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Prop-2-ynyl 3-(3,4-dihydroxyphenyl)-prop-2-enoate monohydrate

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Crystals of the title compound, $C_{12}H_{10}O_4 \cdot H_2O$, were obtained from the Knoevenagel condensation reaction of 3,4-dihydroxybenzaldehyde and monoprop-2-ynyl malonate. The almost planar molecule is the *E* isomer. There are intermolecular O—H$\cdots$O and C—H$\cdots$O hydrogen bonds and π–π interactions, which contribute to the formation of the crystal structure.

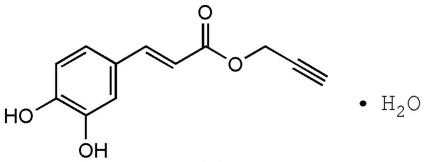
Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.003 Å
R factor = 0.034
wR factor = 0.101
Data-to-parameter ratio = 12.6

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

Comment

Caffeic acid and its derivatives are widely distributed in the plant kingdom (Chen *et al.*, 1999). These compounds demonstrate anti-atherosclerotic, antibacterial, anti-inflammatory, antiproliferative, immunostimulatory, anti-oxidative, antiviral and neuroprotective properties (Son & Lewis, 2002). In a continuation of our research of structure–activity relationships in caffeic acid derivatives (Xia *et al.*, 2004), we have obtained the title compound, (I), as a product of the Knoevenagel condensation reaction of 3,4-dihydroxybenzaldehyde and monoprop-2-ynyl malonate.



(I)

The molecular structure of (I), incorporating one water molecule as crystallization solvent, is illustrated in Fig. 1. The molecular configuration of the caffeic acid ester is the *E* form. Selected bond lengths and angles are listed in the Table 1. All non-H atoms in the molecule are coplanar within 0.0077 (15) Å. The crystal packing (Fig. 2) is stabilized by intermolecular O—H$\cdots$O and C—H$\cdots$O hydrogen bonds (Table 2). The molecules of caffeic acid ester form stacks along the *b* axis in a head-to-tail manner with the interplanar separation of 3.390 Å, showing the presence of $\pi\cdots\pi$ interactions.

Experimental

3,4-Dihydroxybenzaldehyde (1.4 g, 10 mmol) and monoprop-2-ynyl malonate (3.6 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 16 h and dried *in vacuo* to give a dark-brown mixture. The cooled mixture was dissolved in dry diethyl ether (10 ml), washed with saturated $NaHCO_3$ solution, then dried with anhydrous $MgSO_4$. The solution was filtered and concentrated to yield a light brown crystalline product (2.1 g, 96%). Recrystallization from a mixture of benzene, acetone and H_2O (4:4:1) gave light-brown crystalline prisms (m.p. 389–392 K).

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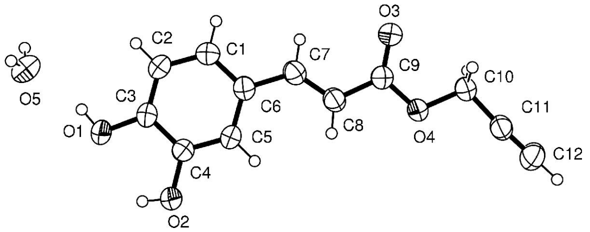

Figure 1
View of (I), with 30% probability displacement ellipsoids.

Crystal data

$C_{12}H_{10}O_4 \cdot H_2O$
$M_r = 236.22$
Monoclinic, $P2_1/n$
$a = 12.857$ (3) Å
$b = 7.0660$ (12) Å
$c = 13.5410$ (12) Å
$\beta = 109.560$ (17)°
$V = 1159.2$ (4) Å^3
$Z = 4$
$D_x = 1.354$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 10.0$–13.7°
$\mu = 0.11$ mm^{-1}
$T = 296$ (2) K
Prism, light brown
0.50 × 0.40 × 0.30 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.939$, $T_{\max} = 0.969$
2387 measured reflections
2087 independent reflections
1461 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.022$
$\theta_{\max} = 25.2°$
$h = -1 \rightarrow 15$
$k = 0 \rightarrow 8$
$l = -16 \rightarrow 15$
3 standard reflections
frequency: 60 min
intensity decay: 0.3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.101$
$S = 1.03$
2087 reflections
165 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0424P)^2 + 0.3495P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.16$ e Å^{-3}
$\Delta\rho_{\min} = -0.13$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0107 (18)

Table 1
Selected geometric parameters (Å, °).

