



The synthesis, crystal structure and spectroscopic analysis of (*E*)-3-(4-chlorophenyl)-1-(2,3-dihydrobenzo[*b*][1,4]dioxin-6-yl)prop-2-en-1-one

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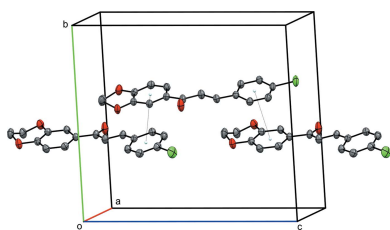
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The synthesis, crystal structure and spectroscopic analysis of (*E*)-1-(2,3-dihydrobenzo[*b*][1,4]dioxin-6-yl)-3-(4-chlorophenyl)prop-2-en-1-one (C₁₇H₁₃ClO₃), a substituted chalcone, are described. The overall geometry of the molecule is largely planar (r.m.s. deviation = 0.1742 Å), but slightly kinked, leading to a dihedral angle between the planes of the benzene rings at either side of the molecule of 8.31 (9)°. In the crystal, only weak interactions determine the packing motifs. These include C—H···O and C—H···Cl hydrogen bonds and π - π overlap of aromatic rings.

1. Chemical context

Chalcones exhibit a wide range of fascinating biological and pharmacological properties. Some of their beneficial attributes include anti-inflammatory, antimicrobial, antifungal, antioxidant, cytotoxic and anticancer activities (Dimmock *et al.*, 1999). Moreover, several chalcones have demonstrated significant antimalarial properties (Troeborg *et al.*, 2000). The efficient second-harmonic generation (SHG) conversion efficiency of some chalcones has also made them promising organic nonlinear optical (NLO) materials (Sarojini *et al.*, 2006). The presence of phenolic groups in many chalcones has garnered attention due to their radical quenching capabilities, making these compounds and chalcone-rich plant extracts potential candidates for drug development or food preservation (Dhar *et al.*, 1981; Di Carlo *et al.*, 1999). The design, synthesis and evaluation of 1,4-benzodioxane-substituted chalcones as selective and reversible inhibitors of human monoamine oxidase B was reported by Kong *et al.* (2020). Furthermore, Shinde *et al.* (2020) have conducted experimental and theoretical studies on the molecular structure, FT-IR, NMR, HOMO/LUMO frontier orbital, molecular electrostatic potential (MESP) and reactivity descriptors of (*E*)-1-(2,3-dihydrobenzo[*b*][1,4]dioxin-6-yl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one. They have also reported the synthesis, antibacterial and computational studies of halo-chalcone hybrids derived from 1-(2,3-dihydrobenzo[*b*][1,4]dioxin-6-yl)ethan-1-one (Shinde *et al.*, 2021) and of two trifluorinated chalcones derived from 1-(2,3-dihydrobenzo[*b*][1,4]dioxin-6-yl)ethan-1-one (Shinde *et al.*, 2022a), as well as investigations of (*E*)-4-(3-(2,3-dihydrobenzo[*b*][1,4]dioxin-6-yl)-3-oxoprop-1-en-1-yl)benzotrile (Shinde *et al.*, 2022b). Additionally, Zhuang *et al.* (2017) presented a comprehensive review on the chalcone



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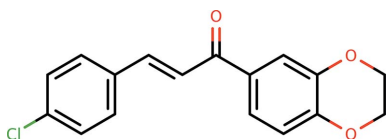
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Table 1
Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C5-H5A\cdots O3^i$	0.99	2.61	3.595 (4)	172
$C5-H5B\cdots Cl1^{ii}$	0.99	2.80	3.455 (3)	124

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

framework as a privileged structure in medicinal chemistry, while Elkanzi *et al.* (2022) have published a review on the synthesis of chalcone derivatives and their biological activities.



Given the general significance of chalcones, we report herein the synthesis of (*E*)-1-(2,3-dihydrobenzo[*b*][1,4]dioxin-6-yl)-3-(4-chlorophenyl)prop-2-en-1-one, $C_{17}H_{13}ClO_3$ (**I**), along with its crystal structure and related studies.

