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Investigations of new potential photo-acid generators: crystal structures of 2-[(*E*)-2-phenylethenyl]phenol (orthorhombic polymorph) and (2*E*)-3-(2-bromophenyl)-2-phenylprop-2-enoic acid

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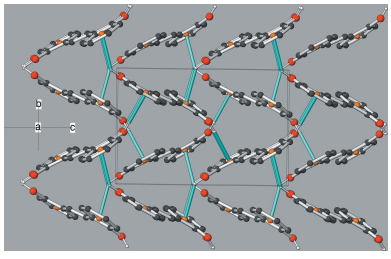
The title compounds, $C_{14}H_{12}O$, (I), and $C_{15}H_{11}BrO_2$, (II), were prepared and characterized as part of our studies of potential new photo-acid generators. In (I), which crystallizes in the orthorhombic space group $Pca2_1$, compared to $P2_1/n$ for the previously known monoclinic polymorph [Cornella & Martin (2013). *Org. Lett.* **15**, 6298–6301], the dihedral angle between the aromatic rings is 4.35 (6)° and the OH group is disordered over two sites in a 0.795 (3):0.205 (3) ratio. In the crystal of (I), molecules are linked by O—H···π interactions involving both the major and minor –OH disorder components, generating [001] chains as part of the herringbone packing motif. The asymmetric unit of (II) contains two molecules with similar conformations (weighted r.m.s. overlay fit = 0.183 Å). In the crystal of (II), both molecules form carboxylate inversion dimers linked by pairs of O—H···O hydrogen bonds, generating $R_2^2(8)$ loops in each case. The dimers are linked by pairs of C—H···O hydrogen bonds to form [010] chains.

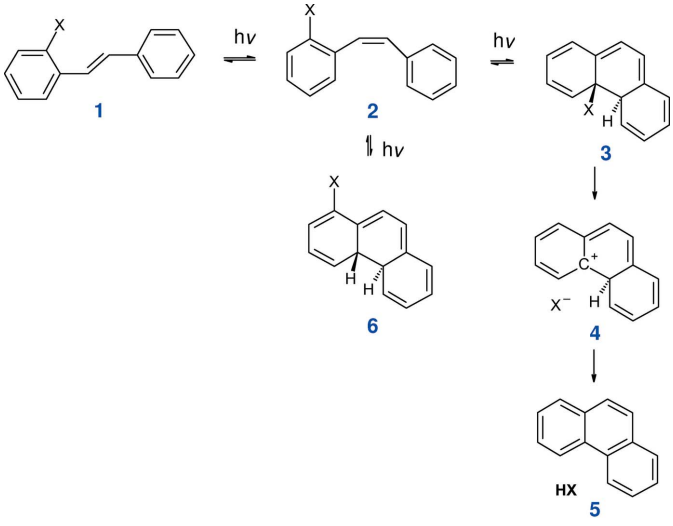
1. Chemical context

Photo-acid generators can be used as additives for creating patterns in a polymer film by irradiation through a mask followed by thermal development and base treatment (Ayothi *et al.*, 2007; Kudo *et al.*, 2008; Steidl *et al.*, 2009). The UV irradiation degrades a small amount of the photo-acid generator in exposed areas, which releases a catalytic amount of a strong acid (commonly triflic acid). This acid subsequently catalyses the degradation of the *tert*-butylcarboxylate groups of a polymer film in a thermal development step, releasing carboxylic acid groups and isobutene. Treatment with base then solubilizes and removes the degraded polymer film in exposed areas, thereby creating a positive resist image (Ito *et al.*, 1994).

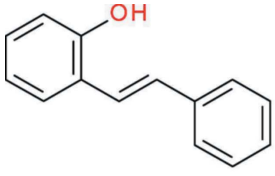
We are exploring new types of organic structures as potential photo-acid generators, which might offer improvements over existing substances. Scheme 1 shows how substituted *trans*-stilbenes might act as photo-acid generators *via* sequential photochemical *trans–cis* isomerization and ring-closing reactions. It should be noted that the photochemical cyclization of stilbenes to phenanthenes in the presence of a hydrogen acceptor such as iodine or propylene oxide is well known (Mallory & Mallory, 2005). However, in the absence of an oxidant, if a leaving group is present at the ring-closure site, as in structure **3**, a rapid elimination of HX (structure **5**) might



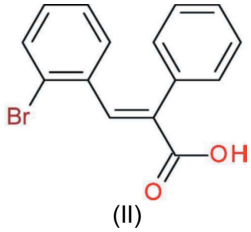
occur *via* a stabilized carbocation intermediate **4**. In the absence of an oxidant, the cyclized dihydro-phenanthrene compound **6** will equilibrate back to *cis*-stilbene **2**. Stilbenes can also undergo $2\pi + 2\pi$ photochemical cycloadditions (Fulton & Dunitz, 1947; Shechter *et al.*, 1963), a possible competing reaction, but the molecular structures and morphology may still favour the desired reaction to proceed in a thin film.



As part of these studies, the syntheses and crystal structures of the title substituted stilbenes, (I) and (II), are now described [compound (II) could also be described as a cinnamic acid derivative: the photochemical reactions of this family of compounds were reported by Schmidt (1971)]. Compound (I) is an intermediate in the synthesis, whereas a close analogue of compound (II) has already been shown to undergo photochemical cyclization to a phenanthrene with concomitant release of HCl (Geirsson & Kvaran, 2001). A monoclinic polymorph (space group $P2_1/n$) of (I) was reported recently (Cornella & Martin, 2013) although its crystal structure was not described in detail.



(I)



(II)

2. Structural commentary

Compound (I) comprises one molecule in the asymmetric unit (Fig. 1), with the –OH group disordered over two sites in a 0.795 (3):0.205 (3) ratio. For the major disorder component, the $C_{ar}-C_{ar}-O-H$ (ar = aromatic) torsion angle is 172°. The molecule is close to planar and the dihedral angle between the aromatic rings is 4.35 (6)°. The bond lengths of the central unit [C6—C7 = 1.4703 (19); C7—C8 = 1.3407 (16); C8—C9 = 1.4720 (18) Å] are consistent with data from previous studies

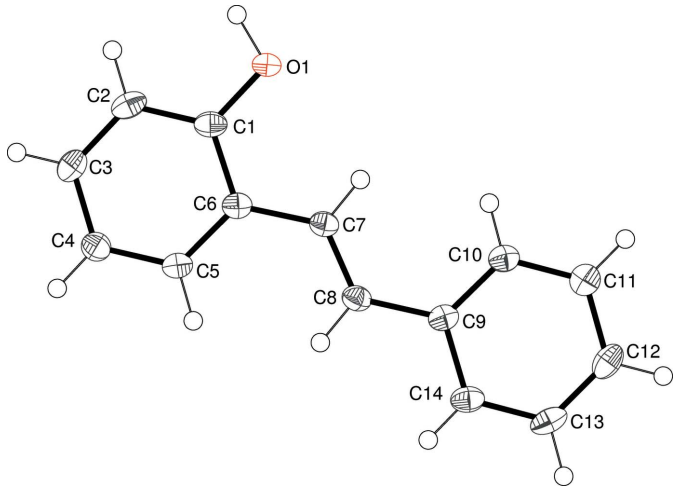


Figure 1
The asymmetric unit of (I), showing 50% displacement ellipsoids. Only the major disordered component for the OH group is shown (the minor component is attached to C14).

of similar compounds (Tirado-Rives *et al.*, 1984; Jungk *et al.*, 1984). In the monoclinic polymorph of (I) (Cornella & Martin, 2013), the asymmetric unit consists of a half-molecule, which is completed by crystallographic inversion symmetry and therefore, of course, the aromatic rings are exactly coplanar: the OH group is statistically disordered by symmetry and the corresponding C—C—O—H torsion angle for the monoclinic phase is −175°.

