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Pentyl (*E*)-3-(3,4-dihydroxyphenyl)-acrylate

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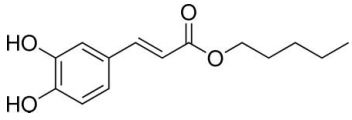
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.057; wR factor = 0.169; data-to-parameter ratio = 14.8.

In the molecule of the title compound, $C_{14}H_{18}O_4$, the C=C double bond is in an *E* configuration. The molecule is almost planar (r.m.s. deviation of all non-H atoms = 0.04 Å). An intramolecular O—H···O hydrogen bond occurs. In the crystal, intermolecular O—H···O interactions link the molecules into ribbons extending in [110].

Related literature

For general background to the biological activity of caffeic acid and its esters, see: Uwai *et al.* (2008); Buzzi *et al.* (2009); For the preparation, see: Xia *et al.* (2006); Son *et al.* (2011). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{14}H_{18}O_4$
M_r = 250.28
Triclinic, $P\overline{1}$
a = 5.3070 (11) Å
b = 10.567 (2) Å
c = 11.816 (2) Å
α = 90.96 (3)°
β = 91.84 (3)°
γ = 98.60 (3)°
V = 654.7 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 K
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.973, T_{max} = 0.991
2703 measured reflections
2419 independent reflections
1627 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.169
S = 1.00
2419 reflections
163 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1B···O2	0.82	2.30	2.738 (2)	114
O1—H1B···O2^{i}	0.82	2.15	2.840 (2)	142
O2—H2A···O3^{ii}	0.82	1.98	2.800 (2)	173
C5—H5A···O3^{ii}	0.93	2.52	3.230 (3)	133

Symmetry codes: (i) $-x, -y+3, -z$; (ii) $-x+1, -y+2, -z$.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GW2109).

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Pentyl (*E*)-3-(3,4-dihydroxyphenyl)acrylate

Jun Wang, Shuangshuang Gu, Leixia Zhang, Fuan Wu and Xijie Guo

S1. Comment

Caffeic acid and its esters have been a research hot spot for a long time. These compounds are known to show a variety of biological effects such as anti-tumor, anti-oxidant, and anti-inflammatory activities (Uwai *et al.*, 2008; Buzzi *et al.*, 2009). As a part of our studies into the synthesis of caffeic acid derivatives, the title compound (1) pentyl (*E*)-3-(3,4-dihydroxyphenyl)acrylate was synthesized (Xia *et al.* (2006); Son *et al.* (2011)). We report herein the crystal structure of the title compound.

The molecule of (I) has an *E* configuration (Fig. 1); All non-H atoms of (I) are almost coplanar, with a root mean square deviating from the least-squares plane of 0.04 A°. The bond lengths (Allen *et al.*, 1987) and angles are within normal ranges.

In the crystal structure, hydroxy groups contribute to intermolecular O—H···O interactions (Table 1) link the molecules into ribbons extended in the [110] direction (Fig. 2), in which they may be effective in the stabilization of thestructure. On the other hand, the intramolecular O—H···O H-bond also contribute to the stability of the molecular configuration (Fig. 1 and Table 1).

S2. Experimental

Esterification of caffeic acid with amyl alcohol was performed in a column (inner diameter= 15 mm, length = 200 mm). Caffeic acid (8.95 g, 0.05 mol) was dissolved in amyl alcohol (100 ml). The mixture was stirred at 80°C for 60 minutes and fed from the top of the reactor with syringe pumps. The feed rate of the mixture was fixed at 10.0 ml/h. Cation exchange resin CD-552 particles(5 g) and molecular sieve(5 g) were packed into the middle of the reactor and glass beads of 2 mm in diameter were loaded into the rest of the column. The reaction temperature continued at 90°C for 20 h. The mixture was evaporated to dryness and followed by the addition of ethanol and extracted with chloroform three times. The chloroform extract was dried over evaporated to give a solid residue, and dissolved in ethanol/petroleum ether (1:1) to crystal. The solution was filtered and concentrated to yield a brown crystalline product (5.3 g, 59.2%). Recrystallization from ethanol gave colourless crystal.

S3. Refinement

The H atoms were placed in calculated positions (O—H = 0.82 A ° and C—H = 0.93–0.97 A °) and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2 or 1.5Ueq(O,*C*).

