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Pentafluorophenyl 3-phenylprop-2-enoate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.045
wR factor = 0.134
Data-to-parameter ratio = 12.3

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

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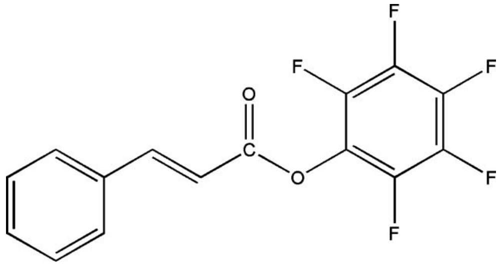
In the title compound, $C_{15}H_7F_5O_2$, an intramolecular C—H···O hydrogen bond induces coplanarity of the central chain and the phenyl ring. An intermolecular C—H···O hydrogen bond leads to the formation of a dimer.

Comment

As part of a study of cinnamic acid reactivity towards pentafluorophenol *versus* pentafluorothiophenol, the title compound, (I), has been synthesized according to a literature method (Zhao *et al.*,1997). The regular geometry of the carbon skeleton of the phenyl ring (C4–C9) is slightly modified due to the substituent atom C3 (Fig. 1). The deformation can be attributed to hybridization and conjugation effects (Domenicano *et al.*, 1975) and can be described in the following way: an endocyclic angle α [C5—C4—C9 118.4 (3)°] smaller than 120°, aromatic bonds adjacent to atom C4 [average of C4—C5 and C4—C9 1.386 (1) Å] longer than the neighboring ones [average of C5—C6 and C8—C9 1.380 (1) Å] and a significant shortening of the C3—C4 bond [1.459 (4) Å] compared to the reported $C_{aromatic}—Csp^2$ average value of 1.483 Å (Allen *et al.*, 1987).



(I)

In the pentafluorophenyl group, the average $C_{aromatic}—C_{aromatic}$ and $C_{aromatic}—F$ bond lengths agree very well with reported values (Allen *et al.*, 1987), although there are significant differences among several equivalent bonds (Table 1). These are probably due to significant librational motion of the pentafluorophenyl group, as shown by the shape of the displacement ellipsoids. Within the central chain, the C1═O2 and C2═C3 distances agree well with expected values; however, the C1—C2 [1.454 (4) Å] and C10—O1 [1.376 (4) Å] bonds are significantly shorter, and the C1—O1 [1.382 (4) Å] bond is significantly larger than reported values (Allen *et al.*, 1987). All of these observed differences can be ascribed to the resonance effects of the two terminal aryl rings and the C2═C3 double bond.

The molecule is characterized by an intramolecular C3—H3···O2 hydrogen bond (Table 2), which is responsible for

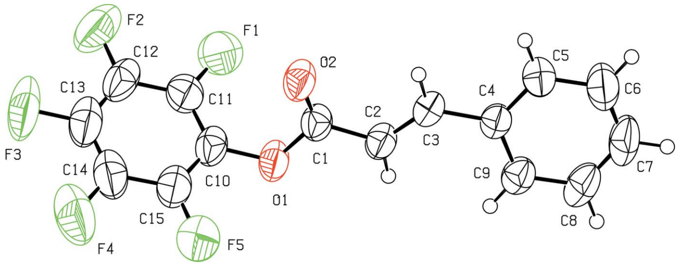


Figure 1
Molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

the planarity of the group of atoms C1–C10/O1/O2 including the central chain and the phenyl ring (maximum deviation 0.162 Å for atom O2). The dihedral angle between this plane and the O1/C10–C15/F1–F5 plane (maximum deviation 0.017 Å for atom F5) is 62.89 (6)°. The crystal packing is stabilized by a weak intermolecular C3—H3···O2^{i} hydrogen bond (symmetry code as in Table 2), which leads to the formation of a centrosymmetric dimer.

Experimental

The title compound was prepared according to the method reported by Zhao *et al.* (1997), treating 3-phenyl-2-propenoic acid with dicyclohexylcarbodiimide (DCC) and pentafluorophenol in anhydrous dioxane. The mixture was stirred at room temperature for 30 min and cooled. Then dicyclohexylurea was removed by filtration and the filtrate was purified by silica gel flash column chromatography (diethyl ether-petroleum ether 1:9), to provide the title compound as a crystalline product. Suitable crystals for X-ray diffraction were obtained from slow evaporation of a diethyl ether solution.

Crystal data

$C_{15}H_7F_5O_2$
$M_r = 314.21$
Monoclinic, $P2_1/n$
$a = 7.261$ (5) Å
$b = 15.1156$ (17) Å
$c = 12.3823$ (15) Å
$\beta = 101.01$ (2)°
$V = 1334.0$ (9) Å^3
$Z = 4$
$D_x = 1.564$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 7.4$–12.5°
$\mu = 0.15$ mm^{-1}
$T = 293$ (2) K
Prism, colourless
0.49 × 0.18 × 0.12 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
3210 measured reflections
2463 independent reflections
1086 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.025$
$\theta_{\rm max} = 25.5°$
$h = -4 \rightarrow 8$
$k = -8 \rightarrow 18$
$l = -14 \rightarrow 14$
3 standard reflections every 200 reflections intensity decay: 10.6%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.134$
$S = 1.01$
2463 reflections
200 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0424P)^2 + 0.684P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.003$
$\Delta\rho_{\rm max} = 0.24$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.23$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0144 (19)

Table 1
Selected bond lengths (Å).

F1—C11	1.347 (4)	C10—C15	1.375 (5)
F2—C12	1.330 (5)	C11—C12	1.386 (5)
F3—C13	1.351 (4)	C12—C13	1.366 (6)
F4—C14	1.344 (5)	C13—C14	1.345 (6)
F5—C15	1.329 (4)	C14—C15	1.380 (5)
C10—C11	1.356 (5)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O2	0.93	2.53	2.858 (4)	101
C3—H3···O2^{i}	0.93	2.44	3.308 (4)	156

Symmetry code: (i) $-x+2, -y+2, -z+2$.

H atoms were refined as riding on their parent atoms, with C—H = 0.93 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$. The linear decay of the intensity with exposure time appears to be related to the crystal and not to the source.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *HELENA* (Spek,1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson,1976); software used to prepare material for publication: *SHELXL97*.

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