There are two molecules in the asymmetric unit of (II) (Fig. 2). In the first (C1) molecule, the dihedral angles between the carboxylic acid group and the phenyl and bromobenzene rings are 61.52 (6) and 55.43 (5)°, respectively; the dihedral angle between the aromatic rings is 54.45 (5)°. The equivalent data for the second (C16) molecule are 50.72 (6), 60.28 (5) and 61.48 (6)°, respectively. The C1 and C16 molecules have a similar overall conformation with an r.m.s. deviation of 0.183 Å for the overlay fit for all non-hydrogen atoms. Otherwise, their bond lengths and bond angles are unexceptional and fall within the expected range of values.

3. Supramolecular features

The crystal of (I) features O—H$\cdots\pi$ interactions as the main supramolecular interaction (Table 1). The major disorder component (O1—H1*O*) generates [001] zigzag chains, as seen

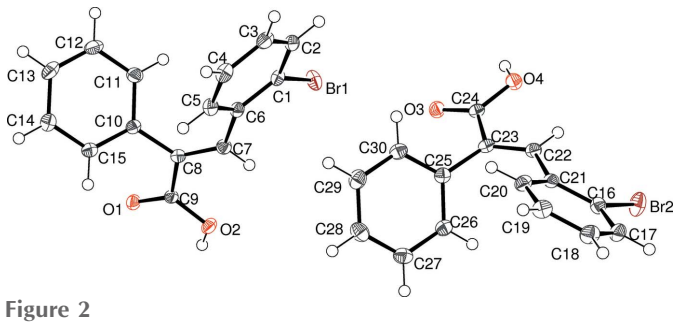


Figure 2
The asymmetric unit of (II), showing 50% displacement ellipsoids.

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

*Cg*1 and *Cg*2 are the centroids of rings C1–C6 and C9–C14, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1o···*Cg*2^{i}	0.98	2.66	3.5028 (13)	144
O2—H2o···*Cg*1	0.91	2.74	3.646 (2)	179
C5—H5···*Cg*2^{ii}	0.95	2.86	3.5337 (12)	129
C10—H10···*Cg*1^{iii}	0.95	2.87	3.5742 (14)	132
C13—H13···*Cg*1^{iv}	0.95	2.87	3.6015 (14)	135

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

Table 2
Hydrogen-bond geometry (Å, °) for (II).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*O*···O1^{i}	0.84 (2)	1.80 (2)	2.6402 (16)	174 (2)
O4—H4*O*···O3^{ii}	0.81 (2)	1.84 (2)	2.6478 (16)	178 (2)
C5—H5···O3^{iii}	0.95	2.42	3.323 (2)	158
C20—H20···O1^{iii}	0.95	2.52	3.3072 (19)	141

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

in Fig. 3. The minor disorder component (O2—H2*O*) also forms [001] chains. There are also some possible very weak C—H···π interactions. The packing can be described as herringbone when viewed down [100] (Fig. 4). The monoclinic polymorph (Cornella & Martin, 2013) also features supramolecular chains with the molecules linked by O—H···π interactions but a different overall herringbone packing motif (Fig. 5).

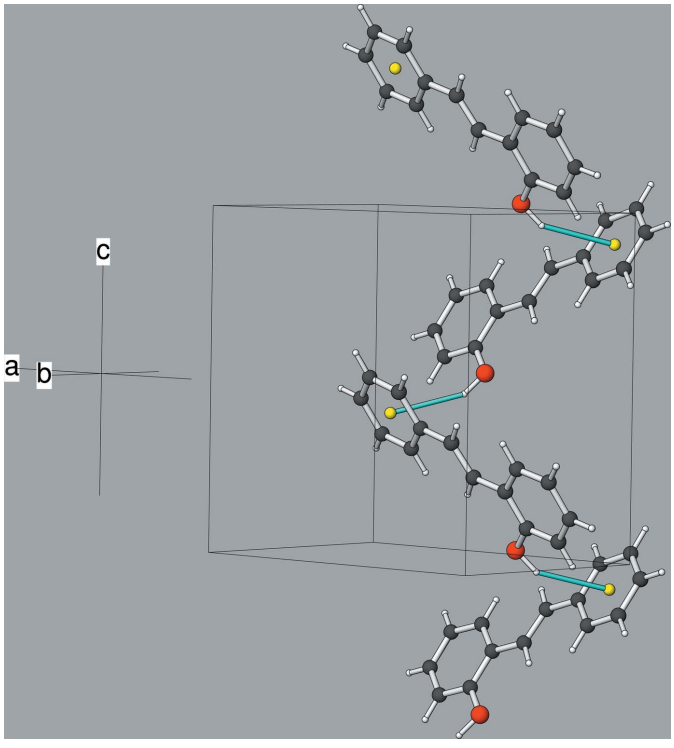


Figure 3
Part of a [001] chain of molecules in the crystal of (I), connected by O—H···π interactions (cyan lines).

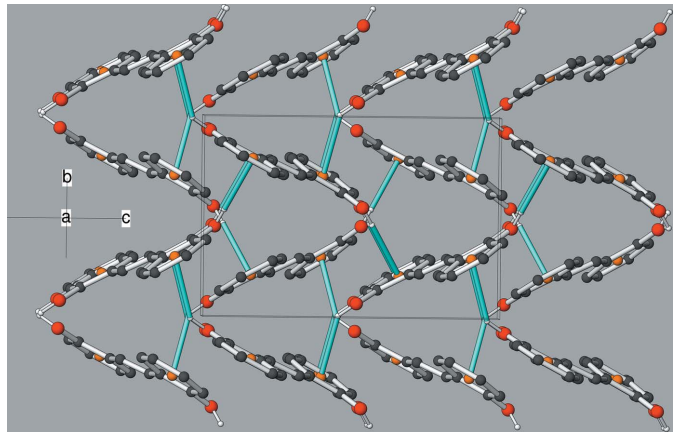


Figure 4
The unit-cell packing in (I), viewed approximately down [100]. The O—H···π interactions from both disordered components are shown as cyan lines.