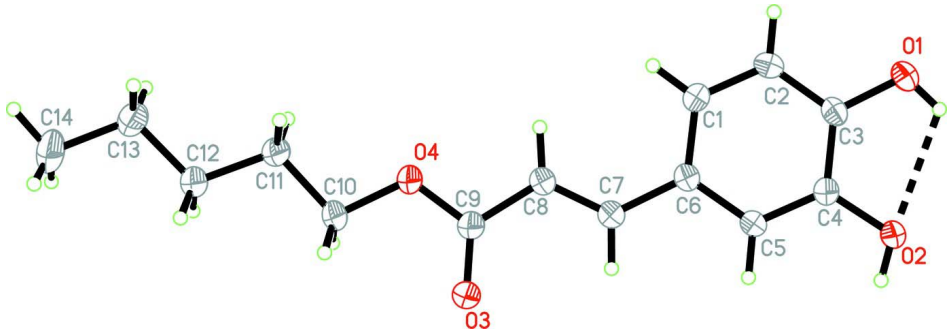

Figure 1

The molecular structure of the title molecule, with the atom numbering scheme. Displacement ellipsoids are drawn at 30% probability levels.

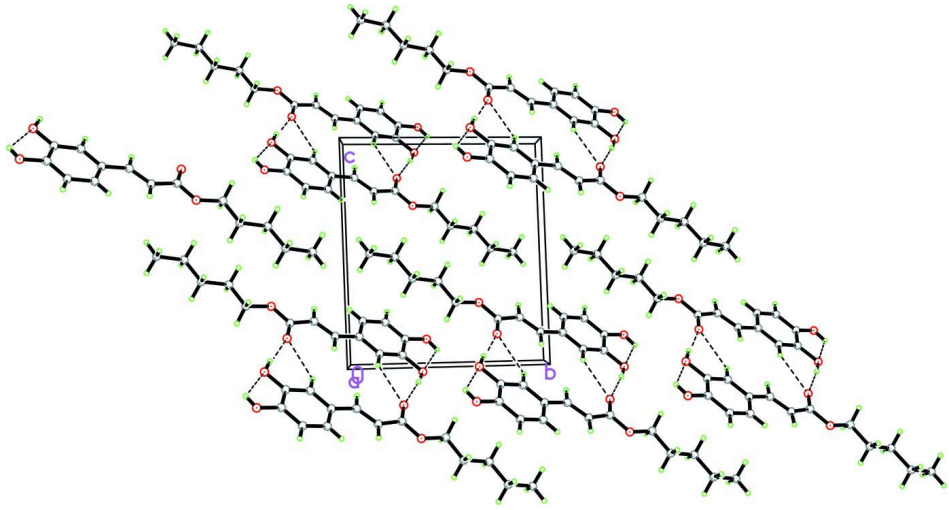

Figure 2

A partial packing diagram. Hydrogen bonds are shown as dashed lines.

Pentyl (*E*)-3-(3,4-dihydroxyphenyl)acrylate

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$V = 654.7\ (2)$ Å^3
$Z = 2$
$F(000) = 268$
$D_x = 1.270$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9–13°$
$\mu = 0.09$ mm^{-1}
$T = 293$ K
Block, colourless
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.973, T_{max} = 0.991
2703 measured reflections
2419 independent reflections
1627 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 25.4°, θ_{min} = 1.7°
h = 0→6
k = −12→12
l = −14→14
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.169
S = 1.00
2419 reflections
163 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2 + 0.040P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
O1	−0.2441 (3)	1.39149 (14)	0.11914 (14)	0.0617 (5)
H1B	−0.1743	1.4410	0.0731	0.093*
C1	−0.1068 (4)	1.08131 (19)	0.20524 (17)	0.0469 (5)
H1A	−0.1712	1.0179	0.2547	0.056*
O2	0.1576 (3)	1.36636 (13)	−0.01491 (12)	0.0535 (4)
H2A	0.2656	1.3441	−0.0559	0.080*
C2	−0.2175 (4)	1.1903 (2)	0.19717 (18)	0.0506 (6)
H2B	−0.3549	1.2002	0.2416	0.061*
C3	−0.1266 (4)	1.28540 (18)	0.12364 (17)	0.0435 (5)
O3	0.4807 (3)	0.73067 (15)	0.14536 (14)	0.0699 (6)
O4	0.2176 (3)	0.65259 (13)	0.27822 (13)	0.0578 (5)
C4	0.0776 (4)	1.26997 (18)	0.05777 (16)	0.0408 (5)
C5	0.1902 (4)	1.16118 (18)	0.06704 (16)	0.0408 (5)
H5A	0.3293	1.1521	0.0233	0.049*
C6	0.0999 (4)	1.06446 (18)	0.14062 (16)	0.0396 (5)

