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

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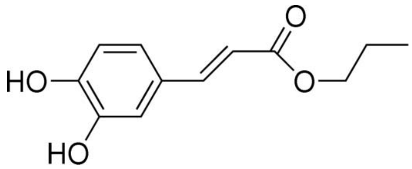
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.034; wR factor = 0.101; data-to-parameter ratio = 16.1.

The title compound, $C_{12}H_{14}O_4$, was prepared from the condensation reaction of 3,4-dihydroxybenzaldehyde, Meldrum's acid and propan-1-ol. The acyclic double bond is E configured. Intra- and intermolecular hydrogen bonds stabilize the molecular conformation and the crystal structure.

Related literature

For related literature, see: Chen *et al.* (1999); Hu *et al.* (2006); Son & Lewis (2002).



Experimental

Crystal data

$C_{12}H_{14}O_4$
M_r = 222.23
Monoclinic, $C2/c$
a = 18.883 (3) Å
b = 10.9652 (16) Å
c = 12.4783 (18) Å
β = 116.948 (2)°
V = 2303.2 (6) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 296 (2) K
0.30 × 0.20 × 0.15 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.972, T_{max} = 0.983
7001 measured reflections
2495 independent reflections
1949 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.101
S = 1.03
2495 reflections
155 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1X···O2	0.853 (14)	2.298 (19)	2.7109 (14)	110.0 (15)
O1—H1X···O2^{i}	0.853 (14)	2.057 (17)	2.7956 (14)	144.4 (18)
O2—H2X···O3^{ii}	0.840 (13)	1.849 (14)	2.6878 (13)	175.9 (17)
C8—H8···O1^{iii}	0.93	2.51	3.3979 (16)	160

Symmetry codes: (i) $-x+\frac{1}{2}, -y+\frac{5}{2}, -z$; (ii) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (iii) $x, -y+2, z+\frac{1}{2}$.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2003); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; 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: BT2339).

References

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supplementary materials

Acta Cryst. (2007). E**63**, o2482 [**doi:10.1107/S1600536807017849**]

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

C.-N. Xia, W.-X. Hu and W. Zhou

Comment

Caffeic acid and its derivatives are widely distributed in the plant kingdom (Chen *et al.*, 1999). The compounds are known to have antiatherosclerotic, antibacterial, anti-inflammatory, antiproliferative, immunostimulatory, antioxidative, antiviral and neuroprotective properties (Son & Lewis, 2002). In a continuation of our work on the structure–activity relationship of caffeic acid derivatives, we have obtained the title light-brown crystalline compound, (I), synthesized by a one-pot method (Hu *et al.*, 2006).

The molecular structure of (I) is illustrated in Fig. 1. The acyclic double bond is E configured. The crystal packing (Fig. 2) is stabilized by intermolecular O—H···O and C—H···O hydrogen bonds (Table 1). The molecules of the caffeic acid ester form stacks along the a axis in a head-to-head manner

to form a dimeric structure.

Experimental

Compound (I) was obtained by the method of Hu *et al.* (2006). Crystals suitable for structure analysis were obtained by slow evaporation of a solution in a mixture of tetrafuaran and acetone (2:1 v/v) as light-brown crystalline prisms.

Refinement

The hydroxyl H atoms were located in difference Fourier maps and refined isotropically, with the O—H bond restrained to 0.82 (2) Å. Carbon-bound H atoms were added in calculated positions and refined using a riding model, with

C—H = 0.93–0.98 Å, and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C) or 1.5$U_{\rm eq}$($C_{\rm methyl}$). The methyl group was allowed to rotate but not to tip.

Figures

Fig. 1. The structure of (I), shown with 30% probability displacement ellipsoids.

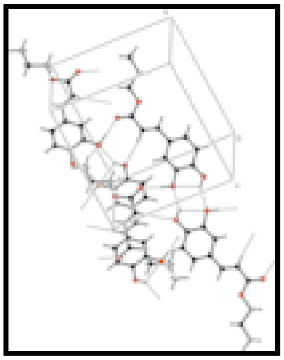

Fig. 2. A packing diagram for (I), showing the hydrogen-bond pattern (dashed lines).

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

Crystal data

$C_{12}H_{14}O_4$
$M_r = 222.23$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 18.883\ (3)$ Å
$b = 10.9652\ (16)$ Å
$c = 12.4783\ (18)$ Å
$\beta = 116.948\ (2)$°
$V = 2303.2\ (6)$ Å^3
$Z = 8$

$F_{000} = 944$
$D_x = 1.282$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 2969 reflections
$\theta = 2.2$–27.9°
$\mu = 0.10$ mm^{-1}
$T = 296\ (2)$ K
Block, light brown
0.30 × 0.20 × 0.15 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Monochromator: graphite
$T = 296(2)$ K
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
$T_{min} = 0.972$, $T_{max} = 0.983$
7001 measured reflections

2495 independent reflections
1949 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 27.0$°
$\theta_{min} = 2.2$°
$h = -24 \rightarrow 23$
$k = -13 \rightarrow 14$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.035$

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0484P)^2 + 0.7676P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$