In the crystal of (II), both molecules (*A* and *B*) form carboxylic acid inversion dimers linked by pairs of O—H···O hydrogen bonds (Table 2), which generate $R_2^2(8)$ loops in each case. The (*A* + *A*) and (*B* + *B*) dimers are in turn linked by pairs of C—H···O hydrogen bonds to generate [010] chains (Figs. 6 and 7). This hydrogen-bond scheme is 'balanced,' with both O1 and O3 accepting one O—H···O and one C—H···O hydrogen bond. The shortest Br···Br contact distance of 3.6504 (4) Å in the crystal of (II) is slightly shorter than the van der Waals radius sum of 3.70 Å for two Br atoms (Bondi, 1964).

4. Database survey

A survey of the Cambridge Structural Database (Groom & Allen, 2014) (entries updated to 22 December 2015) revealed ten crystal structures of *E*-2-hydroxy stilbenes with different substituents including (*E*)-1,2-bis(2-hydroxyphenyl)ethene (refcode CEYKUM; Tirado-Rives *et al.*, 1984), in which the molecules are linked by O—H···O hydrogen bonds. Two substituted *Z*-isomers are also known. A total of 28 analogues

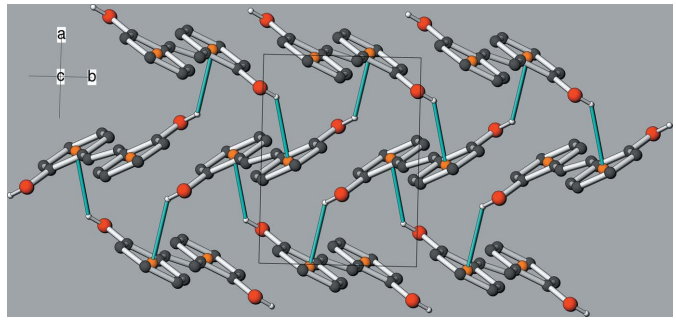


Figure 5
The unit-cell packing in the monoclinic polymorph of $C_{14}H_{12}O$, viewed approximately down [000] (data from Cornella & Martin, 2013). The O—H···π interactions are shown as cyan lines.

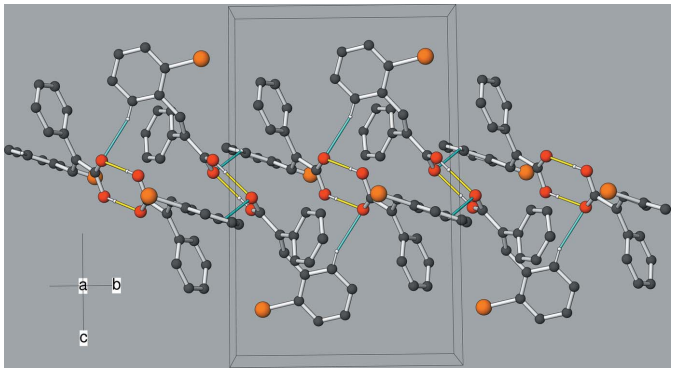


Figure 6
Part of a [010] chain in the crystal of (II), with O—H···O hydrogen bonds shown as yellow lines and C—H···O hydrogen bonds shown as cyan lines.

of (II) with different substituents to the aromatic rings were found in the same survey, including the parent compound, 2,3-diphenylacrylic acid (refcode OJOFEZ; Fujihara *et al.*, 2011).

5. Synthesis and crystallization

Salicylaldehyde (0.2 g, 1.64 mmol) and benzyltriphenylphosphonium bromide (1.0 g, 2.31 mmol) in dry dimethylformamide (DMF) (30 ml) were treated with sodium methoxide powder (0.2 g, 3.70 mmol) and refluxed for 4 h (Mylona *et al.*, 1986). The reaction mixture was then cooled, acidified with dilute aqueous HCl and extracted into CH_2Cl_2.

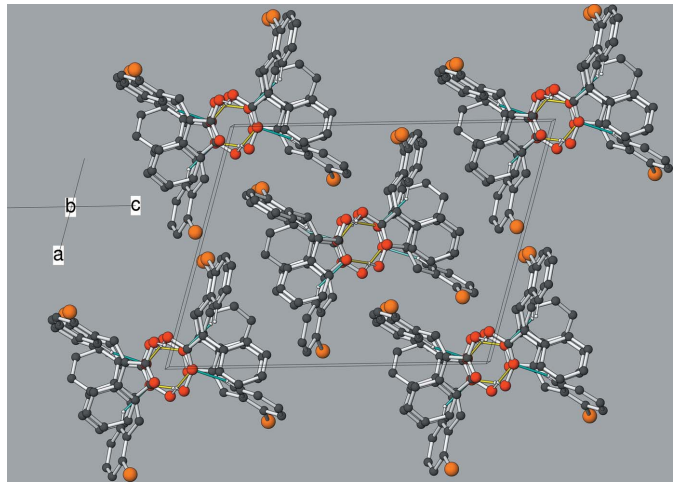


Figure 7
The unit-cell packing in (II), viewed approximately down [010].

The organic layer was washed twice with water to remove DMF, dried over Na_2SO_4, concentrated *in vacuo* and purified by flash chromatography on silica gel. Hexane–diethyl ether (50:50) eluted the title compound (52 mg, 16%) as a white solid (m.p. 418–419 K), which was recrystallized from hexane/diethyl ether solution to yield colourless slabs of (I); *m*/*z* 196.0886 (M^+) $C_{14}H_{12}O$ requires 196.0883. UV $\lambda_{max}(CHCl_3)$/nm 230 (log ε 4.30), 288 (4.39) and 315 (4.40). IR (ν_{max}/cm^{-1}) 3528*s*, 3019*w*, 2923*w*, 2852*w*, 1585*s*, 1498*s*, 1454*s*,

Table 3
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	$C_{14}H_{12}O$	$C_{15}H_{11}BrO_2$
M_r	196.24	303.15
Crystal system, space group	Orthorhombic, $Pca2_1$	Monoclinic, $P2_1/n$
Temperature (K)	100	100
a, b, c (Å)	11.6193 (8), 7.6800 (5), 11.3584 (8)	13.890 (1), 10.9048 (8), 17.8121 (10)
α, β, γ (°)	90, 90, 90	90, 106.064 (1), 90
V (Å^3)	1013.58 (12)	2592.6 (3)
Z	4	8
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	0.08	3.16
Crystal size (mm)	0.27 × 0.16 × 0.04	0.19 × 0.07 × 0.07
Data collection		
Diffractometer	Rigaku CCD	Rigaku CCD
Absorption correction	–	Multi-scan (*SADABS*; Sheldrick, 2004)
T_{min}, T_{max}	–	0.585, 0.809
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6984, 2271, 2132	31964, 5922, 5297
R_{int}	0.031	0.035
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.649	0.650
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.034, 0.091, 1.06	0.025, 0.063, 1.04
No. of reflections	2271	5922
No. of parameters	146	331
No. of restraints	1	0
H-atom treatment	H-atom parameters constrained	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.19, −0.15	0.56, −0.74

Computer programs: *CrystalClear* (Rigaku, 2010), *SHELXS97* and *SHELXL97* (Sheldrick, 2008), *ORTEP-3 for Windows* (Farrugia, 2012) and *ATOMS* (Dowty, 1999).

