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Key indicators

Single-crystal X-ray study
 $T = 298\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$
 R factor = 0.068
 wR factor = 0.213
Data-to-parameter ratio = 14.3For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

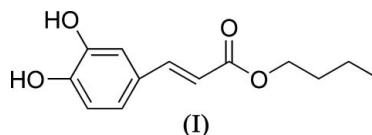
Butyl 3-(3,4-dihydroxyphenyl)prop-2-enoate

The $\text{C}=\text{C}$ double bond of the title compound, $\text{C}_{13}\text{H}_{16}\text{O}_4$, synthesized by the Knoevenagel–Doebner condensation, is in the *E* configuration. There are intra- and intermolecular hydrogen bonds in the crystal structure.

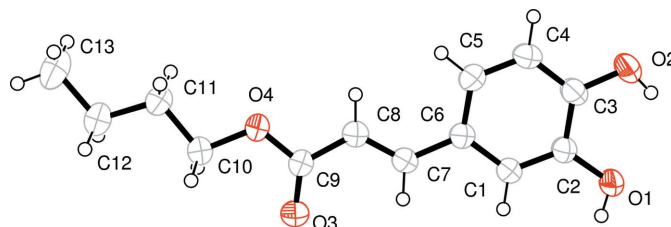
Received 14 February 2006

Accepted 15 February 2006

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). In a continuation of our research on structure–activity relationships, we have obtained the title compound, (I), as a product of the Knoevenagel–Doebner condensation reaction of 3,4-dihydroxybenzaldehyde and monobutyl malonate (Xia & Hu, 2005).The molecular structure of (I) is illustrated in Fig. 1. The $\text{C}=\text{C}$ double bond is in the *E* configuration. Selected bond lengths and angles are listed in Table 1. The molecule is almost planar (r.m.s. deviation of all non-H atoms is 0.041 Å). There are intra- and intermolecular hydrogen bonds in the crystal structure (Table 1 and Fig. 2).

Experimental

3,4-Dihydroxybenzaldehyde (1.4 g, 10 mmol) and monobutyl malonate (4.0 g, 25 mmol) were dissolved in a mixture of pyridine (5 ml) and piperidine (0.2 ml). The solution was stirred at room temperature for 24 h and concentrated *in vacuo* to give a dark-brown mixture. The mixture was dissolved in diethyl ether (30 ml), and washed with a saturated NaHCO_3 solution, a diluted HCl solution and distilled water. It was then dried with anhydrous MgSO_4 . The solution was**Figure 1**
The structure of (I), shown with 30% probability displacement ellipsoids.

filtered and concentrated to yield a light-yellow crystalline product (yield 1.6 g, 67.8%). Recrystallization from a mixture of benzene and diethyl ether (8:2) gave colourless prisms (m.p. 382–384 K). Single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of a benzene solution.

Crystal data

$C_{13}H_{16}O_4$	$Z = 2$
$M_r = 236.26$	$D_x = 1.251 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 5.282 (5) \text{ \AA}$	Cell parameters from 25 reflections
$b = 10.490 (5) \text{ \AA}$	$\theta = 9.8\text{--}13.7^\circ$
$c = 11.558 (7) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$\alpha = 83.95 (6)^\circ$	$T = 298 (2) \text{ K}$
$\beta = 84.31 (7)^\circ$	Prism, colourless
$\gamma = 81.14 (6)^\circ$	$0.50 \times 0.40 \times 0.30 \text{ mm}$
$V = 627.0 (8) \text{ \AA}^3$	

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{\text{int}} = 0.014$
$\omega/2\theta$ scans	$\theta_{\text{max}} = 25.2^\circ$
Absorption correction: ψ scan (North <i>et al.</i> , 1968)	$h = -1 \rightarrow 6$
$T_{\text{min}} = 0.964$, $T_{\text{max}} = 0.964$	$k = -12 \rightarrow 12$
3050 measured reflections	$l = -13 \rightarrow 13$
2259 independent reflections	3 standard reflections
1301 reflections with $I > 2\sigma(I)$	frequency: 60 min
	intensity decay: 0.3%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1452P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.068$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.213$	$(\Delta/\sigma)_{\text{max}} = 0.004$
$S = 1.02$	$\Delta\rho_{\text{max}} = 0.31 \text{ e \AA}^{-3}$
2259 reflections	$\Delta\rho_{\text{min}} = -0.33 \text{ e \AA}^{-3}$
158 parameters	Extinction correction: <i>SHELXL97</i>
H-atom parameters constrained	Extinction coefficient: 0.019 (3)

Table 1

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

$D\text{---}H\cdots A$	$D\text{---}H$	$H\cdots A$	$D\cdots A$	$D\text{---}H\cdots A$
$O1\text{---}H1\cdots O3^i$	0.83	1.97	2.788 (3)	167
$O2\text{---}H2\cdots O1$	0.83	2.29	2.726 (3)	113
$O2\text{---}H2\cdots O1^{ii}$	0.83	2.11	2.818 (3)	143
$C1\text{---}H1A\cdots O3^i$	0.93	2.55	3.255 (3)	133

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

H atoms were included at calculated positions and refined using a riding model, with $U_{\text{iso}}(\text{H})$ values set at 1.2 times (1.5 times for methyl) the equivalent isotropic displacement parameters of their

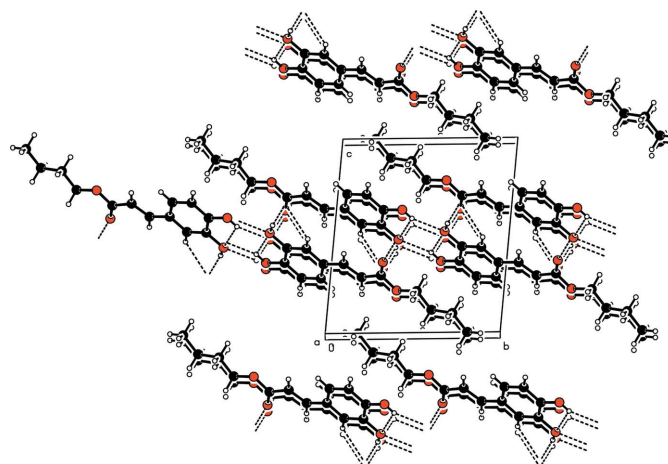


Figure 2

Packing diagram of (I), viewed along the a axis, with hydrogen bonds shown as dashed lines.

parent atoms. C–H distances were set at 0.97 \AA for the methylene H atoms, at 0.96 \AA for the methyl H atoms and at 0.93 \AA for the remainder, while O–H distances were fixed at 0.83 \AA .

Data collection: *CAD-4 Software* (Enraf–Nonius, 1993); cell refinement: *CAD-4 Software*; data reduction: *CAD-4 Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

We are very grateful to the National Natural and Scientific Foundation (grant No. 20272053). We also acknowledge financial support from the Science and Technology Bureau of Zhejiang Province (grant No. 2005 C23022).

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