2. Structural commentary

As shown in Fig. 1, the overall conformation of the molecule of **I** is determined by the torsion angles $C3-C2-C1-C10$ [$169.9(2)^\circ$], $C2-C1-C10-C11$ [$-171.3(3)^\circ$], $C1-C10-C11-C12$ [$-179.0(3)^\circ$] and $C10-C11-C12-C17$ [$-176.7(3)^\circ$] of the propenone moiety. In magnitude, these are all within about 10° of 180° , which makes the molecule largely flat (r.m.s. deviation for the non-H atoms is 0.1742 \AA), though slightly bent, leading to a dihedral angle between the chlorophenyl and dihydrobenzodioxine rings of $8.31(9)^\circ$. The conformation of the dihydrodioxine ring, as determined by its Cremer-Pople puckering parameters (Cremer & Pople, 1975) [$Q = 0.466(3) \text{ \AA}$, $\theta = 51.2(4)^\circ$ and $\varphi = 282.6(4)^\circ$], is closest to half-chair (Boeyens, 1978). This puckering places atoms C5 and C6 at $0.499(4)$ and $-0.207(4) \text{ \AA}$ on either side of the mean plane of the dihydrobenzodioxine ring. All bond lengths and angles lie within the typical ranges found in organic structures.

3. Supramolecular features

The crystal packing in **I** is governed solely by weak interactions, as the only possible hydrogen-bond donors are of type

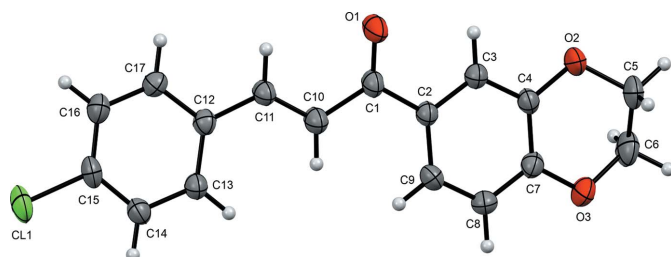


Figure 1

The molecular structure of the title compound, showing the atom labelling and displacement ellipsoids drawn at the 50% probability level.

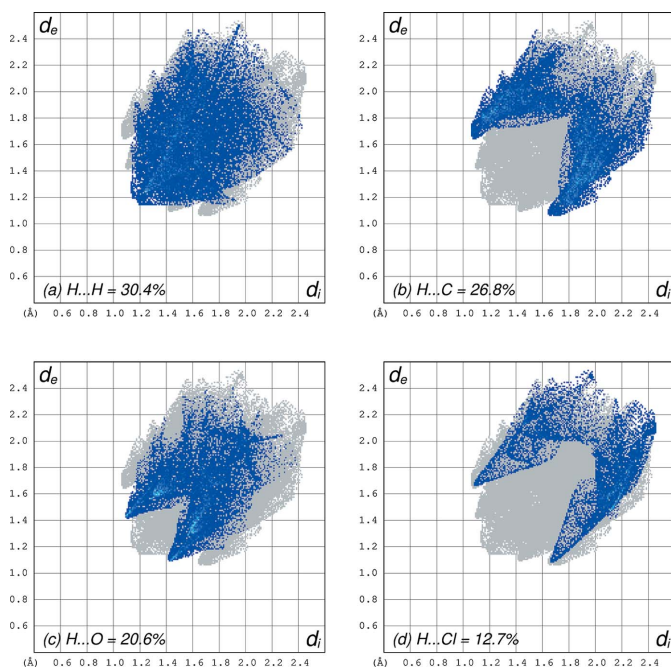


Figure 2

Hirshfeld surface fingerprint plots showing (a) $H\cdots H$ contacts = 30.4% coverage, (b) $H\cdots C$ = 26.8%, (c) $H\cdots O$ = 20.6% and (d) $H\cdots Cl$ = 12.7%. Reciprocal contacts are included in the coverage fractions. Other contact types were unremarkable.