1332*s*, 1249*s*, 1195*s*, 1088*s*, 974*vs*, 845*s*, 752*vs*, 724*vs*, 691*vs*, 507*vs*. ^{1}H NMR (400MHz, $CDCl_3$) δ 5.07 (1H, *s*), 6.79 (1H, *d*, *J* = 8.0), 6.95 (1H, *t*, *J* = 7.4), 7.14 (2H, *m*), 7.25 (1H, *t*, *J* = 6.3), 7.35 (3H, *m*), 7.52 (3H, *m*). ^{13}C NMR (99.5 MHz, $CDCl_3$) δ 116.1, 121.3, 123.1, 124.8, 126.7, 127.3, 127.7, 128.8, 130.3, 137.7 and 153.1 (one resonance is missing).

2-Bromobenzaldehyde (0.5 g, 2.70 mmol) and methyl phenylacetate (0.6 g, 4.0 mmol) in dry DMF (30 ml) were treated with sodium methoxide powder (0.3 g, 5.6 mmol) and refluxed for 4 h. The reaction mixture was then cooled, acidified with dilute aqueous HCl and extracted into CH_2Cl_2. The organic layer was washed twice with water to remove DMF, dried over Na_2SO_4, concentrated *in vacuo* and purified by flash chromatography on silica gel. Hexane–diethyl ether (75:25) eluted (II) (65 mg, 8%) as a colourless solid, which was recrystallized from hexane/diethyl ether solution as colourless rods. The starting ester was evidently hydrolysed either during the reaction or at the work-up stage; *m*/*z* 300.9866 (*M* + H) $C_{15}H_{10}O_2Br$ requires 300.9870.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. Atom H1*O* in (I) was located in a difference Fourier map and refined as riding in its as-found relative position with U_{iso}(H) = 1.2U_{eq}(O). The other H atoms were placed geometrically (C—H = 0.95 Å, O—H = 0.91 Å) and refined as riding atoms with U_{iso}(H) = 1.2U_{eq}(C,O). The O-bound H atoms in (II) were located in a difference Fourier map and refined with U_{iso}(H) = 1.2U_{eq}(O). The C-bound H atoms were placed geometrically (C—H = 0.95 Å) and refined as riding atoms with U_{iso}(H) = 1.2U_{eq}(C).

Acknowledgements

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supporting information

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Investigations of new potential photo-acid generators: crystal structures of 2-[(*E*)-2-phenylethenyl]phenol (orthorhombic polymorph) and (2*E*)-3-(2-bromophenyl)-2-phenylprop-2-enoic acid

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Computing details

For both compounds, data collection: *CrystalClear* (Rigaku, 2010); cell refinement: *CrystalClear* (Rigaku, 2010); data reduction: *CrystalClear* (Rigaku, 2010); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *ATOMS* (Dowty, 1999); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

(I) 2-[(*E*)-2-Phenylethenyl]phenol

Crystal data

$C_{14}H_{12}O$
M_r = 196.24
Orthorhombic, $Pca2_1$
Hall symbol: P 2c -2ac
a = 11.6193 (8) Å
b = 7.6800 (5) Å
c = 11.3584 (8) Å
V = 1013.58 (12) Å^3
Z = 4
$F(000)$ = 416
D_x = 1.286 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 7085 reflections
θ = 2.5–27.5°
μ = 0.08 mm^{-1}
T = 100 K
Slab, colourless
0.27 × 0.16 × 0.04 mm

Data collection

Rigaku CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
6984 measured reflections
2271 independent reflections
2132 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 27.5°, θ_{min} = 2.7°
h = −15→13
k = −9→8
l = −13→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.091
S = 1.06
2271 reflections
146 parameters
1 restraint
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

$w = 1/[\sigma^2(F_o^2) + (0.0581P)^2 + 0.0566P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.30887 (12)	0.13815 (16)	0.08810 (11)	0.0230 (3)	
H1	0.3652	0.0929	0.0386	0.028*	0.205 (3)
C2	0.19331 (12)	0.12759 (16)	0.05641 (11)	0.0258 (3)	
H2	0.1721	0.0723	−0.0152	0.031*	
C3	0.10941 (12)	0.19707 (16)	0.12852 (12)	0.0249 (3)	
H3	0.0307	0.1890	0.1067	0.030*	
C4	0.14037 (12)	0.27928 (17)	0.23349 (12)	0.0244 (3)	
H4	0.0829	0.3280	0.2831	0.029*	
C5	0.25559 (12)	0.28929 (14)	0.26490 (11)	0.0222 (3)	
H5	0.2759	0.3456	0.3364	0.027*	
C6	0.34307 (12)	0.21877 (16)	0.19426 (10)	0.0207 (3)	
C7	0.46529 (12)	0.22186 (16)	0.22775 (11)	0.0210 (3)	
H7	0.5184	0.1701	0.1746	0.025*	
C8	0.50832 (12)	0.29131 (15)	0.32669 (11)	0.0219 (3)	
H8	0.4550	0.3453	0.3786	0.026*	
C9	0.62995 (11)	0.29221 (15)	0.36292 (11)	0.0203 (3)	
C10	0.71722 (12)	0.21042 (16)	0.29798 (11)	0.0229 (3)	
H10	0.6982	0.1498	0.2277	0.027*	
C11	0.83100 (13)	0.21675 (16)	0.33495 (12)	0.0259 (3)	
H11	0.8892	0.1621	0.2893	0.031*	
C12	0.86041 (12)	0.30333 (17)	0.43927 (13)	0.0273 (3)	
H12	0.9383	0.3073	0.4646	0.033*	
C13	0.77504 (12)	0.38320 (16)	0.50516 (11)	0.0265 (3)	
H13	0.7943	0.4413	0.5763	0.032*	
C14	0.66125 (13)	0.37858 (16)	0.46739 (11)	0.0241 (3)	
H14	0.6045	0.4340	0.5121	0.029*	0.795 (3)
O1	0.39106 (10)	0.07105 (15)	0.01715 (10)	0.0261 (3)	0.795 (3)
H1O	0.3492	0.0078	−0.0443	0.031*	0.795 (3)
O2	0.5932 (4)	0.4523 (7)	0.5351 (5)	0.0305 (15)	0.205 (3)
H2O	0.6381	0.5365	0.5670	0.037*	0.205 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0315 (7)	0.0191 (5)	0.0183 (6)	−0.0012 (5)	0.0010 (5)	0.0000 (4)
C2	0.0358 (8)	0.0207 (6)	0.0208 (6)	−0.0060 (5)	−0.0057 (5)	0.0003 (5)
C3	0.0259 (7)	0.0222 (6)	0.0266 (7)	−0.0043 (5)	−0.0061 (5)	0.0043 (5)
C4	0.0255 (7)	0.0231 (6)	0.0246 (7)	0.0004 (5)	0.0012 (5)	0.0001 (5)
C5	0.0270 (6)	0.0207 (6)	0.0190 (6)	−0.0014 (5)	−0.0005 (5)	−0.0017 (5)
C6	0.0255 (6)	0.0171 (5)	0.0194 (6)	−0.0026 (5)	0.0011 (5)	0.0005 (4)
C7	0.0245 (6)	0.0204 (6)	0.0180 (6)	−0.0004 (5)	0.0025 (5)	−0.0005 (4)
C8	0.0240 (7)	0.0207 (6)	0.0208 (6)	−0.0026 (5)	0.0045 (5)	−0.0022 (4)
C9	0.0260 (7)	0.0182 (6)	0.0166 (6)	−0.0040 (5)	−0.0001 (5)	0.0025 (4)
C10	0.0275 (7)	0.0213 (6)	0.0199 (6)	−0.0030 (5)	0.0005 (5)	0.0003 (4)
C11	0.0276 (7)	0.0233 (6)	0.0269 (7)	−0.0001 (5)	−0.0015 (5)	0.0031 (5)
C12	0.0285 (7)	0.0238 (7)	0.0296 (8)	−0.0056 (5)	−0.0102 (6)	0.0072 (5)
C13	0.0388 (8)	0.0225 (6)	0.0184 (6)	−0.0084 (5)	−0.0072 (6)	0.0017 (5)
C14	0.0330 (7)	0.0207 (6)	0.0185 (6)	−0.0038 (5)	0.0014 (5)	0.0001 (5)
O1	0.0247 (6)	0.0328 (7)	0.0209 (6)	0.0027 (5)	−0.0004 (4)	−0.0101 (5)
O2	0.027 (3)	0.035 (3)	0.030 (3)	−0.004 (2)	0.003 (2)	−0.014 (2)

