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

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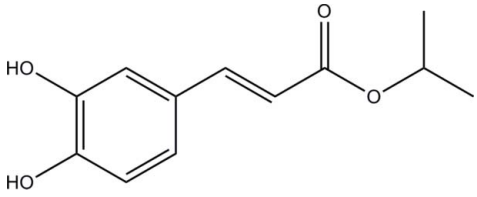
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.045; wR factor = 0.151; data-to-parameter ratio = 13.6.

In the title compound, $C_{12}H_{14}O_4$, a derivative of caffeic acid [(*E*)-3-(3,4-dihydroxyphenyl)-2-propenoic acid], an intramolecular O—H···O hydrogen bond forms an S(5) ring. In the crystal, intermolecular O—H···O hydrogen bonds link molecules into chains propagating in [110].

Related literature

For the properties of caffeate esters, see: Uwai *et al.* (2008); Buzzi *et al.* (2009); Calheiros *et al.*(2008); Xia *et al.*(2008). For the preparation of the title compound, see: Hu *et al.* (2006). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{12}H_{14}O_4$
M_r = 222.23
Triclinic, $P\overline{1}$
a = 5.8830 (14) Å
b = 9.644 (2) Å
c = 11.428 (3) Å
α = 65.690 (2)°
β = 89.370 (3)°
γ = 81.018 (3)°
V = 582.6 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 296 K
0.31 × 0.27 × 0.19 mm

Data collection

Bruker APEXII CCD diffractometer
2938 measured reflections
2042 independent reflections
1436 reflections with $I > 2\sigma(I)$
R_{int} = 0.015

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.151
S = 1.05
2042 reflections
150 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O2^{i}	0.82	1.92	2.725 (2)	169
O4—H4···O3^{ii}	0.82	2.09	2.792 (2)	143
O4—H4···O3	0.82	2.28	2.721 (2)	114

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; 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: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5158).

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

Xu-Ji Shen, Shi-Yu Liu, Pu Jia, Shi-Xiang Wang and Xiao-Hui Zheng

S1. Comment

Caffeate esters have been shown to have, an inhibitory effect on lipopolysaccharide-induced nitric oxide production, antinociceptive properties, and anticancer activity (Uwai *et al.*, 2008; Buzzi *et al.*, 2009; Calheiros *et al.*, 2008; Xia *et al.*, 2008).

The molecular structure of the title compound (I) is shown in Fig. 1. An intramolecular O-H···O hydrogen bond forms an S(5) ring (Bernstein *et al.*, 1995). In the crystal structure, intermolecular O—H···O hydrogen bonds link molecules into one-dimensional chains along [110] (see Fig .2).

S2. Experimental

The synthesis follows the method of Hu *et al.* (2006). To a solution of 0.02 *M* caffeic acid in 120 ml of 2-propanol at room temperature, 0.2 *M* HCl in 2-propanol was added. After the solution had been allowed to stir and reflux for 16 h, the solvent was removed under reduced pressure. The residue was extracted with ethyl acetate three times and filtered. The filtrate was washed successively with dilute saturated aqueous $NaHCO_3$ solution, saturated aqueous NaCl, dried over $MgSO_4$, and evaporated. The crude products were purified by chromatography (SiO_2; elution with petroleum ether-aceto-acetate, 6:1 *v*/*v*). Yield 80%. X-ray quality crystals were grown from a solution of the title compound in acetone and toluene at room temperature. Spectroscopic analysis: IR(KBr, χm^{-1}): 3464, 3310, 2973, 2926, 1675, 1629, 1599, 1529, 1370, 1276; ^{1}H NMR (DMSO, δ, p.p.m.): 9.606 (s, 1 H), 9.146 (s, 1 H), 7.424—7.464 (d, 1 H), 7.029 (s, 1 H), 6.990—7.011 (d, 1 H), 6.742—6.762 (d, 1 H), 6.246—6.206(d, 1 H), 4.951—5.013(m, 1 H), 1.245 (s, 3 H), 1.229 (s, 3 H).

S3. Refinement

H atoms were placed in calculated positions with O—H = 0.82Å and C—H = 0.93–0.96 Å with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C_{methyl}, O).

