 (3)*
C7	−0.33180 (12)	0.27092 (8)	0.46229 (4)	0.0258 (2)
C8	−0.23404 (13)	0.18514 (8)	0.45093 (4)	0.0257 (2)
C9	−0.06871 (12)	0.17915 (7)	0.47294 (4)	0.0242 (2)
C10	−0.00402 (12)	0.25640 (8)	0.50783 (4)	0.0232 (2)
H10	0.1073 (16)	0.2501 (10)	0.5222 (5)	0.030 (3)*
C11	−0.58786 (15)	0.36096 (10)	0.44473 (6)	0.0382 (3)
H11A	−0.695 (2)	0.3482 (11)	0.4226 (6)	0.049 (4)*
H11B	−0.6089 (18)	0.3711 (11)	0.4843 (6)	0.043 (4)*
H11C	−0.5323 (17)	0.4216 (11)	0.4303 (6)	0.040 (4)*
C12	−0.2718 (2)	0.11984 (11)	0.36107 (5)	0.0511 (4)
H12A	−0.146 (2)	0.1203 (14)	0.3561 (7)	0.069 (5)*
H12B	−0.327 (2)	0.0602 (13)	0.3429 (7)	0.058 (4)*
H12C	−0.323 (2)	0.1859 (15)	0.3475 (8)	0.075 (5)*
C13	0.18670 (15)	0.08494 (9)	0.47890 (6)	0.0377 (3)
H13A	0.191 (2)	0.0816 (13)	0.5213 (7)	0.056 (4)*
H13B	0.2257 (19)	0.0207 (13)	0.4624 (6)	0.052 (4)*
H13C	0.2571 (19)	0.1425 (12)	0.4654 (6)	0.048 (4)*
O5	0.39547 (9)	0.24392 (6)	0.56998 (3)	0.03445 (19)
O6	0.65526 (9)	0.66230 (6)	0.75722 (3)	0.03113 (18)

