

Mukhtiar Hussain,^a Saqib Ali,^{a*}
Tahira Zahur,^a
Muhammad Hanif^a and
Helen Stoeckli-Evans^b

^aDepartment of Chemistry, Quaid-i-Azam University, Islamabad, Pakistan, and ^bInstitute of Microtechnology, University of Neuchâtel, Rue Emile-Argand 11, CH-2009 Neuchâtel, Switzerland

Correspondence e-mail: drsa54@yahoo.com

Key indicators

Single-crystal X-ray study
 $T = 173$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.044
 wR factor = 0.122
Data-to-parameter ratio = 11.4

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

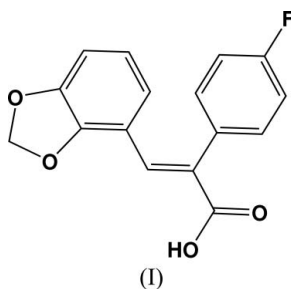
(*E*)-3-(1,3-Benzodioxol-4-yl)-2-(4-fluorophenyl)acrylic acid

In the solid-state structure of the title compound, $\text{C}_{16}\text{H}_{11}\text{NO}_4$, the double bond is *E*, and the mean planes through the two aromatic rings are inclined to one another by $64.81(12)^\circ$. In the crystal structure, symmetry-related molecules are linked by $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds, forming centrosymmetric carboxylic acid dimers. The crystal structure is further stabilized by $\text{C}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{F}$ intermolecular hydrogen bonds, leading to the formation of a slab-like structure, two molecules thick, extending in the *ab* plane.

Received 16 September 2006
Accepted 18 September 2006

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 fluoro group has been shown to increase their effectiveness in prototype medicinals (Nodiff *et al.*, 1971). The title compound, (I), a fluorinated phenylacrylic acid, was synthesized for such studies.



The molecular structure of compound (I) is shown in Fig. 1, and selected bond distances and angles are given in Table 1. The bond lengths and angles are similar to those found in (*E*)-3-(6-nitro-1,3-benzodioxol-5-yl)-2-phenylacrylic acid, (II) (Hussain *et al.*, 2006), and 3-(4-nitrophenyl)-2-phenylacrylic acid, (III) (Kuleshova *et al.*, 2003). The $\text{F1}-\text{C13}$ bond distance is quite normal (Allen *et al.*, 1987). The central $\text{C}=\text{C}$ bond ($\text{C8}=\text{C9}$) shows clear double-bond character, and bonds $\text{C1}-\text{C8}$ and $\text{C9}-\text{C10}$ single-bond character, which confirms the localized bonding. The $\text{C1}-\text{C6}$ benzene ring is not perfectly planar and has a weighted average absolute torsion angle of 2.6° (Domenicano *et al.*, 1975) compared to 1.1° in (II). This is explained by the lower precision of the crystal structure analysis of compound (I) (see *Experimental* refinement section). The planar $\text{C10}-\text{C15}$ benzene ring is inclined to the mean plane through the $\text{C1}-\text{C6}$ benzene ring by $64.81(12)^\circ$, which is slightly less than the same dihedral angles in (II) or (III) [$67.96(7)$ and 68.7° , respectively].

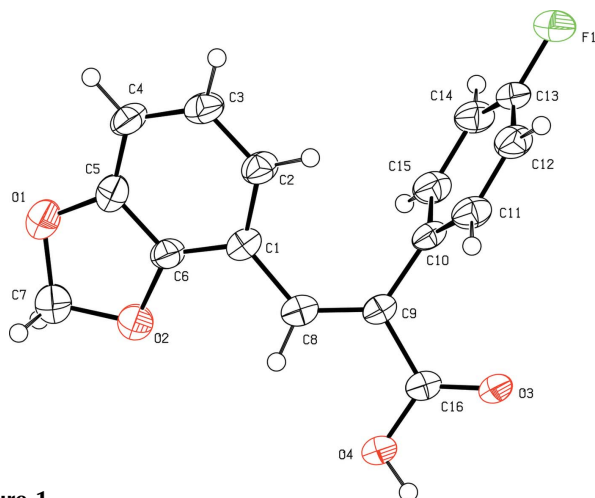


Figure 1
The molecular structure of (I), showing the atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level.

