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(*E*)-3-(1,3-Benzodioxol-4-yl)-2-(4-bromophenyl)-acrylic acid

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Key indicators

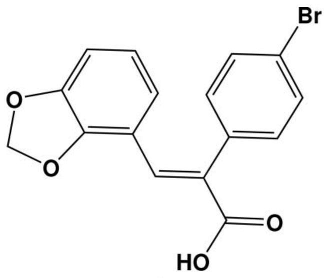
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.086
Data-to-parameter ratio = 20.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the solid-state structure of the title compound, $C_{16}H_{11}BrO_4$, the configuration about the double bond is *E*, and the mean planes through the two aromatic rings are inclined to one another by 63.71 (12)°. In the crystal structure, symmetry-related molecules are linked by O—H···O hydrogen bonds, forming centrosymmetric carboxylic acid dimers. The crystal structure is further stabilized by C—H···Br and C—H···O intermolecular hydrogen bonds, leading to the formation of a layer-like structure, which extends in the *ab* plane.

Comment

Cinnamic acid derivatives are used in the Shikimic acid metabolic pathways of higher plants (Forgó *et al.*, 2005). These compounds are also widely used as starting materials for the synthesis of antimalarial drugs. The presence of a halide group has been shown to increase their effectiveness in prototype medicinals (Nodiff *et al.*, 1971). The title compound, (I), a brominated phenylacrylic acid, was synthesized for such studies.



(I)

The molecular structure of compound (I), is shown in Fig. 1. The bond lengths and angles are similar to those found in the fluoro analogue, (*E*)-3-(1,3-benzodioxol-4-yl)-2-(4-fluorophenyl)acrylic acid, (II) (Hussain *et al.*, 2006). While compound (II) crystallized in the triclinic space group $P\overline{1}$, the title compound was found to crystallize in the monoclinic space group $P2_1/c$.

The Br1—C13 bond distance of 1.907 (2) Å is quite normal (Allen *et al.*, 1987). The benzene rings are inclined to each other by 63.71 (12)°, which is similar to the arrangement in (II) [64.81 (12)°].

The carboxylic acid group (C16/O3/O4) is inclined to the mean plane through the C10–C15 benzene ring by 63.17 (15)°. This is slightly larger than the corresponding dihedral angle in (II) [59.59 (12)°]. The dioxole ring (C5/O1/C7/O2/C6) has an envelope conformation, with atom C7 at the flap, similar to the conformation in (II). The Cremer & Pople (1975) puckerimg parameters are q_2 = 0.099 (3) Å and φ_2 = 149.4 (17)°.

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In the crystal structure of (I), centrosymmetric dimers are formed *via* O—H⋯O hydrogen bonds involving the carboxylic acid groups (Fig. 2). These dimers are further linked by C—H⋯Br and C—H⋯O hydrogen bonds, forming a layer-like structure, which extends in the *ab* plane (Fig. 2). Details of the hydrogen bonding are given in Table 1.

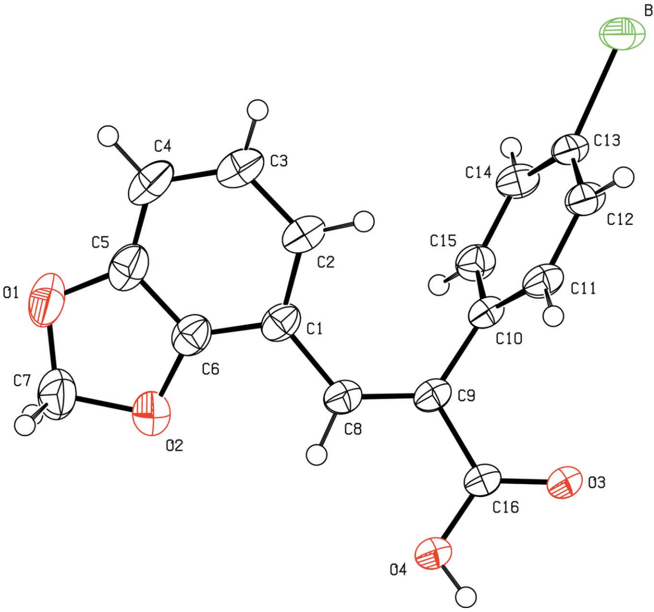

Figure 1
The molecular structure of (I), showing the atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level.