Geometric parameters (Å, º)

C1—O1	1.3517 (17)	C8—H8	0.9500
C1—C2	1.3925 (18)	C9—C10	1.4024 (17)
C1—C6	1.4126 (18)	C9—C14	1.4073 (18)
C1—H1	0.9300	C10—C11	1.388 (2)
C2—C3	1.381 (2)	C10—H10	0.9500
C2—H2	0.9500	C11—C12	1.401 (2)
C3—C4	1.3963 (19)	C11—H11	0.9500
C3—H3	0.9500	C12—C13	1.386 (2)
C4—C5	1.388 (2)	C12—H12	0.9500
C4—H4	0.9500	C13—C14	1.390 (2)
C5—C6	1.4036 (18)	C13—H13	0.9500
C5—H5	0.9500	C14—O2	1.240 (5)
C6—C7	1.4703 (19)	C14—H14	0.9340
C7—C8	1.3407 (16)	O1—H1O	0.9794
C7—H7	0.9500	O2—H2O	0.9057
C8—C9	1.4720 (18)		
O1—C1—C2	120.33 (12)	C7—C8—H8	116.8
O1—C1—C6	118.51 (12)	C9—C8—H8	116.8
C2—C1—C6	121.17 (12)	C10—C9—C14	117.89 (12)
O1—C1—H1	0.7	C10—C9—C8	123.03 (11)
C2—C1—H1	120.0	C14—C9—C8	119.08 (12)
C6—C1—H1	118.8	C11—C10—C9	120.92 (12)
C3—C2—C1	120.33 (11)	C11—C10—H10	119.5
C3—C2—H2	119.8	C9—C10—H10	119.5
C1—C2—H2	119.8	C10—C11—C12	120.32 (13)

C2—C3—C4	119.97 (13)	C10—C11—H11	119.8
C2—C3—H3	120.0	C12—C11—H11	119.8
C4—C3—H3	120.0	C13—C12—C11	119.49 (13)
C5—C4—C3	119.55 (13)	C13—C12—H12	120.3
C5—C4—H4	120.2	C11—C12—H12	120.3
C3—C4—H4	120.2	C12—C13—C14	120.18 (12)
C4—C5—C6	122.03 (12)	C12—C13—H13	119.9
C4—C5—H5	119.0	C14—C13—H13	119.9
C6—C5—H5	119.0	O2—C14—C13	113.8 (3)
C5—C6—C1	116.96 (12)	O2—C14—C9	125.0 (3)
C5—C6—C7	123.05 (11)	C13—C14—C9	121.19 (13)
C1—C6—C7	119.98 (11)	C13—C14—H14	119.4
C8—C7—C6	125.69 (12)	C9—C14—H14	119.4
C8—C7—H7	117.2	C1—O1—H1O	105.2
C6—C7—H7	117.2	C14—O2—H2O	101.9
C7—C8—C9	126.46 (12)		

O1—C1—C2—C3	−179.60 (12)	C7—C8—C9—C10	−3.09 (18)
C6—C1—C2—C3	0.32 (18)	C7—C8—C9—C14	176.96 (11)
C1—C2—C3—C4	0.36 (19)	C14—C9—C10—C11	−0.88 (17)
C2—C3—C4—C5	−0.47 (19)	C8—C9—C10—C11	179.16 (12)
C3—C4—C5—C6	−0.10 (19)	C9—C10—C11—C12	0.94 (19)
C4—C5—C6—C1	0.74 (17)	C10—C11—C12—C13	−0.22 (19)
C4—C5—C6—C7	−177.62 (12)	C11—C12—C13—C14	−0.54 (19)
O1—C1—C6—C5	179.07 (11)	C12—C13—C14—O2	178.9 (3)
C2—C1—C6—C5	−0.85 (17)	C12—C13—C14—C9	0.59 (19)
O1—C1—C6—C7	−2.52 (17)	C10—C9—C14—O2	−178.0 (3)
C2—C1—C6—C7	177.56 (11)	C8—C9—C14—O2	2.0 (4)
C5—C6—C7—C8	−0.48 (19)	C10—C9—C14—C13	0.12 (18)
C1—C6—C7—C8	−178.79 (11)	C8—C9—C14—C13	−179.92 (11)
C6—C7—C8—C9	178.55 (12)		

Hydrogen-bond geometry (Å, º)