O7	0.96636 (9)	0.64896 (5)	0.80226 (3)	0.02955 (18)
O8	1.15432 (10)	0.48257 (6)	0.78899 (3)	0.0365 (2)
C14	0.61263 (17)	0.13407 (9)	0.60343 (6)	0.0427 (3)
H14A	0.642 (2)	0.1123 (15)	0.6406 (8)	0.073 (5)*
H14B	0.726 (3)	0.1399 (14)	0.5836 (8)	0.077 (6)*
H14C	0.545 (2)	0.0790 (13)	0.5856 (7)	0.055 (4)*
C15	0.51946 (12)	0.23414 (8)	0.60112 (4)	0.0261 (2)
C16	0.57817 (13)	0.32196 (8)	0.63545 (4)	0.0254 (2)
H16	0.5028 (17)	0.3819 (10)	0.6310 (5)	0.036 (3)*
C17	0.71590 (12)	0.32177 (8)	0.66902 (4)	0.0242 (2)
H17	0.7865 (17)	0.2618 (11)	0.6727 (5)	0.034 (3)*
C18	0.77707 (12)	0.40783 (7)	0.70405 (4)	0.0231 (2)
C19	0.67849 (12)	0.49514 (8)	0.71213 (4)	0.0239 (2)
H19	0.5674 (17)	0.5016 (10)	0.6956 (5)	0.032 (3)*
C20	0.74141 (12)	0.57473 (7)	0.74561 (4)	0.0241 (2)
C21	0.90263 (12)	0.56878 (7)	0.77051 (4)	0.0241 (2)
C22	1.00034 (13)	0.48150 (8)	0.76229 (4)	0.0258 (2)
C23	0.93722 (13)	0.40051 (8)	0.72937 (4)	0.0257 (2)
H23	1.0054 (17)	0.3403 (10)	0.7234 (5)	0.035 (3)*
C24	0.49369 (15)	0.67358 (10)	0.73020 (5)	0.0356 (3)
H24A	0.417 (2)	0.6202 (12)	0.7418 (6)	0.049 (4)*
H24B	0.4551 (18)	0.7421 (11)	0.7418 (6)	0.043 (4)*
H24C	0.5000 (18)	0.6698 (11)	0.6896 (6)	0.045 (4)*
C25	0.92100 (17)	0.64289 (11)	0.85914 (5)	0.0416 (3)
H25A	0.9666 (19)	0.7044 (12)	0.8772 (6)	0.051 (4)*
H25B	0.970 (2)	0.5766 (14)	0.8773 (7)	0.063 (5)*
H25C	0.799 (2)	0.6440 (12)	0.8602 (7)	0.056 (4)*
C26	1.25528 (17)	0.39295 (11)	0.78302 (7)	0.0509 (4)
H26A	1.355 (2)	0.4060 (13)	0.8057 (7)	0.064 (5)*
H26B	1.280 (2)	0.3805 (14)	0.7407 (8)	0.072 (5)*
H26C	1.199 (2)	0.3332 (13)	0.7988 (6)	0.050 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0310 (4)	0.0410 (5)	0.0538 (5)	0.0040 (3)	−0.0153 (4)	−0.0138 (4)
O2	0.0234 (4)	0.0391 (4)	0.0394 (4)	0.0022 (3)	−0.0075 (3)	−0.0058 (3)
O3	0.0380 (4)	0.0309 (4)	0.0358 (4)	−0.0055 (3)	−0.0078 (3)	−0.0083 (3)
O4	0.0319 (4)	0.0256 (4)	0.0371 (4)	0.0050 (3)	−0.0050 (3)	−0.0077 (3)
C1	0.0295 (5)	0.0260 (5)	0.0281 (5)	0.0000 (4)	0.0004 (4)	−0.0023 (4)
C2	0.0266 (5)	0.0272 (5)	0.0264 (5)	−0.0012 (4)	−0.0017 (4)	−0.0004 (4)
C3	0.0261 (5)	0.0251 (5)	0.0280 (5)	0.0017 (4)	0.0004 (4)	−0.0018 (4)
C4	0.0259 (5)	0.0237 (5)	0.0227 (4)	0.0013 (4)	0.0016 (4)	0.0001 (4)
C5	0.0248 (5)	0.0243 (5)	0.0202 (4)	−0.0012 (4)	0.0019 (3)	0.0009 (4)
C6	0.0252 (5)	0.0262 (5)	0.0246 (5)	0.0019 (4)	0.0019 (4)	−0.0006 (4)
C7	0.0221 (5)	0.0305 (5)	0.0246 (5)	−0.0021 (4)	−0.0015 (4)	0.0015 (4)
C8	0.0283 (5)	0.0250 (5)	0.0236 (5)	−0.0038 (4)	−0.0022 (4)	−0.0017 (4)
C9	0.0273 (5)	0.0220 (5)	0.0232 (4)	0.0012 (4)	0.0009 (4)	0.0011 (4)

C10	0.0230 (5)	0.0249 (5)	0.0214 (4)	−0.0005 (4)	−0.0010 (4)	0.0013 (4)
C11	0.0250 (5)	0.0435 (7)	0.0454 (7)	0.0065 (5)	−0.0050 (5)	−0.0003 (5)
C12	0.0768 (11)	0.0429 (7)	0.0322 (6)	0.0109 (7)	−0.0146 (6)	−0.0131 (5)
C13	0.0291 (6)	0.0307 (6)	0.0529 (7)	0.0071 (5)	−0.0034 (5)	−0.0092 (5)
O5	0.0272 (4)	0.0387 (4)	0.0367 (4)	0.0010 (3)	−0.0065 (3)	−0.0090 (3)
O6	0.0324 (4)	0.0254 (4)	0.0350 (4)	0.0049 (3)	−0.0049 (3)	−0.0088 (3)
O7	0.0332 (4)	0.0266 (4)	0.0287 (4)	−0.0068 (3)	−0.0006 (3)	−0.0069 (3)
O8	0.0273 (4)	0.0349 (4)	0.0459 (5)	0.0024 (3)	−0.0119 (3)	−0.0097 (3)
C14	0.0440 (7)	0.0287 (6)	0.0539 (8)	0.0059 (5)	−0.0148 (6)	−0.0153 (5)
C15	0.0233 (5)	0.0284 (5)	0.0266 (5)	−0.0011 (4)	0.0019 (4)	−0.0045 (4)
C16	0.0264 (5)	0.0226 (5)	0.0272 (5)	0.0010 (4)	0.0005 (4)	−0.0028 (4)
C17	0.0250 (5)	0.0221 (5)	0.0255 (5)	0.0007 (4)	0.0019 (4)	−0.0019 (4)
C18	0.0249 (5)	0.0225 (5)	0.0221 (4)	−0.0019 (4)	0.0013 (4)	0.0002 (4)
C19	0.0239 (5)	0.0242 (5)	0.0235 (5)	−0.0006 (4)	−0.0005 (4)	−0.0011 (4)
C20	0.0275 (5)	0.0219 (5)	0.0231 (5)	0.0002 (4)	0.0028 (4)	−0.0009 (4)
C21	0.0275 (5)	0.0227 (5)	0.0220 (4)	−0.0049 (4)	0.0005 (4)	−0.0025 (4)
C22	0.0239 (5)	0.0273 (5)	0.0258 (5)	−0.0017 (4)	−0.0021 (4)	−0.0004 (4)
C23	0.0266 (5)	0.0233 (5)	0.0270 (5)	0.0004 (4)	−0.0003 (4)	−0.0012 (4)
C24	0.0344 (6)	0.0339 (6)	0.0379 (6)	0.0101 (5)	−0.0061 (5)	−0.0099 (5)
C25	0.0404 (7)	0.0530 (8)	0.0315 (6)	−0.0104 (6)	0.0044 (5)	−0.0172 (5)
C26	0.0326 (6)	0.0441 (7)	0.0737 (10)	0.0108 (6)	−0.0210 (6)	−0.0125 (7)