The carboxylic acid group (C16/O5/O6) is inclined to the mean plane through the C10–C15 benzene by $59.59 (12)^\circ$. This is larger than the same dihedral angle found in (II) of $48.06 (14)^\circ$, but smaller than that of 65.5° found in (III). The dioxole ring (C3/O3/C7/O4/C4) has an envelope conformation, with atom C7 at the flap, similar to the conformation found in (II). The Cremer & Pople (1975) puckering parameters are $Q(2) = 0.105 (3) \text{ \AA}$ and $\varphi(2) = 148.0 (15)^\circ$.

In the crystal structure of (I), centrosymmetric dimers are formed *via* O–H...O hydrogen bonds involving the carboxylic acid groups (see Table 2 and Fig. 2 for details). This arrangement is similar to that observed in the crystal structures of (II) and (III). In (I), these dimers are further linked by C–H...O and C–H...F hydrogen bonds, forming a slab-like structure, two molecules thick, extending in the *ab* plane (Fig. 2).

Experimental

Compound (I) was synthesized according to the method of Nodiff *et al.* (1971). A mixture of 2,3-methylenedioxybenzaldehyde (6.6 mmol), (4-fluorophenyl)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 of distilled water and 5.0 ml of 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, yellow plate-like crystals were obtained (yield *ca* 85%).

Crystal data

$C_{16}H_{11}FO_4$	$V = 653.5 (2) \text{ \AA}^3$
$M_r = 286.25$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 1.455 \text{ Mg m}^{-3}$
$a = 5.6894 (12) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 8.3041 (19) \text{ \AA}$	$\mu = 0.11 \text{ mm}^{-1}$
$c = 13.959 (3) \text{ \AA}$	$T = 173 (2) \text{ K}$
$\alpha = 97.29 (3)^\circ$	Plate, pale yellow
$\beta = 92.52 (2)^\circ$	$0.30 \times 0.23 \times 0.08 \text{ mm}$
$\gamma = 90.10 (3)^\circ$	

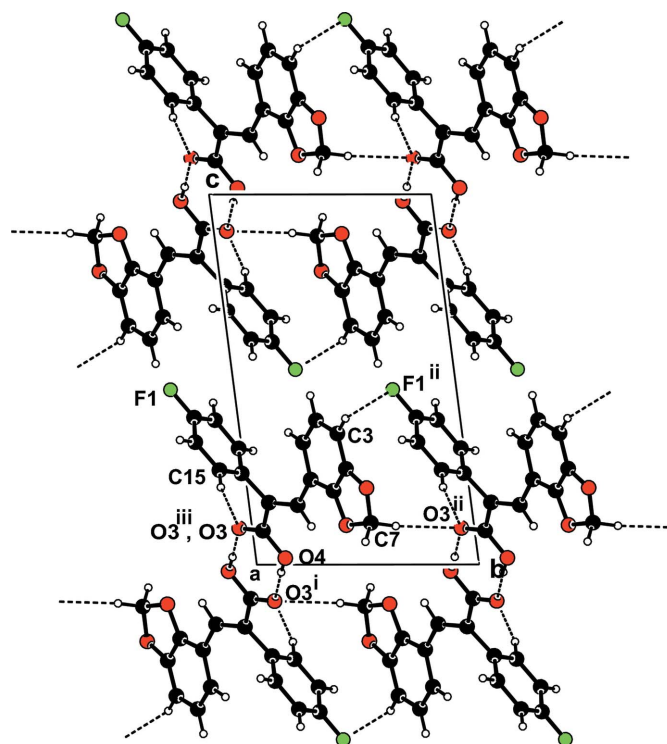


Figure 2
The crystal packing of (I), viewed down the *a* axis. The O–H...O, C–H...O and C–H...F hydrogen bonds are shown as dashed lines (symmetry codes correspond to those given in Table 2).