$C-H$. The absence of any sharp spikes protruding to the lower left in the Hirshfeld surface fingerprint plots (Fig. 2) calculated using *CrystalExplorer* (Spackman *et al.*, 2021) clearly marks the absence of any very close contacts. Indeed, only two such contacts are flagged as intermolecular hydrogen-bond-type interactions by the default *SHELXL* HTAB command. These are $C5-H5A\cdots O3^i$ and $C5-H5B\cdots Cl1^{ii}$ [symmetry codes: (i) $x - \frac{1}{2}, -y + \frac{3}{2}, z$; (ii) $x - 1, y, z - 1$], shown in Table 1. The benzene rings of the 4-chlorophenyl and dihydrobenzodioxine groups, however, exhibit $\pi-\pi$ overlap, which link the molecules into chains that extend parallel to $[001]$, as shown in Fig. 3. The centroid-centroid distances [$3.747(18) \text{ \AA}$] and the dihedral angle between the

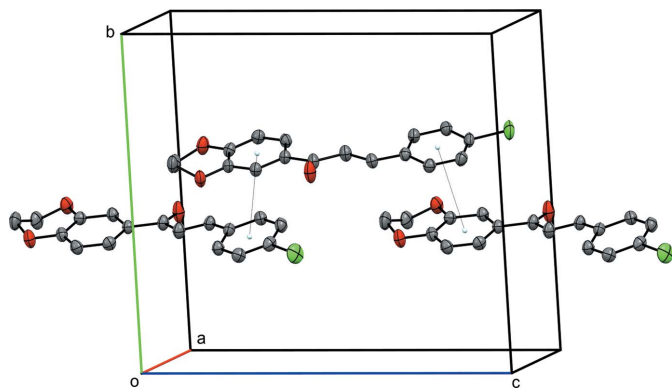


Figure 3

A partial packing plot of **I**, viewed approximately down the a axis. The stacking of the aromatic rings is shown by dotted lines between ring centroids, which lead to chains of molecules that extend parallel to $[001]$.

participating rings [6.12 (11)°] imply that these are rather weak interactions.

4. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.43 with updates to November 2022; Groom *et al.*, 2016) using a molecular fragment consisting of chalcone (C₆H₅C=OCH=CHC₆H₅) with no specific substituents returned 4725 hits. With hydrogen attached across the C=C double bond, the search gave 1200 hits. Of these entries, 155 had ‘any halogen’ at the equivalent of C15 in **I**, 62 of which had Cl. A search fragment without the halogen but with ‘any oxygen bound’ substituent at the equivalent of C4 and C7 produced 78 matches. Addition of the aforementioned halogen reduced this to 14 structures, seven of which were chlorides, but only five are unique. These are: BOJFIQ [(2*E*)-1-(1,3-benzodioxol-5-yl)-3-(4-chlorophenyl)prop-2-en-1-one; Jasinski *et al.*, 2008], FATFIR [(*E*)-3-(2,4-dichlorophenyl)-1-(3,4,5-trimethoxyphenyl)prop-2-en-1-one; Wu *et al.*, 2012], TICDIT [3-(4-chlorophenyl)-1-(3,4-dimethoxyphenyl)prop-2-en-1-one; Teh *et al.*, 2007], VIDIW [3-(2,4-dichlorophenyl)-1-(3,4-dimethoxyphenyl)prop-2-en-1-one; Ng *et al.*, 2007] and XOLLOC [3-(4-chloro-3-fluorophenyl)-1-(3,4-dimethoxyphenyl)prop-2-en-1-one; Çelikesir *et al.*, 2019]. A few other related chalcone structures include: QERYOC [(2*E*)-3-(1,3-benzodioxol-5-yl)-1-(4-bromophenyl)prop-2-en-1-one; Harrison *et al.*, 2006], KUYWOR [(2*E*)-3-(1,3-benzodioxol-5-yl)-1-(3-bromo-2-thienyl)prop-2-en-1-one; Harrison *et al.*, 2010], TUNTAY [(2*E*)-1-(1,3-benzodioxol-5-yl)-3-(2-bromophenyl)prop-2-en-1-one; Li *et al.*, 2010], and UNUZUZ [(*E*)-3-(8-benzyloxy-2,3-dihydro-1,4-benzodioxin-6-yl)-1-[2-hydroxy-4,6-bis(methoxymethoxy)phenyl]prop-2-en-1-one; Zhang *et al.*, 2011].