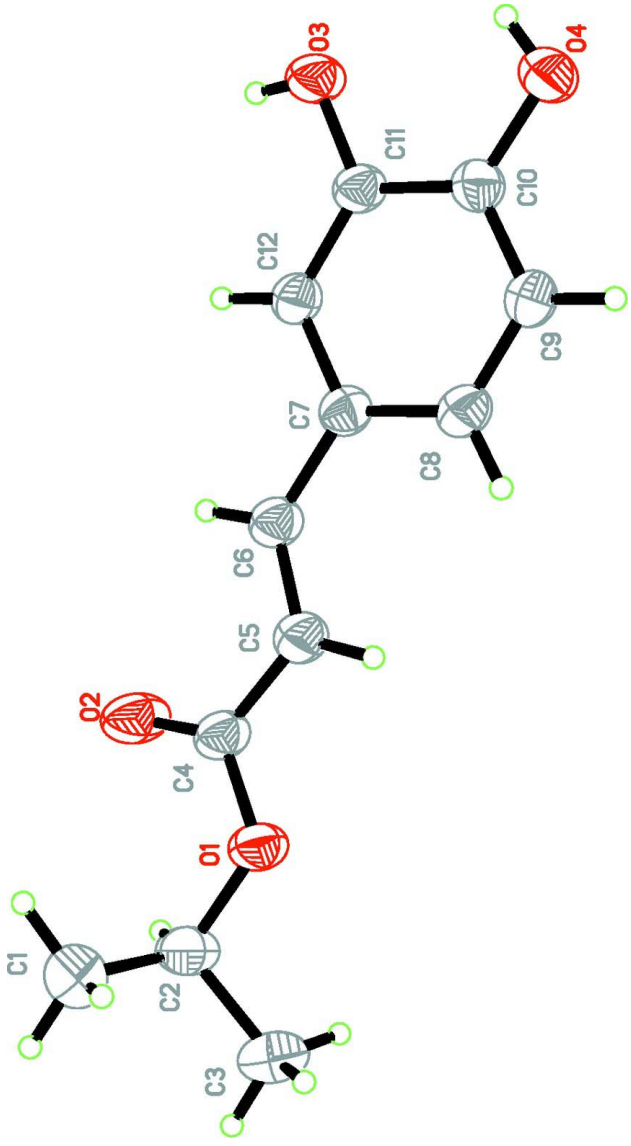

Figure 1

The molecular structure of (I) with the atom numbering scheme, showing displacement ellipsoids at the 30% probability level.

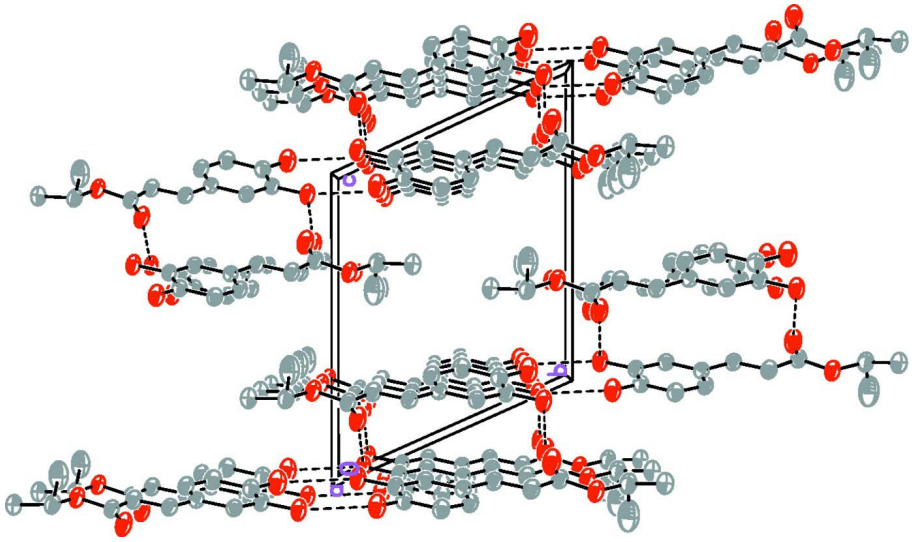

Figure 2

The packing of (I), viewed along the *a* axis. The dashed lines show the donor-acceptor distances of O—H···O hydrogen bonds. H atoms are not shown.

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

Crystal data

$C_{12}H_{14}O_4$
$M_r = 222.23$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 5.8830\ (14)$ Å
$b = 9.644\ (2)$ Å
$c = 11.428\ (3)$ Å
$\alpha = 65.690\ (2)°$
$\beta = 89.370\ (3)°$
$\gamma = 81.018\ (3)°$
$V = 582.6\ (2)$ Å^3
$Z = 2$
$F(000) = 236$
$D_x = 1.267$ Mg m^{-3}
Melting point: 415 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 966 reflections
$\theta = 2.4–26.5°$
$\mu = 0.10$ mm^{-1}
$T = 296$ K
Block, colorless
0.31 × 0.27 × 0.19 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
2938 measured reflections
2042 independent reflections
1436 reflections with $I > 2\sigma(I)$
$R_{int} = 0.015$
$\theta_{max} = 25.1°$, $\theta_{min} = 2.0°$
$h = -7\rightarrow4$
$k = -11\rightarrow11$
$l = -13\rightarrow13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.151$
$S = 1.05$
2042 reflections
150 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
$w = 1/[\sigma^2(F_o^2) + (0.0822P)^2 + 0.0498P]$ 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* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.059 (11)