Cg1 and Cg2 are the centroids of rings C1–C6 and C9–C14, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1o···*Cg*2^{i}	0.98	2.66	3.5028 (13)	144
O2—H2o···*Cg*1	0.91	2.74	3.646 (2)	179
C5—H5···*Cg*2^{ii}	0.95	2.86	3.5337 (12)	129
C10—H10···*Cg*1^{iii}	0.95	2.87	3.5742 (14)	132
C13—H13···*Cg*1^{iv}	0.95	2.87	3.6015 (14)	135

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

(II) (2*E*)-3-(2-Bromophenyl)-2-phenylprop-2-enoic acid

Crystal data

$C_{15}H_{11}BrO_2$	Monoclinic, $P2_1/n$
M_r = 303.15	Hall symbol: -P 2yn

a = 13.890 (1) Å
b = 10.9048 (8) Å
c = 17.8121 (10) Å
β = 106.064 (1)°
V = 2592.6 (3) Å^3
Z = 8
$F(000)$ = 1216
D_x = 1.553 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 30565 reflections
θ = 2.2–27.5°
μ = 3.16 mm^{-1}
T = 100 K
Rod, colourless
0.19 × 0.07 × 0.07 mm

Data collection

Rigaku CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.585, T_{max} = 0.809

31964 measured reflections
5922 independent reflections
5297 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 27.5°, θ_{min} = 2.2°
h = −17→18
k = −13→14
l = −22→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.063
S = 1.04
5922 reflections
331 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0335P)^2 + 1.1714P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.74 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.18672 (11)	0.20670 (14)	0.33555 (9)	0.0171 (3)
C2	0.21064 (12)	0.11955 (16)	0.39459 (9)	0.0208 (3)
H2	0.2476	0.1419	0.4461	0.025*
C3	0.17974 (12)	−0.00082 (16)	0.37725 (10)	0.0228 (3)
H3	0.1954	−0.0614	0.4171	0.027*
C4	0.12604 (12)	−0.03303 (16)	0.30185 (10)	0.0223 (3)
H4	0.1064	−0.1158	0.2899	0.027*
C5	0.10111 (12)	0.05592 (15)	0.24394 (9)	0.0206 (3)

H5	0.0632	0.0334	0.1928	0.025*
C6	0.13072 (11)	0.17808 (15)	0.25938 (9)	0.0171 (3)
C7	0.10814 (12)	0.27030 (14)	0.19619 (9)	0.0177 (3)
H7	0.1615	0.3221	0.1923	0.021*
C8	0.01866 (11)	0.28748 (14)	0.14364 (8)	0.0156 (3)
C9	0.01101 (11)	0.37744 (14)	0.07944 (9)	0.0153 (3)
C10	−0.07591 (11)	0.22525 (14)	0.14608 (9)	0.0158 (3)
C11	−0.10312 (12)	0.21984 (15)	0.21603 (9)	0.0203 (3)
H11	−0.0635	0.2605	0.2612	0.024*
C12	−0.18786 (13)	0.15528 (16)	0.21964 (10)	0.0234 (3)
H12	−0.2057	0.1517	0.2674	0.028*
C13	−0.24673 (12)	0.09582 (16)	0.15388 (10)	0.0239 (3)
H13	−0.3035	0.0497	0.1570	0.029*
C14	−0.22209 (12)	0.10418 (15)	0.08346 (10)	0.0213 (3)
H14	−0.2633	0.0659	0.0379	0.026*
C15	−0.13720 (12)	0.16852 (15)	0.07977 (9)	0.0177 (3)
H15	−0.1207	0.1739	0.0316	0.021*
O1	−0.06993 (8)	0.41209 (10)	0.03689 (6)	0.0185 (2)
O2	0.09775 (8)	0.41647 (11)	0.07169 (7)	0.0204 (2)
H2O	0.0845 (15)	0.471 (2)	0.0373 (12)	0.024*
Br1	0.230078 (13)	0.370757 (16)	0.360313 (9)	0.02454 (6)
C16	0.88556 (12)	0.19354 (15)	0.43750 (9)	0.0194 (3)
C17	0.94766 (12)	0.10532 (16)	0.41971 (10)	0.0234 (3)
H17	1.0158	0.1236	0.4240	0.028*
C18	0.90912 (13)	−0.00957 (16)	0.39570 (10)	0.0249 (4)
H18	0.9510	−0.0708	0.3835	0.030*
C19	0.80940 (13)	−0.03570 (15)	0.38943 (10)	0.0227 (3)
H19	0.7834	−0.1151	0.3739	0.027*
C20	0.74767 (12)	0.05462 (15)	0.40597 (9)	0.0204 (3)
H20	0.6791	0.0364	0.4003	0.025*
C21	0.78410 (11)	0.17179 (15)	0.43077 (9)	0.0173 (3)
C22	0.72086 (12)	0.26361 (14)	0.45511 (9)	0.0179 (3)
H22	0.7525	0.3113	0.4998	0.021*
C23	0.62358 (12)	0.28762 (14)	0.42138 (9)	0.0169 (3)
C24	0.57101 (12)	0.37778 (14)	0.45901 (9)	0.0172 (3)
C25	0.56263 (12)	0.23498 (14)	0.34604 (9)	0.0173 (3)
C26	0.59428 (12)	0.24671 (15)	0.27832 (9)	0.0203 (3)
H26	0.6540	0.2906	0.2801	0.024*
C27	0.53886 (13)	0.19457 (16)	0.20854 (10)	0.0234 (3)
H27	0.5611	0.2024	0.1629	0.028*
C28	0.45100 (14)	0.13098 (15)	0.20517 (10)	0.0249 (4)
H28	0.4137	0.0945	0.1575	0.030*
C29	0.41787 (13)	0.12096 (15)	0.27187 (10)	0.0239 (3)
H29	0.3577	0.0779	0.2698	0.029*
C30	0.47260 (12)	0.17383 (15)	0.34140 (10)	0.0201 (3)
H30	0.4487	0.1685	0.3864	0.024*
O3	0.48613 (8)	0.41328 (11)	0.42624 (6)	0.0207 (2)
O4	0.62187 (9)	0.41561 (11)	0.52942 (6)	0.0207 (2)