5. Synthesis, crystallization, and spectroscopic data

1-(2,3-Dihydrobenzo[1,4]dioxin-6-yl)ethanone was prepared by Friedel–Crafts acylation of benzo[1,4]dioxane using ether

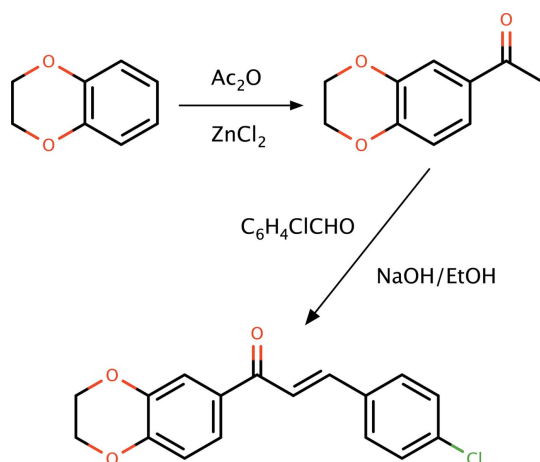


Figure 4
A generalized reaction scheme for the synthesis of **I**.

Table 2
Experimental details.

Crystal data	
Chemical formula	C ₁₇ H ₁₃ ClO ₃
<i>M_r</i>	300.72
Crystal system, space group	Orthorhombic, <i>Pna</i> 2 ₁
Temperature (K)	200
<i>a</i> , <i>b</i> , <i>c</i> (Å)	5.8655 (5), 14.3499 (17), 16.4803 (19)
<i>V</i> (Å ³)	1387.1 (3)
<i>Z</i>	4
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	0.28
Crystal size (mm)	0.26 × 0.23 × 0.22
Data collection	
Diffractometer	Bruker D8 Venture dual source
Absorption correction	Multi-scan (<i>SADABS</i> ; Krause <i>et al.</i> , 2015)
<i>T_{min}</i> , <i>T_{max}</i>	0.788, 0.971
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	17057, 2896, 2694
<i>R_{int}</i>	0.038
(sin θ/λ) _{max} (Å ⁻¹)	0.651
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.033, 0.083, 1.07
No. of reflections	2896
No. of parameters	190
No. of restraints	1
H-atom treatment	H-atom parameters constrained
Δρ _{max} , Δρ _{min} (e Å ⁻³)	0.21, −0.16
Absolute structure	Flack <i>x</i> determined using 1035 quotients [(<i>I</i> ⁺) − (<i>I</i> [−])] / [(<i>I</i> ⁺) + (<i>I</i> [−])] (Parsons <i>et al.</i> , 2013)
Absolute structure parameter	0.01 (2)

Computer programs: *APEX3* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2019* (Sheldrick, 2015*b*), *Mercury* (Macrae *et al.*, 2020), *SHELX* (Sheldrick, 2008) and *publCIF* (Westrip, 2010).

as solvent (Fig. 4). 1-(2,3-Dihydrobenzo[1,4]dioxin-6-yl)ethanone (1.78 g, 0.01 mol) and 4-chlorobenzaldehyde (1.40 g, 0.01 mol) were stirred in a 30% ethanolic NaOH and water mixture at 293 K for 4–6 h. The reaction mixture was refrigerated overnight. The precipitate that formed was filtered off and dried. X-ray-quality crystals were obtained from a solvent mixture of DMF–DMSO (dimethylformamide–dimethyl sulfide) (1:1 *v/v*) and the corresponding melting point was found to be 410–411 K.