C7	0.2247 (4)	0.95104 (18)	0.14637 (17)	0.0453 (5)
H7A	0.3589	0.9487	0.0980	0.054*
C8	0.1716 (4)	0.85117 (19)	0.21148 (17)	0.0493 (6)
H8A	0.0415	0.8506	0.2624	0.059*
C9	0.3082 (4)	0.74206 (19)	0.20677 (17)	0.0457 (5)
C10	0.3335 (4)	0.53739 (19)	0.28150 (19)	0.0519 (6)
H10A	0.5153	0.5582	0.2982	0.062*
H10B	0.3079	0.4919	0.2091	0.062*
C11	0.2072 (5)	0.4569 (2)	0.37279 (18)	0.0525 (6)
H11A	0.0261	0.4364	0.3542	0.063*
H11B	0.2270	0.5057	0.4437	0.063*
C12	0.3173 (4)	0.3342 (2)	0.38791 (18)	0.0512 (6)
H12A	0.2967	0.2858	0.3169	0.061*
H12B	0.4987	0.3553	0.4055	0.061*
C13	0.1962 (5)	0.2508 (2)	0.4799 (2)	0.0664 (7)
H13A	0.0162	0.2262	0.4609	0.080*
H13B	0.2109	0.2998	0.5506	0.080*
C14	0.3177 (7)	0.1313 (3)	0.4962 (3)	0.0930 (10)
H14A	0.2336	0.0813	0.5549	0.140*
H14B	0.4949	0.1551	0.5172	0.140*
H14C	0.3016	0.0818	0.4268	0.140*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0648 (10)	0.0488 (9)	0.0805 (11)	0.0307 (8)	0.0265 (8)	0.0173 (8)
C1	0.0505 (13)	0.0405 (11)	0.0512 (12)	0.0086 (9)	0.0125 (10)	0.0095 (9)
O2	0.0618 (10)	0.0405 (8)	0.0644 (9)	0.0217 (7)	0.0251 (8)	0.0162 (7)
C2	0.0468 (12)	0.0504 (13)	0.0587 (13)	0.0164 (10)	0.0180 (10)	0.0057 (10)
C3	0.0438 (12)	0.0371 (11)	0.0522 (12)	0.0139 (9)	0.0048 (9)	0.0010 (9)
O3	0.0806 (12)	0.0553 (10)	0.0841 (12)	0.0321 (8)	0.0460 (10)	0.0256 (8)
O4	0.0741 (11)	0.0403 (8)	0.0658 (10)	0.0227 (7)	0.0301 (8)	0.0172 (7)
C4	0.0456 (11)	0.0339 (10)	0.0439 (11)	0.0081 (9)	0.0059 (9)	0.0039 (8)
C5	0.0408 (11)	0.0383 (11)	0.0456 (11)	0.0116 (9)	0.0091 (9)	0.0025 (9)
C6	0.0427 (11)	0.0342 (10)	0.0430 (10)	0.0089 (8)	0.0040 (9)	0.0008 (8)
C7	0.0488 (12)	0.0400 (11)	0.0492 (11)	0.0114 (9)	0.0116 (10)	0.0039 (9)
C8	0.0573 (13)	0.0371 (11)	0.0569 (13)	0.0147 (10)	0.0189 (11)	0.0065 (10)
C9	0.0515 (13)	0.0392 (11)	0.0482 (11)	0.0099 (9)	0.0112 (10)	0.0048 (9)
C10	0.0640 (14)	0.0362 (11)	0.0601 (13)	0.0190 (10)	0.0157 (11)	0.0082 (10)
C11	0.0644 (15)	0.0430 (12)	0.0534 (12)	0.0151 (10)	0.0181 (11)	0.0072 (10)
C12	0.0580 (14)	0.0430 (12)	0.0555 (13)	0.0142 (10)	0.0090 (11)	0.0084 (10)
C13	0.0863 (19)	0.0551 (14)	0.0610 (14)	0.0161 (13)	0.0184 (13)	0.0158 (11)
C14	0.126 (3)	0.0666 (17)	0.095 (2)	0.0349 (17)	0.0191 (19)	0.0386 (15)

Geometric parameters (Å, º)

O1—C3	1.363 (2)	C7—H7A	0.9300
O1—H1B	0.8200	C8—C9	1.452 (3)