H4O	0.5877 (16)	0.467 (2)	0.5428 (12)	0.025*
Br2	0.942911 (13)	0.350346 (17)	0.471076 (12)	0.03017 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0130 (7)	0.0176 (7)	0.0209 (7)	−0.0002 (6)	0.0049 (6)	0.0010 (6)
C2	0.0144 (7)	0.0290 (9)	0.0184 (7)	0.0024 (6)	0.0034 (6)	0.0036 (6)
C3	0.0168 (7)	0.0269 (9)	0.0254 (8)	0.0037 (6)	0.0071 (6)	0.0107 (7)
C4	0.0187 (8)	0.0179 (8)	0.0308 (9)	0.0006 (6)	0.0075 (7)	0.0030 (6)
C5	0.0175 (7)	0.0228 (8)	0.0207 (8)	0.0006 (6)	0.0039 (6)	0.0007 (6)
C6	0.0131 (7)	0.0200 (8)	0.0181 (7)	0.0014 (6)	0.0041 (6)	0.0013 (6)
C7	0.0180 (7)	0.0179 (8)	0.0176 (7)	−0.0003 (6)	0.0055 (6)	−0.0008 (6)
C8	0.0171 (7)	0.0156 (7)	0.0152 (7)	0.0002 (6)	0.0061 (6)	0.0000 (5)
C9	0.0156 (7)	0.0164 (7)	0.0147 (7)	−0.0006 (6)	0.0056 (6)	−0.0020 (5)
C10	0.0147 (7)	0.0160 (7)	0.0174 (7)	0.0027 (6)	0.0057 (6)	0.0029 (6)
C11	0.0192 (8)	0.0246 (8)	0.0171 (7)	0.0031 (6)	0.0053 (6)	0.0023 (6)
C12	0.0214 (8)	0.0298 (9)	0.0224 (8)	0.0042 (7)	0.0117 (7)	0.0068 (7)
C13	0.0184 (8)	0.0239 (9)	0.0320 (9)	−0.0010 (6)	0.0112 (7)	0.0063 (7)
C14	0.0188 (8)	0.0202 (8)	0.0247 (8)	−0.0009 (6)	0.0057 (6)	−0.0010 (6)
C15	0.0184 (7)	0.0180 (7)	0.0179 (7)	0.0013 (6)	0.0070 (6)	0.0008 (6)
O1	0.0147 (5)	0.0218 (6)	0.0185 (5)	0.0001 (4)	0.0036 (4)	0.0037 (4)
O2	0.0143 (5)	0.0249 (6)	0.0224 (6)	0.0001 (4)	0.0058 (4)	0.0086 (5)
Br1	0.02498 (9)	0.02182 (9)	0.02174 (9)	−0.00288 (6)	−0.00199 (6)	−0.00055 (6)
C16	0.0192 (7)	0.0187 (8)	0.0193 (7)	−0.0005 (6)	0.0040 (6)	0.0007 (6)
C17	0.0173 (8)	0.0286 (9)	0.0248 (8)	0.0035 (7)	0.0069 (6)	0.0019 (7)
C18	0.0243 (9)	0.0246 (9)	0.0276 (9)	0.0090 (7)	0.0100 (7)	0.0008 (7)
C19	0.0270 (8)	0.0168 (8)	0.0261 (8)	0.0010 (6)	0.0103 (7)	−0.0001 (6)
C20	0.0197 (8)	0.0213 (8)	0.0219 (8)	−0.0003 (6)	0.0085 (6)	0.0026 (6)
C21	0.0174 (7)	0.0204 (8)	0.0146 (7)	0.0019 (6)	0.0051 (6)	0.0028 (6)
C22	0.0188 (7)	0.0182 (8)	0.0177 (7)	−0.0008 (6)	0.0070 (6)	0.0002 (6)
C23	0.0187 (7)	0.0158 (7)	0.0186 (7)	−0.0005 (6)	0.0091 (6)	0.0011 (6)
C24	0.0181 (7)	0.0171 (7)	0.0180 (7)	−0.0016 (6)	0.0079 (6)	0.0015 (6)
C25	0.0184 (7)	0.0150 (7)	0.0192 (7)	0.0030 (6)	0.0063 (6)	−0.0001 (6)
C26	0.0200 (8)	0.0202 (8)	0.0222 (8)	0.0016 (6)	0.0085 (6)	0.0007 (6)
C27	0.0274 (9)	0.0240 (9)	0.0205 (8)	0.0034 (7)	0.0097 (7)	−0.0004 (6)
C28	0.0294 (9)	0.0211 (8)	0.0224 (8)	0.0012 (7)	0.0044 (7)	−0.0052 (6)
C29	0.0227 (8)	0.0196 (8)	0.0297 (9)	−0.0033 (6)	0.0077 (7)	−0.0029 (7)
C30	0.0219 (8)	0.0180 (8)	0.0228 (8)	0.0005 (6)	0.0099 (6)	−0.0003 (6)
O3	0.0174 (5)	0.0240 (6)	0.0207 (5)	0.0026 (5)	0.0052 (4)	−0.0035 (5)
O4	0.0193 (6)	0.0233 (6)	0.0195 (5)	0.0049 (5)	0.0054 (4)	−0.0045 (5)
Br2	0.02001 (9)	0.02494 (10)	0.04386 (12)	−0.00432 (6)	0.00599 (8)	−0.00679 (7)

Geometric parameters (Å, º)

C1—C2	1.388 (2)	C16—C17	1.386 (2)
C1—C6	1.400 (2)	C16—C21	1.401 (2)
C1—Br1	1.9002 (16)	C16—Br2	1.9111 (16)

C2—C3	1.389 (2)	C17—C18	1.383 (3)
C2—H2	0.9500	C17—H17	0.9500
C3—C4	1.389 (2)	C18—C19	1.388 (2)
C3—H3	0.9500	C18—H18	0.9500
C4—C5	1.388 (2)	C19—C20	1.390 (2)
C4—H4	0.9500	C19—H19	0.9500
C5—C6	1.399 (2)	C20—C21	1.400 (2)
C5—H5	0.9500	C20—H20	0.9500
C6—C7	1.477 (2)	C21—C22	1.474 (2)
C7—C8	1.346 (2)	C22—C23	1.344 (2)
C7—H7	0.9500	C22—H22	0.9500
C8—C9	1.488 (2)	C23—C25	1.489 (2)
C8—C10	1.490 (2)	C23—C24	1.490 (2)
C9—O1	1.2287 (18)	C24—O3	1.2247 (19)
C9—O2	1.3205 (18)	C24—O4	1.3236 (19)
C10—C15	1.395 (2)	C25—C30	1.399 (2)
C10—C11	1.400 (2)	C25—C26	1.399 (2)
C11—C12	1.388 (2)	C26—C27	1.390 (2)
C11—H11	0.9500	C26—H26	0.9500
C12—C13	1.390 (3)	C27—C28	1.390 (2)
C12—H12	0.9500	C27—H27	0.9500
C13—C14	1.391 (2)	C28—C29	1.392 (2)
C13—H13	0.9500	C28—H28	0.9500
C14—C15	1.389 (2)	C29—C30	1.387 (2)
C14—H14	0.9500	C29—H29	0.9500
C15—H15	0.9500	C30—H30	0.9500
O2—H2O	0.84 (2)	O4—H4O	0.81 (2)