FT-IR (*ν* in cm⁻¹): 3156–2934 (Ar-CH), 1655 (C=O), 1575 (C=C); ¹H NMR (CDCl₃, 400 MHz): δ 7.71 (*d*, 1H, β-CH), 7.92 (*d*, 2H, Ar-H), 7.62 (*d*, 2H, Ar-H), 7.23 (*d*, 1H, 2,3-dihydrobenzo[1,4]dioxine Ar-H), 7.21 (*d*, 1H, 2,3-dihydrobenzo[1,4]dioxine Ar-H), 7.01 (*d*, 1H, α-CH), 6.93 (*s*, 1H, 2,3-dihydrobenzo[1,4]dioxine Ar-H), 4.14 (*m*, 4H, 1,4-dioxane CH₂); ¹³C NMR (CDCl₃, 100 MHz): δ 196.1, 156.5, 149.8, 145.1, 133.5, 133.3, 129.0, 128.7, 122.1, 121.4, 121.3, 112.0, 106.8, 64.2.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All H atoms were found in difference Fourier maps and subsequently included in the

refinement using riding models, with constrained distances set to 0.95 [$C(sp^2)H$] and 0.99 Å (R_2CH_2). $U_{iso}(H)$ values were set to $1.2U_{eq}$ of the attached atom. The absolute structure was determined using 1035 quotients of the form $[(I^+) - (I^-)] / [(I^+) + (I^-)]$ (Parsons *et al.*, 2013).

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Computing details

Data collection: *APEX3* (Bruker, 2016); cell refinement: *APEX3* (Bruker, 2016); data reduction: *APEX3* (Bruker, 2016); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2019* (Sheldrick, 2015b); molecular graphics: *Mercury* (Macrae *et al.*, 2020); software used to prepare material for publication: *SHELX* (Sheldrick, 2008) and *pubCIF* (Westrip, 2010).

(*E*)-3-(4-Chlorophenyl)-1-(2,3-dihydrobenzo[*b*][1,4]dioxin-6-yl)prop-2-en-1-one

Crystal data

$C_{17}H_{13}ClO_3$

$M_r = 300.72$

Orthorhombic, *Pna*2₁

$a = 5.8655$ (5) Å

$b = 14.3499$ (17) Å

$c = 16.4803$ (19) Å

$V = 1387.1$ (3) Å³

$Z = 4$

$F(000) = 624$

$D_x = 1.440$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 9943 reflections

$\theta = 2.5$ – 27.6°

$\mu = 0.28$ mm⁻¹

$T = 200$ K

Cut block, pale yellow

$0.26 \times 0.23 \times 0.22$ mm

Data collection

Bruker D8 Venture dual source diffractometer

Radiation source: microsource

Detector resolution: 7.41 pixels mm⁻¹

φ and ω scans

Absorption correction: multi-scan (*SADABS*; Krause *et al.*, 2015)

$T_{\min} = 0.788$, $T_{\max} = 0.971$

17057 measured reflections

2896 independent reflections

2694 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.038$

$\theta_{\max} = 27.6^\circ$, $\theta_{\min} = 2.5^\circ$

$h = -7 \rightarrow 7$

$k = -18 \rightarrow 18$

$l = -19 \rightarrow 21$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.033$

$wR(F^2) = 0.083$

$S = 1.07$

2896 reflections

190 parameters

1 restraint

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: difference Fourier map

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0322P)^2 + 0.4666P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$$\Delta\rho_{\max} = 0.21 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.16 \text{ e } \text{\AA}^{-3}$$

Absolute structure: Flack x determined using
1035 quotients [(I+)-(I-)]/[(I+)+(I-)] [Parsons *et al.* (2013)]
Absolute structure parameter: 0.01 (2)

Special details

Experimental. The crystal was mounted using polyisobutene oil on the tip of a fine glass fibre, which was fastened in a copper mounting pin with electrical solder. It was placed directly into the cold gas stream of a liquid-nitrogen based cryostat (Hope, 1994; Parkin & Hope, 1998).