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	0.2370 (2)	−0.06896 (13)	0.33802 (11)	0.0681 (4)
O2	−0.0059 (3)	0.11022 (15)	0.18106 (14)	0.0914 (6)
O3	0.3441 (2)	0.87422 (13)	−0.01294 (13)	0.0716 (4)
H3	0.2302	0.8794	−0.0562	0.107*
O4	0.7644 (2)	0.80457 (15)	0.11324 (14)	0.0808 (5)
H4	0.6907	0.8884	0.0653	0.121*
C1	−0.0940 (5)	−0.1810 (3)	0.4336 (3)	0.1171 (10)
H1A	−0.0341	−0.1952	0.5163	0.176*
H1B	−0.1815	−0.2610	0.4439	0.176*
H1C	−0.1918	−0.0823	0.3940	0.176*
C2	0.1019 (4)	−0.1880 (2)	0.34997 (19)	0.0718 (6)
H2	0.0411	−0.1707	0.2647	0.086*
C3	0.2645 (4)	−0.3381 (2)	0.4062 (2)	0.0847 (7)
H3A	0.3904	−0.3349	0.3515	0.127*
H3B	0.1847	−0.4205	0.4132	0.127*
H3C	0.3228	−0.3552	0.4900	0.127*
C4	0.1664 (3)	0.0739 (2)	0.25094 (17)	0.0612 (5)
C5	0.3170 (3)	0.1807 (2)	0.25085 (17)	0.0631 (5)
H5	0.4498	0.1430	0.3053	0.076*
C6	0.2693 (3)	0.3291 (2)	0.17549 (16)	0.0585 (5)
H6	0.1344	0.3605	0.1232	0.070*
C7	0.4000 (3)	0.45108 (19)	0.16300 (15)	0.0539 (5)
C8	0.6149 (3)	0.4221 (2)	0.22568 (17)	0.0606 (5)
H8	0.6797	0.3213	0.2795	0.073*
C9	0.7324 (3)	0.5406 (2)	0.20894 (17)	0.0639 (5)
H9	0.8752	0.5194	0.2522	0.077*
C10	0.6403 (3)	0.69140 (19)	0.12826 (16)	0.0582 (5)
C11	0.4264 (3)	0.72247 (19)	0.06532 (15)	0.0550 (5)
C12	0.3083 (3)	0.60347 (18)	0.08283 (15)	0.0550 (5)
H12	0.1645	0.6252	0.0403	0.066*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0731 (9)	0.0470 (7)	0.0691 (8)	−0.0095 (6)	−0.0157 (6)	−0.0089 (6)
O2	0.1011 (12)	0.0557 (8)	0.0969 (10)	−0.0118 (8)	−0.0444 (9)	−0.0104 (8)
O3	0.0749 (9)	0.0457 (7)	0.0806 (9)	−0.0074 (6)	−0.0259 (7)	−0.0128 (6)
O4	0.0724 (9)	0.0577 (8)	0.1017 (11)	−0.0153 (7)	−0.0234 (8)	−0.0204 (8)
C1	0.0813 (17)	0.0802 (16)	0.167 (3)	−0.0154 (13)	0.0211 (17)	−0.0286 (17)
C2	0.0847 (14)	0.0521 (11)	0.0697 (12)	−0.0175 (10)	−0.0131 (10)	−0.0139 (9)
C3	0.1079 (18)	0.0533 (11)	0.0819 (14)	−0.0073 (11)	0.0031 (12)	−0.0192 (10)
C4	0.0665 (12)	0.0488 (10)	0.0578 (10)	−0.0044 (9)	−0.0117 (9)	−0.0132 (8)
C5	0.0617 (11)	0.0536 (11)	0.0644 (11)	−0.0074 (9)	−0.0098 (9)	−0.0153 (9)
C6	0.0622 (11)	0.0515 (10)	0.0549 (10)	−0.0062 (8)	−0.0053 (8)	−0.0161 (8)
C7	0.0561 (10)	0.0501 (10)	0.0515 (9)	−0.0055 (8)	−0.0005 (7)	−0.0180 (8)
C8	0.0613 (11)	0.0503 (10)	0.0593 (10)	−0.0018 (8)	−0.0065 (8)	−0.0143 (8)
C9	0.0550 (10)	0.0594 (11)	0.0686 (11)	−0.0038 (9)	−0.0117 (9)	−0.0196 (9)
C10	0.0574 (11)	0.0533 (10)	0.0610 (10)	−0.0099 (8)	−0.0040 (8)	−0.0204 (8)
C11	0.0593 (10)	0.0459 (9)	0.0537 (9)	−0.0051 (8)	−0.0066 (8)	−0.0157 (8)
C12	0.0539 (10)	0.0517 (10)	0.0540 (9)	−0.0049 (8)	−0.0072 (8)	−0.0176 (8)