C1—C2	1.372 (3)	C8—H8A	0.9300
C1—C6	1.388 (3)	C10—C11	1.498 (3)
C1—H1A	0.9300	C10—H10A	0.9700
O2—C4	1.371 (2)	C10—H10B	0.9700
O2—H2A	0.8200	C11—C12	1.512 (3)
C2—C3	1.382 (3)	C11—H11A	0.9700
C2—H2B	0.9300	C11—H11B	0.9700
C3—C4	1.382 (3)	C12—C13	1.509 (3)
O3—C9	1.205 (2)	C12—H12A	0.9700
O4—C9	1.324 (2)	C12—H12B	0.9700
O4—C10	1.444 (2)	C13—C14	1.513 (3)
C4—C5	1.377 (3)	C13—H13A	0.9700
C5—C6	1.393 (3)	C13—H13B	0.9700
C5—H5A	0.9300	C14—H14A	0.9600
C6—C7	1.455 (3)	C14—H14B	0.9600
C7—C8	1.318 (3)	C14—H14C	0.9600

C3—O1—H1B	109.5	O4—C10—C11	106.74 (16)
C2—C1—C6	120.89 (18)	O4—C10—H10A	110.4
C2—C1—H1A	119.6	C11—C10—H10A	110.4
C6—C1—H1A	119.6	O4—C10—H10B	110.4
C4—O2—H2A	109.5	C11—C10—H10B	110.4
C1—C2—C3	120.64 (18)	H10A—C10—H10B	108.6
C1—C2—H2B	119.7	C10—C11—C12	112.26 (17)
C3—C2—H2B	119.7	C10—C11—H11A	109.2
O1—C3—C2	118.08 (17)	C12—C11—H11A	109.2
O1—C3—C4	122.54 (18)	C10—C11—H11B	109.2
C2—C3—C4	119.38 (18)	C12—C11—H11B	109.2
C9—O4—C10	117.71 (15)	H11A—C11—H11B	107.9
O2—C4—C5	123.18 (17)	C13—C12—C11	113.87 (18)
O2—C4—C3	116.98 (17)	C13—C12—H12A	108.8
C5—C4—C3	119.85 (18)	C11—C12—H12A	108.8
C4—C5—C6	121.30 (17)	C13—C12—H12B	108.8
C4—C5—H5A	119.4	C11—C12—H12B	108.8
C6—C5—H5A	119.4	H12A—C12—H12B	107.7
C1—C6—C5	117.94 (18)	C12—C13—C14	112.6 (2)
C1—C6—C7	123.06 (18)	C12—C13—H13A	109.1
C5—C6—C7	119.01 (17)	C14—C13—H13A	109.1
C8—C7—C6	128.16 (19)	C12—C13—H13B	109.1
C8—C7—H7A	115.9	C14—C13—H13B	109.1
C6—C7—H7A	115.9	H13A—C13—H13B	107.8
C7—C8—C9	122.58 (19)	C13—C14—H14A	109.5
C7—C8—H8A	118.7	C13—C14—H14B	109.5
C9—C8—H8A	118.7	H14A—C14—H14B	109.5
O3—C9—O4	122.77 (19)	C13—C14—H14C	109.5
O3—C9—C8	125.58 (19)	H14A—C14—H14C	109.5
O4—C9—C8	111.64 (17)	H14B—C14—H14C	109.5

C6—C1—C2—C3	0.5 (3)	C4—C5—C6—C7	179.50 (18)
C1—C2—C3—O1	179.97 (19)	C1—C6—C7—C8	−1.5 (4)
C1—C2—C3—C4	0.1 (3)	C5—C6—C7—C8	178.6 (2)
O1—C3—C4—O2	−0.8 (3)	C6—C7—C8—C9	178.43 (19)
C2—C3—C4—O2	179.06 (18)	C10—O4—C9—O3	0.1 (3)
O1—C3—C4—C5	179.26 (18)	C10—O4—C9—C8	179.17 (18)
C2—C3—C4—C5	−0.8 (3)	C7—C8—C9—O3	0.3 (4)
O2—C4—C5—C6	−178.85 (17)	C7—C8—C9—O4	−178.7 (2)
C3—C4—C5—C6	1.0 (3)	C9—O4—C10—C11	176.82 (18)
C2—C1—C6—C5	−0.4 (3)	O4—C10—C11—C12	−178.17 (18)
C2—C1—C6—C7	179.72 (19)	C10—C11—C12—C13	179.56 (19)
C4—C5—C6—C1	−0.4 (3)	C11—C12—C13—C14	−177.7 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*B*···O2	0.82	2.30	2.738 (2)	114
O1—H1*B*···O2^{i}	0.82	2.15	2.840 (2)	142
O2—H2*A*···O3^{ii}	0.82	1.98	2.800 (2)	173
C5—H5*A*···O3^{ii}	0.93	2.52	3.230 (3)	133

Symmetry codes: (i) −*x*, −*y*+3, −*z*; (ii) −*x*+1, −*y*+2, −*z*.