The crystals appeared to undergo a destructive phase transition when cooled to 90K. Visual inspection of crystal integrity and diffraction quality vs temperature established a safe temperature for data collection of -73° C.

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 progress was checked using *Platon* (Spek, 2020) and by an *R*-tensor (Parkin, 2000). The final model was further checked with the IUCr utility *checkCIF*.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.86056 (16)	0.65710 (6)	0.91506 (5)	0.0577 (3)
O1	-0.0096 (3)	0.59370 (18)	0.48876 (12)	0.0505 (6)
O2	0.0030 (3)	0.57318 (14)	0.18914 (12)	0.0416 (5)
O3	0.4399 (4)	0.64129 (16)	0.14256 (12)	0.0443 (5)
C1	0.1903 (5)	0.6112 (2)	0.47247 (15)	0.0338 (6)
C2	0.2634 (4)	0.61897 (18)	0.38646 (15)	0.0286 (5)
C3	0.1080 (4)	0.59307 (18)	0.32641 (17)	0.0318 (5)
H3	-0.038161	0.570520	0.341626	0.038*
C4	0.1637 (4)	0.59976 (17)	0.24503 (16)	0.0300 (5)
C5	0.0414 (6)	0.6143 (2)	0.11107 (17)	0.0453 (7)
H5A	0.008461	0.681919	0.113519	0.054*
H5B	-0.062554	0.585853	0.070731	0.054*
C6	0.2829 (6)	0.5995 (2)	0.08557 (18)	0.0475 (8)
H6A	0.313824	0.531780	0.081738	0.057*
H6B	0.306758	0.627076	0.031178	0.057*
C7	0.3778 (5)	0.63300 (19)	0.22209 (16)	0.0320 (6)
C8	0.5313 (5)	0.66010 (18)	0.28102 (18)	0.0343 (6)
H8	0.675979	0.683954	0.265581	0.041*
C9	0.4761 (5)	0.65280 (18)	0.36243 (17)	0.0329 (6)
H9	0.584046	0.671004	0.402365	0.040*
C10	0.3628 (5)	0.62304 (19)	0.53720 (16)	0.0361 (6)
H10	0.511960	0.643759	0.523877	0.043*
C11	0.3090 (5)	0.60465 (18)	0.61436 (15)	0.0314 (6)
H11	0.158246	0.583195	0.624038	0.038*
C12	0.4563 (4)	0.61380 (17)	0.68570 (15)	0.0297 (5)
C13	0.6738 (4)	0.65334 (18)	0.68259 (17)	0.0326 (5)
H13	0.737075	0.671519	0.631943	0.039*
C14	0.7979 (5)	0.66609 (18)	0.75398 (18)	0.0347 (6)