Geometric parameters (Å, º)

O1—C4	1.327 (2)	C3—H3C	0.9600
O1—C2	1.457 (2)	C4—C5	1.459 (3)
O2—C4	1.210 (2)	C5—C6	1.317 (2)
O3—C11	1.3735 (19)	C5—H5	0.9300
O3—H3	0.8200	C6—C7	1.462 (2)
O4—C10	1.358 (2)	C6—H6	0.9300
O4—H4	0.8200	C7—C8	1.391 (3)
C1—C2	1.500 (3)	C7—C12	1.395 (2)
C1—H1A	0.9600	C8—C9	1.372 (3)
C1—H1B	0.9600	C8—H8	0.9300
C1—H1C	0.9600	C9—C10	1.386 (2)
C2—C3	1.496 (3)	C9—H9	0.9300
C2—H2	0.9800	C10—C11	1.384 (2)
C3—H3A	0.9600	C11—C12	1.377 (2)
C3—H3B	0.9600	C12—H12	0.9300
C4—O1—C2	118.69 (14)	C6—C5—C4	121.51 (17)
C11—O3—H3	109.5	C6—C5—H5	119.2
C10—O4—H4	109.5	C4—C5—H5	119.2
C2—C1—H1A	109.5	C5—C6—C7	128.67 (18)
C2—C1—H1B	109.5	C5—C6—H6	115.7
H1A—C1—H1B	109.5	C7—C6—H6	115.7
C2—C1—H1C	109.5	C8—C7—C12	118.10 (16)
H1A—C1—H1C	109.5	C8—C7—C6	122.98 (16)
H1B—C1—H1C	109.5	C12—C7—C6	118.91 (16)
O1—C2—C3	106.18 (17)	C9—C8—C7	120.69 (17)

O1—C2—C1	108.92 (17)	C9—C8—H8	119.7
C3—C2—C1	112.69 (18)	C7—C8—H8	119.7
O1—C2—H2	109.7	C8—C9—C10	120.69 (17)
C3—C2—H2	109.7	C8—C9—H9	119.7
C1—C2—H2	109.7	C10—C9—H9	119.7
C2—C3—H3A	109.5	O4—C10—C11	122.00 (15)
C2—C3—H3B	109.5	O4—C10—C9	118.59 (16)
H3A—C3—H3B	109.5	C11—C10—C9	119.41 (16)
C2—C3—H3C	109.5	O3—C11—C12	123.54 (15)
H3A—C3—H3C	109.5	O3—C11—C10	116.67 (15)
H3B—C3—H3C	109.5	C12—C11—C10	119.79 (15)
O2—C4—O1	123.04 (17)	C11—C12—C7	121.31 (16)
O2—C4—C5	124.39 (16)	C11—C12—H12	119.3
O1—C4—C5	112.57 (15)	C7—C12—H12	119.3
C4—O1—C2—C3	155.38 (16)	C7—C8—C9—C10	−0.6 (3)
C4—O1—C2—C1	−83.0 (2)	C8—C9—C10—O4	−179.05 (16)
C2—O1—C4—O2	0.3 (3)	C8—C9—C10—C11	0.8 (3)
C2—O1—C4—C5	179.43 (15)	O4—C10—C11—O3	−0.4 (3)
O2—C4—C5—C6	2.5 (3)	C9—C10—C11—O3	179.78 (16)
O1—C4—C5—C6	−176.64 (16)	O4—C10—C11—C12	179.41 (17)
C4—C5—C6—C7	−179.87 (16)	C9—C10—C11—C12	−0.4 (3)
C5—C6—C7—C8	5.7 (3)	O3—C11—C12—C7	179.71 (14)
C5—C6—C7—C12	−175.59 (17)	C10—C11—C12—C7	−0.1 (3)
C12—C7—C8—C9	0.1 (3)	C8—C7—C12—C11	0.2 (3)
C6—C7—C8—C9	178.84 (17)	C6—C7—C12—C11	−178.53 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O2^{i}	0.82	1.92	2.725 (2)	169
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