C2—C1—C6	122.28 (15)	C17—C16—C21	122.53 (15)
C2—C1—Br1	118.35 (12)	C17—C16—Br2	117.43 (12)
C6—C1—Br1	119.36 (12)	C21—C16—Br2	120.03 (12)
C1—C2—C3	118.96 (15)	C18—C17—C16	119.17 (15)
C1—C2—H2	120.5	C18—C17—H17	120.4
C3—C2—H2	120.5	C16—C17—H17	120.4
C4—C3—C2	120.28 (15)	C17—C18—C19	120.21 (15)
C4—C3—H3	119.9	C17—C18—H18	119.9
C2—C3—H3	119.9	C19—C18—H18	119.9
C5—C4—C3	119.91 (16)	C18—C19—C20	119.86 (16)
C5—C4—H4	120.0	C18—C19—H19	120.1
C3—C4—H4	120.0	C20—C19—H19	120.1
C4—C5—C6	121.33 (15)	C19—C20—C21	121.55 (15)
C4—C5—H5	119.3	C19—C20—H20	119.2
C6—C5—H5	119.3	C21—C20—H20	119.2
C5—C6—C1	117.21 (14)	C20—C21—C16	116.66 (15)
C5—C6—C7	120.64 (14)	C20—C21—C22	121.33 (14)
C1—C6—C7	122.06 (14)	C16—C21—C22	121.80 (15)
C8—C7—C6	125.82 (14)	C23—C22—C21	127.45 (15)
C8—C7—H7	117.1	C23—C22—H22	116.3

C6—C7—H7	117.1	C21—C22—H22	116.3
C7—C8—C9	118.81 (14)	C22—C23—C25	125.33 (14)
C7—C8—C10	124.62 (14)	C22—C23—C24	118.99 (14)
C9—C8—C10	116.54 (13)	C25—C23—C24	115.63 (13)
O1—C9—O2	122.81 (14)	O3—C24—O4	122.96 (14)
O1—C9—C8	122.37 (13)	O3—C24—C23	121.41 (14)
O2—C9—C8	114.82 (13)	O4—C24—C23	115.63 (13)
C15—C10—C11	118.89 (14)	C30—C25—C26	118.78 (14)
C15—C10—C8	120.92 (13)	C30—C25—C23	120.85 (13)
C11—C10—C8	120.17 (14)	C26—C25—C23	120.37 (14)
C12—C11—C10	120.26 (15)	C27—C26—C25	120.34 (15)
C12—C11—H11	119.9	C27—C26—H26	119.8
C10—C11—H11	119.9	C25—C26—H26	119.8
C11—C12—C13	120.41 (15)	C26—C27—C28	120.33 (15)
C11—C12—H12	119.8	C26—C27—H27	119.8
C13—C12—H12	119.8	C28—C27—H27	119.8
C12—C13—C14	119.68 (15)	C27—C28—C29	119.73 (16)
C12—C13—H13	120.2	C27—C28—H28	120.1
C14—C13—H13	120.2	C29—C28—H28	120.1
C15—C14—C13	119.96 (16)	C30—C29—C28	120.04 (16)
C15—C14—H14	120.0	C30—C29—H29	120.0
C13—C14—H14	120.0	C28—C29—H29	120.0
C14—C15—C10	120.74 (14)	C29—C30—C25	120.73 (15)
C14—C15—H15	119.6	C29—C30—H30	119.6
C10—C15—H15	119.6	C25—C30—H30	119.6
C9—O2—H2O	106.5 (14)	C24—O4—H4O	106.9 (14)

C6—C1—C2—C3	1.3 (2)	C21—C16—C17—C18	−1.2 (3)
Br1—C1—C2—C3	−179.76 (12)	Br2—C16—C17—C18	179.57 (13)
C1—C2—C3—C4	0.2 (2)	C16—C17—C18—C19	0.1 (3)
C2—C3—C4—C5	−1.5 (2)	C17—C18—C19—C20	1.2 (3)
C3—C4—C5—C6	1.3 (2)	C18—C19—C20—C21	−1.5 (2)
C4—C5—C6—C1	0.2 (2)	C19—C20—C21—C16	0.4 (2)
C4—C5—C6—C7	176.79 (14)	C19—C20—C21—C22	−174.34 (15)
C2—C1—C6—C5	−1.5 (2)	C17—C16—C21—C20	0.9 (2)
Br1—C1—C6—C5	179.60 (11)	Br2—C16—C21—C20	−179.89 (11)
C2—C1—C6—C7	−178.07 (14)	C17—C16—C21—C22	175.67 (15)
Br1—C1—C6—C7	3.0 (2)	Br2—C16—C21—C22	−5.1 (2)
C5—C6—C7—C8	48.0 (2)	C20—C21—C22—C23	−41.2 (2)
C1—C6—C7—C8	−135.58 (17)	C16—C21—C22—C23	144.30 (17)
C6—C7—C8—C9	−174.75 (14)	C21—C22—C23—C25	−7.6 (3)
C6—C7—C8—C10	7.3 (2)	C21—C22—C23—C24	175.15 (14)
C7—C8—C9—O1	−168.38 (15)	C22—C23—C24—O3	171.51 (15)
C10—C8—C9—O1	9.7 (2)	C25—C23—C24—O3	−6.0 (2)
C7—C8—C9—O2	11.9 (2)	C22—C23—C24—O4	−8.7 (2)
C10—C8—C9—O2	−169.98 (13)	C25—C23—C24—O4	173.74 (13)
C7—C8—C10—C15	−131.17 (17)	C22—C23—C25—C30	125.69 (17)
C9—C8—C10—C15	50.8 (2)	C24—C23—C25—C30	−57.0 (2)

C7—C8—C10—C11	47.0 (2)	C22—C23—C25—C26	−54.8 (2)
C9—C8—C10—C11	−131.04 (15)	C24—C23—C25—C26	122.57 (16)
C15—C10—C11—C12	2.3 (2)	C30—C25—C26—C27	−2.3 (2)
C8—C10—C11—C12	−175.92 (15)	C23—C25—C26—C27	178.17 (15)
C10—C11—C12—C13	−0.3 (3)	C25—C26—C27—C28	0.5 (2)
C11—C12—C13—C14	−1.9 (3)	C26—C27—C28—C29	0.8 (3)
C12—C13—C14—C15	2.0 (3)	C27—C28—C29—C30	−0.3 (3)
C13—C14—C15—C10	0.0 (2)	C28—C29—C30—C25	−1.6 (3)
C11—C10—C15—C14	−2.1 (2)	C26—C25—C30—C29	2.9 (2)
C8—C10—C15—C14	176.04 (14)	C23—C25—C30—C29	−177.60 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*O*···O1^{i}	0.84 (2)	1.80 (2)	2.6402 (16)	174 (2)
O4—H4*O*···O3^{ii}	0.81 (2)	1.84 (2)	2.6478 (16)	178 (2)
C5—H5···O3^{iii}	0.95	2.42	3.323 (2)	158
C20—H20···O1^{iii}	0.95	2.52	3.3072 (19)	141

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