H14	0.945533	0.693264	0.752293	0.042*
C15	0.7047 (5)	0.63898 (18)	0.82670 (18)	0.0355 (6)
C16	0.4927 (5)	0.59816 (19)	0.83158 (18)	0.0367 (6)
H16	0.432553	0.578743	0.882366	0.044*
C17	0.3696 (5)	0.58612 (18)	0.76102 (16)	0.0330 (6)
H17	0.222733	0.558409	0.763644	0.040*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0663 (5)	0.0745 (5)	0.0324 (4)	−0.0046 (4)	−0.0248 (4)	0.0048 (4)
O1	0.0324 (10)	0.0939 (18)	0.0252 (10)	−0.0057 (11)	0.0000 (9)	0.0001 (10)
O2	0.0414 (11)	0.0586 (12)	0.0248 (9)	−0.0105 (10)	−0.0082 (9)	0.0018 (9)
O3	0.0440 (11)	0.0634 (14)	0.0254 (10)	−0.0025 (10)	0.0040 (9)	0.0059 (9)
C1	0.0345 (14)	0.0442 (15)	0.0226 (13)	0.0005 (12)	−0.0028 (11)	−0.0017 (12)
C2	0.0283 (12)	0.0340 (13)	0.0234 (12)	0.0011 (10)	−0.001 (1)	0.0006 (10)
C3	0.0312 (12)	0.0381 (13)	0.0261 (13)	−0.0009 (10)	−0.0011 (11)	0.0015 (11)
C4	0.0313 (13)	0.0327 (12)	0.0259 (12)	−0.0001 (11)	−0.0058 (11)	0.0007 (10)
C5	0.0526 (18)	0.0612 (18)	0.0221 (13)	−0.0025 (15)	−0.0101 (13)	0.0041 (13)
C6	0.0591 (19)	0.062 (2)	0.0217 (13)	0.0047 (16)	−0.0007 (14)	−0.0005 (13)
C7	0.0326 (14)	0.0374 (13)	0.0259 (13)	0.0032 (11)	0.0014 (11)	0.004 (1)
C8	0.0288 (13)	0.0410 (14)	0.0330 (14)	−0.0026 (11)	0.0014 (12)	0.0034 (12)
C9	0.0303 (12)	0.0380 (14)	0.0305 (13)	−0.0033 (10)	−0.0059 (11)	−0.0012 (11)
C10	0.0326 (13)	0.0489 (16)	0.0268 (14)	−0.0015 (13)	−0.0020 (11)	−0.0015 (13)
C11	0.0291 (13)	0.0403 (14)	0.0247 (13)	−0.0004 (11)	−0.0012 (10)	−0.0031 (11)
C12	0.0316 (13)	0.0330 (13)	0.0246 (13)	0.0023 (10)	0.0005 (11)	−0.0017 (11)
C13	0.0324 (13)	0.0396 (13)	0.0259 (13)	−0.0005 (10)	−0.0024 (11)	0.0010 (11)
C14	0.0321 (13)	0.0387 (14)	0.0332 (14)	0.0003 (11)	−0.0069 (12)	0.0010 (12)
C15	0.0416 (15)	0.0393 (13)	0.0255 (12)	0.0060 (11)	−0.0117 (12)	0.0000 (11)
C16	0.0430 (15)	0.0416 (14)	0.0256 (13)	0.0023 (12)	−0.0005 (12)	0.0033 (12)
C17	0.0337 (13)	0.0362 (13)	0.0292 (14)	−0.0005 (10)	0.0021 (11)	0.0013 (11)

Geometric parameters (Å, °)

C11—C15	1.739 (3)	C7—C8	1.380 (4)
O1—C1	1.229 (3)	C8—C9	1.384 (4)
O2—C4	1.372 (3)	C8—H8	0.9500
O2—C5	1.433 (3)	C9—H9	0.9500
O3—C7	1.366 (3)	C10—C11	1.336 (4)
O3—C6	1.446 (4)	C10—H10	0.9500
C1—C10	1.480 (4)	C11—C12	1.465 (4)
C1—C2	1.485 (3)	C11—H11	0.9500
C2—C3	1.396 (4)	C12—C13	1.397 (4)
C2—C9	1.396 (4)	C12—C17	1.399 (4)
C3—C4	1.384 (4)	C13—C14	1.396 (4)
C3—H3	0.9500	C13—H13	0.9500
C4—C7	1.395 (4)	C14—C15	1.373 (4)
C5—C6	1.493 (5)	C14—H14	0.9500