Geometric parameters (Å, º)

O1—C2	1.2198 (13)	O5—C15	1.2219 (12)
O2—C7	1.3642 (12)	O6—C20	1.3655 (12)
O2—C11	1.4266 (14)	O6—C24	1.4281 (13)
O3—C8	1.3729 (12)	O7—C21	1.3748 (12)
O3—C12	1.4234 (16)	O7—C25	1.4321 (14)
O4—C9	1.3581 (12)	O8—C22	1.3635 (12)
O4—C13	1.4352 (13)	O8—C26	1.4280 (15)
C1—C2	1.5023 (14)	C14—C15	1.4982 (15)
C1—H1A	0.968 (15)	C14—H14A	0.956 (19)
C1—H1B	0.988 (15)	C14—H14B	1.05 (2)
C1—H1C	1.008 (15)	C14—H14C	0.982 (17)
C2—C3	1.4765 (14)	C15—C16	1.4716 (13)
C3—C4	1.3336 (14)	C16—C17	1.3358 (14)
C3—H3	0.992 (14)	C16—H16	0.988 (14)
C4—C5	1.4682 (13)	C17—C18	1.4693 (13)
C4—H4	0.993 (14)	C17—H17	0.964 (14)
C5—C6	1.3946 (14)	C18—C23	1.3971 (14)
C5—C10	1.4035 (14)	C18—C19	1.4003 (14)
C6—C7	1.3915 (14)	C19—C20	1.3897 (14)
C6—H6	0.969 (14)	C19—H19	0.961 (13)
C7—C8	1.3955 (15)	C20—C21	1.3998 (14)
C8—C9	1.4049 (14)	C21—C22	1.3964 (14)
C9—C10	1.3918 (14)	C22—C23	1.3965 (14)
C10—H10	0.945 (13)	C23—H23	0.968 (14)

