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Key indicators

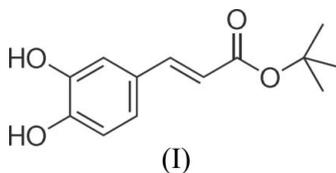
Single-crystal X-ray study
 $T = 298$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.067
 wR factor = 0.226
Data-to-parameter ratio = 17.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.*tert*-Butyl 3-(3,4-dihydroxyphenyl)prop-2-enoate

The molecule of the title compound, $\text{C}_{13}\text{H}_{16}\text{O}_4$, adopts an *E* configuration, the carboxyl group and benzene ring being located on opposite sides of the $\text{C}=\text{C}$ bond. Intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonding helps to stabilize the crystal structure.

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Comment

Caffeic acid and its derivatives are widely distributed in the plant kingdom (Chen *et al.*, 1999). These compounds are known to have anti-atherosclerotic, antibacterial, anti-inflammatory, antiproliferative, immunostimulatory, anti-oxidative, antiviral and neuroprotective properties (Son & Lewis, 2002). As part of our ongoing research on caffeic acid derivatives (Xia & Hu, 2005), we present here the structure of the title compound, (I).



The molecular structure of (I) is illustrated in Fig. 1. The molecule displays an *E* configuration, with the benzene ring and carboxyl group located on opposite sides of the $\text{C}7=\text{C}8$ double bond. Bond distances agree with those found in similar caffeate structures (Xia *et al.*, 2004, 2006). The propenoate group is tilted with respect to the benzene ring with a dihedral angle of $22.71(10)^\circ$.

Intermolecular hydrogen bonding between hydroxy groups and between carboxyl and hydroxy groups (Table 1) helps to stabilize the crystal structure.

Experimental

Meldrum's acid (3.6 g, 25 mmol) and *tert*-butanol (1.85 g, 25 mmol) were mixed in toluene (50 ml) and the mixture was refluxed for 5 h. After cooling to room temperature, 3,4-dihydroxybenzaldehyde (1.4 g, 10 mmol), pyridine (2.5 ml) and piperidine (0.25 ml) were added. The resulting mixture was stirred for 12 h at room temperature. The solvents were then distilled out under vacuum. The residue was dissolved in diethyl ether (30 ml), washed in turn with a saturated solution of sodium bicarbonate (20 ml), dilute hydrochloric acid (20 ml) and distilled water (20 ml). The ether phase was dried by anhydrous MgSO_4 overnight. After removal of the drier, the solvent was distilled out, giving a yellowish sheet-like solid (1.45 g, 61.4%). Recrystallization from a mixture of acetone, diethyl ether and water (8:1:1) gave single crystals of (I).

Crystal data

$C_{13}H_{16}O_4$
 $M_r = 236.26$
 Monoclinic, $P2_1/c$
 $a = 9.334 (3) \text{ \AA}$
 $b = 11.000 (3) \text{ \AA}$
 $c = 12.701 (4) \text{ \AA}$
 $\beta = 98.005 (4)^\circ$
 $V = 1291.4 (7) \text{ \AA}^3$

$Z = 4$
 $D_x = 1.215 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 $\mu = 0.09 \text{ mm}^{-1}$
 $T = 298 (2) \text{ K}$
 Prism, light brown
 $0.25 \times 0.20 \times 0.20 \text{ mm}$

Data collection

Bruker APEX CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: none
 6285 measured reflections

2826 independent reflections
 1857 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.078$
 $\theta_{\text{max}} = 27.1^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.067$
 $wR(F^2) = 0.226$
 $S = 1.11$
 2826 reflections
 160 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1179P)^2 + 0.1691P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.26 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.42 \text{ e \AA}^{-3}$
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.031 (6)

Table 1

Hydrogen-bond geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O1-H1\cdots O3^i$	0.82	1.85	2.669 (2)	172
$O2-H2\cdots O1^{ii}$	0.82	2.12	2.826 (2)	144

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

H atoms bonded to O atoms were located in a difference Fourier map and refined with the constraint $O-H = 0.82 \text{ \AA}$ [$U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$]. Methyl H atoms were placed in calculated positions, with $C-H = 0.96 \text{ \AA}$ and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$. Other H atoms were placed

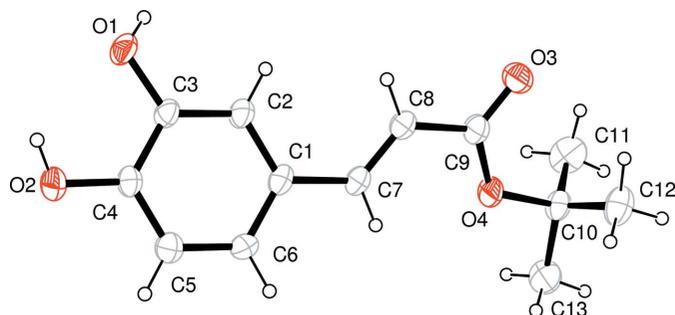


Figure 1

The molecular structure of (I), shown with 30% probability displacement ellipsoids (arbitrary spheres for H atoms).

in calculated positions, with $C-H = 0.93 \text{ \AA}$, and refined in riding mode [$U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$].

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINTE* (Bruker, 2003); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXTL* (Bruker, 2003); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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