C5—H5A	0.9900	C15—C16	1.377 (4)
C5—H5B	0.9900	C16—C17	1.379 (4)
C6—H6A	0.9900	C16—H16	0.9500
C6—H6B	0.9900	C17—H17	0.9500
C4—O2—C5	112.4 (2)	C7—C8—H8	119.7
C7—O3—C6	114.7 (2)	C9—C8—H8	119.7
O1—C1—C10	121.2 (2)	C8—C9—C2	120.7 (2)
O1—C1—C2	120.0 (2)	C8—C9—H9	119.7
C10—C1—C2	118.8 (2)	C2—C9—H9	119.7
C3—C2—C9	118.4 (2)	C11—C10—C1	120.1 (3)
C3—C2—C1	117.9 (2)	C11—C10—H10	119.9
C9—C2—C1	123.7 (2)	C1—C10—H10	119.9
C4—C3—C2	121.0 (2)	C10—C11—C12	127.4 (3)
C4—C3—H3	119.5	C10—C11—H11	116.3
C2—C3—H3	119.5	C12—C11—H11	116.3
O2—C4—C3	118.0 (2)	C13—C12—C17	118.7 (2)
O2—C4—C7	122.1 (3)	C13—C12—C11	123.0 (2)
C3—C4—C7	119.9 (2)	C17—C12—C11	118.2 (2)
O2—C5—C6	110.0 (3)	C14—C13—C12	119.9 (3)
O2—C5—H5A	109.7	C14—C13—H13	120.0
C6—C5—H5A	109.7	C12—C13—H13	120.0
O2—C5—H5B	109.7	C15—C14—C13	119.4 (3)
C6—C5—H5B	109.7	C15—C14—H14	120.3
H5A—C5—H5B	108.2	C13—C14—H14	120.3
O3—C6—C5	111.2 (2)	C14—C15—C16	122.1 (3)
O3—C6—H6A	109.4	C14—C15—C11	118.6 (2)
C5—C6—H6A	109.4	C16—C15—C11	119.3 (2)
O3—C6—H6B	109.4	C15—C16—C17	118.5 (3)
C5—C6—H6B	109.4	C15—C16—H16	120.8
H6A—C6—H6B	108.0	C17—C16—H16	120.8
O3—C7—C8	118.5 (3)	C16—C17—C12	121.5 (3)
O3—C7—C4	122.0 (3)	C16—C17—H17	119.3
C8—C7—C4	119.5 (3)	C12—C17—H17	119.3
C7—C8—C9	120.5 (2)		
O1—C1—C2—C3	-8.9 (4)	C4—C7—C8—C9	1.3 (4)
C10—C1—C2—C3	169.9 (2)	C7—C8—C9—C2	-0.8 (4)
O1—C1—C2—C9	169.5 (3)	C3—C2—C9—C8	-0.2 (4)
C10—C1—C2—C9	-11.7 (4)	C1—C2—C9—C8	-178.6 (3)
C9—C2—C3—C4	0.7 (4)	O1—C1—C10—C11	7.4 (4)
C1—C2—C3—C4	179.1 (2)	C2—C1—C10—C11	-171.3 (3)
C5—O2—C4—C3	158.0 (3)	C1—C10—C11—C12	-179.0 (2)
C5—O2—C4—C7	-21.8 (4)	C10—C11—C12—C13	6.8 (4)
C2—C3—C4—O2	-180.0 (2)	C10—C11—C12—C17	-176.7 (3)
C2—C3—C4—C7	-0.1 (4)	C17—C12—C13—C14	-1.2 (4)
C4—O2—C5—C6	50.4 (3)	C11—C12—C13—C14	175.3 (2)
C7—O3—C6—C5	38.6 (4)	C12—C13—C14—C15	0.4 (4)

O2—C5—C6—O3	-60.1 (4)	C13—C14—C15—C16	0.9 (4)
C6—O3—C7—C8	171.8 (3)	C13—C14—C15—C11	-179.0 (2)
C6—O3—C7—C4	-9.4 (4)	C14—C15—C16—C17	-1.3 (4)
O2—C4—C7—O3	0.2 (4)	C11—C15—C16—C17	178.7 (2)
C3—C4—C7—O3	-179.6 (3)	C15—C16—C17—C12	0.4 (4)
O2—C4—C7—C8	179.0 (2)	C13—C12—C17—C16	0.8 (4)
C3—C4—C7—C8	-0.8 (4)	C11—C12—C17—C16	-175.8 (2)
O3—C7—C8—C9	-179.9 (3)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C5—H5 <i>A</i> \cdots O3 ⁱ	0.99	2.61	3.595 (4)	172
C5—H5 <i>B</i> \cdots C11 ⁱⁱ	0.99	2.80	3.455 (3)	124

Symmetry codes: (i) $x-1/2, -y+3/2, z$; (ii) $x-1, y, z-1$.