Data collection

Stoe IPDS diffractometer	2213 independent reflections
φ scans	1219 reflections with $I > 2\sigma(I)$
Absorption correction: none	$R_{\text{int}} = 0.058$
4576 measured reflections	$\theta_{\text{max}} = 26.1^\circ$

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.044$	$w = 1/[\sigma^2(F_o^2) + (0.0694P)^2]$
$wR(F^2) = 0.122$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.86$	$(\Delta/\sigma)_{\text{max}} < 0.001$
2213 reflections	$\Delta\rho_{\text{max}} = 0.24 \text{ e \AA}^{-3}$
194 parameters	$\Delta\rho_{\text{min}} = -0.19 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

F1–C13	1.365 (3)	O2–C7	1.439 (4)
O1–C5	1.375 (3)	O3–C16	1.229 (3)
O1–C7	1.440 (3)	O4–C16	1.324 (3)
O2–C6	1.385 (3)		
C5–O1–C7	105.5 (2)	O1–C7–O2	107.7 (2)
C6–O2–C7	105.7 (2)	F1–C13–C12	118.8 (2)
O1–C5–C4	128.0 (3)	F1–C13–C14	118.3 (2)
O1–C5–C6	110.4 (2)	O3–C16–C9	122.2 (2)
O2–C6–C5	109.5 (2)	O4–C16–C9	115.3 (2)
O2–C6–C1	126.6 (2)	O3–C16–O4	122.5 (2)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O4-H4O\cdots O3^i$	0.87 (2)	1.79 (2)	2.658 (3)	178 (4)
$C4-H4\cdots F1^{ii}$	0.95	2.53	3.414 (3)	154
$C7-H7A\cdots O3^{ii}$	0.99	2.45	3.401 (4)	160
$C15-H15\cdots O3^{iii}$	0.95	2.58	3.508 (3)	167

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

The crystal was a twin and the integration was carried out using the TWIN integration routine in *IPDS Software* (Stoe & Cie, 2000). 607 reflections were overlapped and removed from the list of reflections, hence the reflection count is only 85% for a θ_{\max} of 26.1°. All H atoms could be located in difference Fourier maps; however, during the refinement the aromatic H atoms were included in calculated positions and treated as riding atoms: C–H = 0.95–0.99 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The OH H atom was refined isotropically, with the O–H distance restrained to 0.84 (2) Å.

Data collection: *EXPOSE* (Stoe & Cie, 2000); cell refinement: *CELL* (Stoe & Cie, 2000); data reduction: *TWIN-INTEGRATE* (Stoe & Cie, 2000); 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*.

M. Hussain and M. Hanif are grateful to the Higher Education Commission of Pakistan for financial support.

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
- Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
- Domenicano, A., Vaciago, A. & Coulson, C. A. (1975). *Acta Cryst.* **B31**, 221–234.
- Forgó, P., Felföldi, K. & Pálinkó, I. (2005). *J. Mol. Struct.* **744–747**, 273–276.
- Kuleshova, L. N., Khrustalev, V. N., Suponitsky, K. Yu., Gusev, D. V. & Bobrikova, E. S. (2003). *Kristallografiya*, **48**, 78–83.
- Hussain, M., Hanif, M., Ali, S., Altaf, M. & Stoeckli-Evans, H. (2006). *Acta Cryst.* **E62**. Submitted.
- Nodiff, E. A., Tanabe, K., Seyfried, C., Matsuura, S., Kondo, Y., Chen, E. H. & Tyagi, M. P. (1971). *J. Med. Chem.* **14**, 921–925.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Göttingen, Germany.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
- Stoe & Cie (2000). *EXPOSE*, *CELL* and *TWIN-INTEGRATE* in *IPDS Software*. Stoe & Cie GmbH, Darmstadt, Germany.