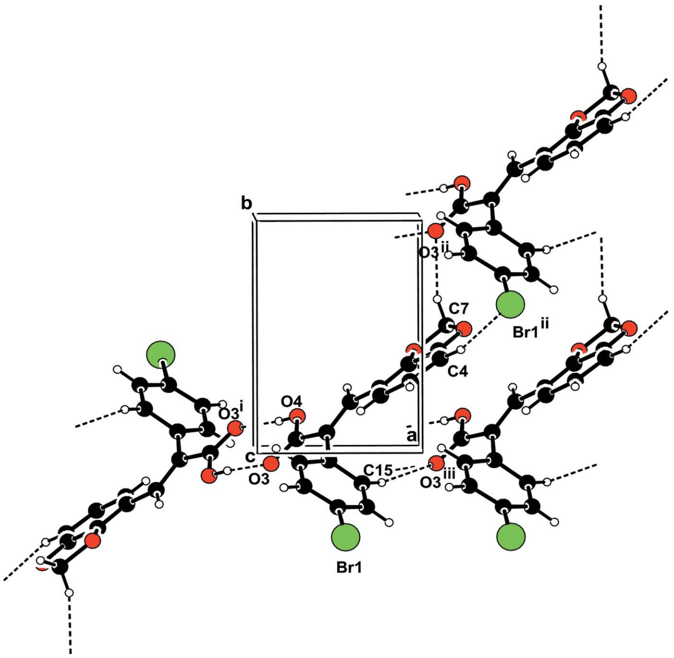

Figure 2
Part of the packing of compound (I), viewed down the *c* axis. The O—H⋯O, C—H⋯Br and C—H⋯O hydrogen bonds are shown as dashed lines (symmetry codes correspond to those given in Table 1).

Experimental

Compound (I) was synthesized according to the method described by Nodiff *et al.* (1971). A mixture of 2,3-methylenedioxybenzaldehyde (6.6 mmol), (4-bromophenyl)acetic acid (6.6 mmol), potassium carbonate (15.4 mmol) and acetic anhydride (15.4 mmol) was slowly heated to 358 K and maintained at that temperature for 24 h. To this hot solution, 10.0 ml distilled water and 5.0 ml 10% HCl were added sequentially. The solution was stirred for a further 2 h and then filtered. The resulting precipitate was washed with water to remove any impurities, including the acetic acid, and then dried. On recrystallization from chloroform, pale-yellow rod-like crystals were obtained (yield *ca* 76%).

Crystal data

$C_{16}H_{11}BrO_4$
$M_r = 347.16$
Monoclinic, $P2_1/c$
$a = 5.6331\ (6)$ Å
$b = 8.2283\ (5)$ Å
$c = 30.259\ (3)$ Å
$\beta = 91.239\ (8)^\circ$
$V = 1402.2\ (2)$ Å^3
$Z = 4$
$D_x = 1.644$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 2.94$ mm^{-1}
$T = 173\ (2)$ K
Rod, pale yellow
0.45 × 0.15 × 0.15 mm

Data collection

Stoe IPDS-2 diffractometer
φ and ω scans
Absorption correction: multi-scan (*MULABS* in *PLATON*; Spek, 2003)
$T_{\min} = 0.469$, $T_{\max} = 0.644$
17926 measured reflections
3793 independent reflections
2830 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.051$
$\theta_{\max} = 29.2^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.086$
$S = 1.04$
3793 reflections
190 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0352P)^2 + 0.7151P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.40$ e Å^{-3}
$\Delta\rho_{\min} = -0.67$ e Å^{-3}

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

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O4—H4*O*⋯O3$^{\rm i}$	0.82	1.84	2.656 (2)	175
C4—H4⋯Br1$^{\rm ii}$	0.95	2.90	3.820 (3)	165
C7—H7*B*⋯O3$^{\rm ii}$	0.99	2.42	3.367 (4)	160
C15—H15⋯O3$^{\rm iii}$	0.95	2.56	3.483 (3)	165

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

All H atoms could be located in difference Fourier maps; however, during the refinement, they were included in calculated positions and treated as riding atoms: C—H = 0.95–0.99 Å with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$, and O—H = 0.82 Å with $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm O})$.

Data collection: *X-AREA* (Stoe & Cie, 2006); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2006); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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