C11—H11A	1.005 (16)	C24—H24A	0.977 (17)
C11—H11B	0.982 (15)	C24—H24B	0.986 (15)
C11—H11C	0.976 (15)	C24—H24C	0.979 (15)
C12—H12A	1.016 (19)	C25—H25A	0.973 (17)
C12—H12B	0.983 (17)	C25—H25B	1.033 (18)
C12—H12C	1.00 (2)	C25—H25C	0.980 (17)
C13—H13A	1.019 (17)	C26—H26A	0.963 (18)
C13—H13B	0.980 (16)	C26—H26B	1.057 (19)
C13—H13C	0.999 (16)	C26—H26C	0.981 (17)
C7—O2—C11	117.11 (8)	C20—O6—C24	116.63 (8)
C8—O3—C12	112.54 (9)	C21—O7—C25	112.55 (8)
C9—O4—C13	117.00 (8)	C22—O8—C26	116.64 (9)
C2—C1—H1A	109.7 (9)	C15—C14—H14A	113.1 (12)
C2—C1—H1B	111.3 (8)	C15—C14—H14B	111.2 (11)
H1A—C1—H1B	108.1 (12)	H14A—C14—H14B	105.1 (15)
C2—C1—H1C	110.9 (8)	C15—C14—H14C	110.7 (9)
H1A—C1—H1C	110.1 (12)	H14A—C14—H14C	107.5 (14)
H1B—C1—H1C	106.6 (11)	H14B—C14—H14C	108.9 (14)
O1—C2—C3	119.34 (9)	O5—C15—C16	119.48 (9)
O1—C2—C1	120.29 (9)	O5—C15—C14	120.13 (9)
C3—C2—C1	120.37 (9)	C16—C15—C14	120.38 (9)
C4—C3—C2	124.30 (9)	C17—C16—C15	124.78 (9)
C4—C3—H3	123.1 (8)	C17—C16—H16	123.4 (8)
C2—C3—H3	112.6 (8)	C15—C16—H16	111.9 (8)
C3—C4—C5	127.09 (9)	C16—C17—C18	126.05 (9)
C3—C4—H4	119.1 (8)	C16—C17—H17	121.5 (8)
C5—C4—H4	113.8 (8)	C18—C17—H17	112.5 (8)
C6—C5—C10	120.19 (9)	C23—C18—C19	120.45 (9)
C6—C5—C4	117.68 (9)	C23—C18—C17	118.21 (9)
C10—C5—C4	122.13 (9)	C19—C18—C17	121.33 (9)
C7—C6—C5	120.33 (9)	C20—C19—C18	119.39 (9)
C7—C6—H6	120.7 (8)	C20—C19—H19	118.6 (7)
C5—C6—H6	119.0 (8)	C18—C19—H19	122.0 (8)
O2—C7—C6	124.78 (9)	O6—C20—C19	124.36 (9)
O2—C7—C8	115.39 (9)	O6—C20—C21	115.06 (8)
C6—C7—C8	119.83 (9)	C19—C20—C21	120.59 (9)
O3—C8—C7	120.11 (9)	O7—C21—C22	119.80 (9)
O3—C8—C9	120.02 (9)	O7—C21—C20	120.44 (9)
C7—C8—C9	119.88 (9)	C22—C21—C20	119.75 (9)
O4—C9—C10	124.71 (9)	O8—C22—C21	115.20 (9)
O4—C9—C8	114.97 (9)	O8—C22—C23	124.74 (9)
C10—C9—C8	120.32 (9)	C21—C22—C23	120.05 (9)
C9—C10—C5	119.40 (9)	C22—C23—C18	119.76 (9)
C9—C10—H10	118.5 (8)	C22—C23—H23	120.0 (8)
C5—C10—H10	122.1 (8)	C18—C23—H23	120.2 (8)
O2—C11—H11A	105.7 (9)	O6—C24—H24A	111.6 (9)
O2—C11—H11B	111.2 (8)	O6—C24—H24B	104.7 (8)

H11A—C11—H11B	110.7 (12)	H24A—C24—H24B	110.4 (12)
O2—C11—H11C	109.8 (8)	O6—C24—H24C	111.1 (9)
H11A—C11—H11C	109.7 (12)	H24A—C24—H24C	108.1 (12)
H11B—C11—H11C	109.7 (12)	H24B—C24—H24C	111.0 (12)
O3—C12—H12A	108.3 (10)	O7—C25—H25A	105.9 (9)
O3—C12—H12B	104.4 (10)	O7—C25—H25B	110.0 (10)
H12A—C12—H12B	112.1 (14)	H25A—C25—H25B	111.7 (13)
O3—C12—H12C	110.1 (11)	O7—C25—H25C	108.7 (10)
H12A—C12—H12C	110.4 (15)	H25A—C25—H25C	109.4 (13)
H12B—C12—H12C	111.3 (14)	H25B—C25—H25C	111.0 (14)
O4—C13—H13A	110.8 (9)	O8—C26—H26A	105.1 (11)
O4—C13—H13B	104.0 (9)	O8—C26—H26B	110.7 (10)
H13A—C13—H13B	111.9 (13)	H26A—C26—H26B	112.4 (14)
O4—C13—H13C	110.4 (8)	O8—C26—H26C	109.5 (9)
H13A—C13—H13C	111.4 (12)	H26A—C26—H26C	107.7 (14)
H13B—C13—H13C	108.1 (12)	H26B—C26—H26C	111.2 (14)

