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Ethyl 3-(3,4-dihydroxyphenyl)-2-propenoate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.034
wR factor = 0.103
Data-to-parameter ratio = 12.8

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

The title compound, $C_{11}H_{12}O_4$, was prepared from the Knoevenagel condensation reaction of 3,4-dihydroxybenzaldehyde and monoethyl malonate. The almost planar molecule is the *E* isomer.

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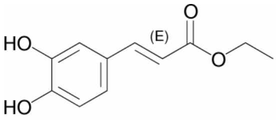
Comment

Caffeic acid and its derivatives are widely distributed in the plant kingdom (Chen *et al.*, 1999). These compounds are known to have antiatherosclerotic, antibacterial, anti-inflammatory, antiproliferative, immunostimulatory, antioxidative, antiviral and neuroprotective properties (Son & Lewis, 2002). In a continuation of our search of structure–activity relationships in caffeic acid derivatives, we have obtained the title compound, (I), as a product of the Knoevenagel condensation reaction of 3,4-dihydroxybenzaldehyde and monoethyl malonate.



(I)

The molecular structure of (I) is illustrated in Fig. 1. Its configuration is the *E* form. Selected bond lengths and angles are listed in Table 1. All non-H atoms of (I) are almost coplanar, deviating from the least-squares plane within 0.0564 (15) Å.

Experimental

3,4-Dihydroxybenzaldehyde (5.6 g, 20 mmol) and monoethyl malonate (13.6 g, 100 mmol) were dissolved in a mixture of pyridine (15 ml) and piperidine (0.5 ml). The solution was stirred at room temperature for 24 h and dried *in vacuo* to give a dark-brown mixture. The cooled mixture was dissolved in dry ether (30 ml), washed with saturated $NaHCO_3$ solution, then dried with anhydrous $MgSO_4$. The solution was filtered and concentrated to yield a brown crystalline product (6.6 g, 78.6%). Recrystallization from ethanol gave light-brown crystalline prisms (m.p. 420–421 K).

Crystal data

$C_{11}H_{12}O_4$
$M_r = 208.21$
Monoclinic, $P2_1/c$
$a = 6.659$ (1) Å
$b = 5.811$ (1) Å
$c = 25.992$ (7) Å
$\beta = 91.51$ (2)°
$V = 1005.4$ (4) Å^3
$Z = 4$
$D_x = 1.375$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 9.9$–13.9°
$\mu = 0.11$ mm^{-1}
$T = 293$ (2) K
Prism, light brown
0.35 × 0.30 × 0.25 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min} = 0.964$, $T_{\max} = 0.974$
2218 measured reflections
1792 independent reflections
1373 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.021$
$\theta_{\max} = 25.2°$
$h = -7 \rightarrow 7$
$k = -1 \rightarrow 6$
$l = 0 \rightarrow 31$
3 standard reflections frequency: 60 min intensity decay: 0.3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.103$
$S = 1.08$
1792 reflections
140 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0513P)^2 + 0.2394P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.17$ e Å^{-3}
$\Delta\rho_{\min} = -0.16$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.010 (2)

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.3568 (17)	O4—C9	1.3296 (18)
O2—C3	1.3637 (16)	O4—C10	1.4491 (16)
O3—C9	1.2154 (18)	C7—C8	1.319 (2)
O1—C2—C1	123.72 (13)	O2—C3—C2	115.98 (13)
O1—C2—C3	116.70 (12)	O3—C9—O4	122.57 (13)
O2—C3—C4	124.02 (13)	O3—C9—C8	123.47 (14)

H atoms were included at calculated positions and refined using a riding model. H atoms were given isotropic displacement parameters equal to 1.2 (or 1.5 for methyl H atoms) times the equivalent isotropic displacement parameters of their parent atoms, and C—H distances were set to 0.93 Å for H atoms bonded to the benzene ring, 0.96 Å for methyl H atoms and 0.97 Å for the remainder. O—H distances were fixed at 0.82 Å.

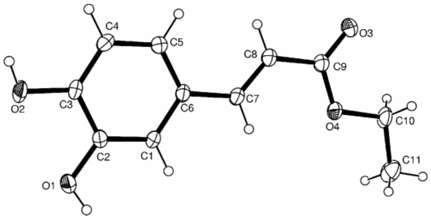


Figure 1
View of (I), with 30% probability displacement ellipsoids.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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