O1—C3	1.356 (2)	O4—C9	1.336 (2)
O2—C4	1.372 (2)	O4—C10	1.446 (2)
O3—C9	1.2108 (19)		
C9—O4—C10	114.15 (13)	O3—C9—O4	121.44 (15)
O1—C3—C2	125.46 (15)	O3—C9—C8	125.79 (16)
O1—C3—C4	115.23 (15)	O4—C9—C8	112.77 (15)
C5—C4—O2	119.31 (14)	O4—C10—C11	109.00 (14)
O2—C4—C3	120.28 (14)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O5	0.83	1.90	2.7150 (19)	166
O2—H2···O5$^{\rm i}$	0.83	2.20	2.920 (2)	145
O2—H2···O1	0.83	2.22	2.6655 (17)	114
O5—H5B···O3$^{\rm ii}$	0.851 (10)	1.961 (16)	2.757 (2)	155 (3)
O5—H5A···O2$^{\rm iii}$	0.847 (10)	2.278 (19)	3.028 (2)	148 (3)
C12—H12···O2$^{\rm iv}$	0.93	2.40	3.260 (3)	154

Symmetry codes: (i) $1 - x, -y, 1 - z$; (ii) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{3}{2} - z$; (iii) $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$; (iv) $-\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$.

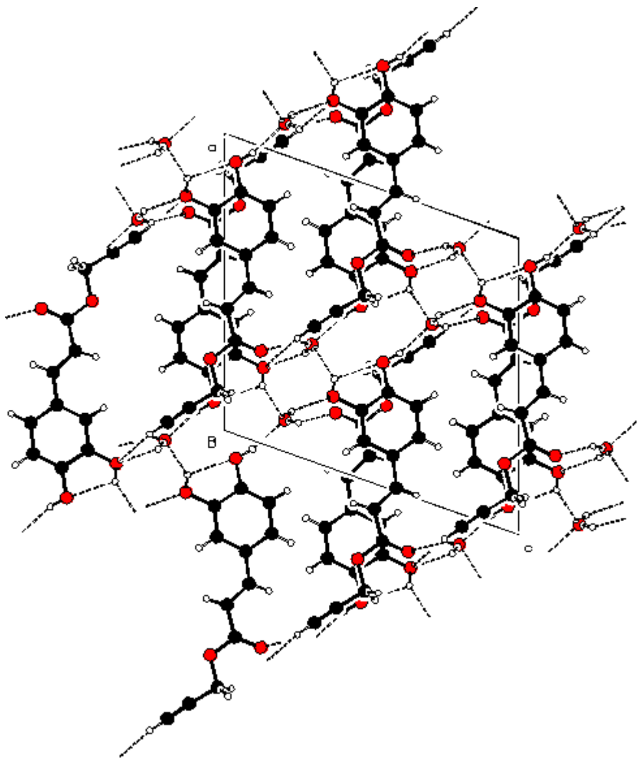

Figure 2
Packing diagram, showing the intermolecular hydrogen bonds (dashed lines), viewed along the *b* axis.

H atoms of H_2O were located in difference Fourier maps and refined isotropically. C-bound H-atoms were placed at calculated positions and refined using a riding model. H atoms were given isotropic displacement parameters equal to $1.2U_{\rm eq}$ of the parent atoms, and C—H distances were set to 0.97 Å for the methylene H atoms and 0.93 Å for the remainder. The O—H distances in the hydroxy groups were fixed at 0.83 Å.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD* (McArdle, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1999); software used to prepare material for publication: *SHELXL97*.

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