O1—C2—C3—C4	−178.21 (10)	O5—C15—C16—C17	−176.86 (10)
C1—C2—C3—C4	1.02 (16)	C14—C15—C16—C17	1.96 (16)
C2—C3—C4—C5	179.85 (9)	C15—C16—C17—C18	−179.55 (9)
C3—C4—C5—C6	179.60 (10)	C16—C17—C18—C23	−169.49 (10)
C3—C4—C5—C10	−0.55 (16)	C16—C17—C18—C19	10.55 (15)
C10—C5—C6—C7	0.96 (14)	C23—C18—C19—C20	0.04 (14)
C4—C5—C6—C7	−179.18 (9)	C17—C18—C19—C20	180.00 (9)
C11—O2—C7—C6	3.10 (15)	C24—O6—C20—C19	−3.61 (14)
C11—O2—C7—C8	−176.46 (10)	C24—O6—C20—C21	176.79 (9)
C5—C6—C7—O2	−178.59 (9)	C18—C19—C20—O6	−178.68 (9)
C5—C6—C7—C8	0.96 (15)	C18—C19—C20—C21	0.89 (14)
C12—O3—C8—C7	91.01 (12)	C25—O7—C21—C22	−94.88 (12)
C12—O3—C8—C9	−89.54 (12)	C25—O7—C21—C20	86.17 (12)
O2—C7—C8—O3	−3.67 (14)	O6—C20—C21—O7	−2.33 (13)
C6—C7—C8—O3	176.75 (9)	C19—C20—C21—O7	178.06 (9)
O2—C7—C8—C9	176.88 (9)	O6—C20—C21—C22	178.72 (9)
C6—C7—C8—C9	−2.70 (15)	C19—C20—C21—C22	−0.89 (14)
C13—O4—C9—C10	1.42 (15)	C26—O8—C22—C21	177.92 (11)
C13—O4—C9—C8	−179.30 (9)	C26—O8—C22—C23	−1.01 (16)
O3—C8—C9—O4	3.80 (13)	O7—C21—C22—O8	2.01 (13)
C7—C8—C9—O4	−176.75 (9)	C20—C21—C22—O8	−179.03 (9)
O3—C8—C9—C10	−176.89 (9)	O7—C21—C22—C23	−179.00 (9)
C7—C8—C9—C10	2.56 (14)	C20—C21—C22—C23	−0.05 (14)
O4—C9—C10—C5	178.59 (9)	O8—C22—C23—C18	179.85 (9)
C8—C9—C10—C5	−0.66 (14)	C21—C22—C23—C18	0.97 (15)
C6—C5—C10—C9	−1.10 (14)	C19—C18—C23—C22	−0.97 (14)
C4—C5—C10—C9	179.05 (9)	C17—C18—C23—C22	179.07 (9)

Hydrogen-bond geometry (Å, º)

Cg1 and Cg2 are the centroids of the C5–C10 andC18–C23 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O5	0.992 (14)	2.409 (14)	3.3993 (13)	175.9 (11)
C10—H10···O5	0.945 (13)	2.531 (13)	3.4713 (12)	173.7 (11)
C16—H16···O1	0.988 (14)	2.442 (14)	3.4106 (13)	166.6 (11)
C19—H19···O1	0.961 (13)	2.471 (13)	3.4267 (13)	173.0 (10)
C23—H23···O7^{i}	0.968 (14)	2.573 (14)	3.4492 (12)	150.7 (10)
C1—H1*B*···*Cg*1^{ii}	0.988 (15)	2.655 (14)	3.411 (1)	133.6 (11)
C1—H1*C*···*Cg*2^{iii}	1.008 (15)	2.662 (14)	3.490 (1)	139.5 (11)

Symmetry codes: (i) −*x*+2, *y*−1/2, −*z*+3/2; (ii) *x*−1, *y*, *z*; (iii) −*x*, −*y*+1, −*z*+1.