$wR(F^2)$ = 0.101

S = 1.03

2495 reflections

155 parameters

2 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

$\Delta\rho_{\max}$ = 0.18 e Å^{-3}

$\Delta\rho_{\min}$ = −0.15 e Å^{-3}

Extinction correction: SHELXTL (Bruker, 2003), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0041 (6)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 > 2sigma(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 (Å^2)

	x	y	z	$U_{\rm iso}$*/$U_{\rm eq}$
O1	0.20300 (7)	1.15279 (9)	−0.16760 (9)	0.0566 (3)
H1X	0.2248 (10)	1.2043 (16)	−0.1107 (15)	0.084 (6)*
O2	0.25503 (6)	1.11303 (8)	0.07069 (8)	0.0499 (3)
H2X	0.2829 (9)	1.0814 (15)	0.1383 (13)	0.067 (5)*
O3	0.15173 (6)	0.50624 (8)	0.21782 (8)	0.0529 (3)
O4	0.10009 (6)	0.47109 (8)	0.02084 (8)	0.0505 (3)
C1	0.13035 (8)	0.84559 (12)	−0.17544 (11)	0.0480 (3)
H1	0.1023	0.7866	−0.2326	0.058*
C2	0.14602 (8)	0.95669 (12)	−0.21239 (11)	0.0491 (3)
H2	0.1285	0.9720	−0.2938	0.059*
C3	0.18741 (7)	1.04461 (11)	−0.12904 (11)	0.0410 (3)
C4	0.21382 (7)	1.02111 (10)	−0.00688 (10)	0.0366 (3)
C5	0.19794 (7)	0.91036 (10)	0.02980 (10)	0.0368 (3)
H5	0.2154	0.8954	0.1113	0.044*
C6	0.15580 (7)	0.82035 (10)	−0.05418 (11)	0.0381 (3)
C7	0.13882 (7)	0.70091 (11)	−0.01975 (11)	0.0398 (3)
H7	0.1140	0.6448	−0.0816	0.048*
C8	0.15487 (7)	0.66342 (11)	0.08999 (11)	0.0415 (3)
H8	0.1793	0.7180	0.1533	0.050*
C9	0.13650 (7)	0.54176 (11)	0.11710 (11)	0.0407 (3)
C10	0.08026 (8)	0.34821 (12)	0.04033 (12)	0.0487 (3)

H10A	0.0427	0.3503	0.0735	0.058*
H10B	0.1276	0.3057	0.0965	0.058*
C11	0.04452 (9)	0.28422 (13)	−0.07845 (13)	0.0552 (4)
H11A	0.0826	0.2829	−0.1107	0.066*
H11B	−0.0020	0.3287	−0.1344	0.066*
C12	0.02101 (10)	0.15461 (14)	−0.06656 (18)	0.0691 (5)
H12A	0.0674	0.1095	−0.0139	0.104*
H12B	−0.0029	0.1166	−0.1443	0.104*
H12C	−0.0163	0.1557	−0.0340	0.104*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0909 (8)	0.0354 (5)	0.0421 (5)	−0.0036 (5)	0.0288 (5)	0.0071 (4)
O2	0.0726 (6)	0.0288 (4)	0.0381 (5)	−0.0043 (4)	0.0161 (5)	0.0008 (4)
O3	0.0723 (6)	0.0436 (5)	0.0362 (5)	−0.0049 (4)	0.0188 (5)	0.0033 (4)
O4	0.0716 (6)	0.0367 (5)	0.0377 (5)	−0.0134 (4)	0.0200 (4)	−0.0017 (4)
C1	0.0613 (8)	0.0415 (7)	0.0352 (6)	−0.0052 (6)	0.0166 (6)	−0.0055 (5)
C2	0.0674 (9)	0.0438 (7)	0.0314 (6)	0.0013 (6)	0.0183 (6)	0.0040 (5)
C3	0.0537 (7)	0.0319 (6)	0.0388 (6)	0.0062 (5)	0.0220 (6)	0.0060 (5)
C4	0.0456 (6)	0.0281 (5)	0.0352 (6)	0.0041 (5)	0.0174 (5)	−0.0005 (4)
C5	0.0469 (6)	0.0323 (6)	0.0311 (6)	0.0040 (5)	0.0175 (5)	0.0018 (4)
C6	0.0445 (6)	0.0331 (6)	0.0368 (6)	0.0017 (5)	0.0186 (5)	0.0001 (5)
C7	0.0462 (7)	0.0332 (6)	0.0387 (6)	−0.0029 (5)	0.0179 (5)	−0.0039 (5)
C8	0.0505 (7)	0.0334 (6)	0.0391 (6)	−0.0027 (5)	0.0191 (5)	−0.0044 (5)
C9	0.0461 (7)	0.0368 (6)	0.0361 (6)	0.0006 (5)	0.0159 (5)	0.0000 (5)
C10	0.0579 (8)	0.0363 (7)	0.0473 (7)	−0.0085 (6)	0.0198 (6)	0.0019 (5)
C11	0.0589 (8)	0.0487 (8)	0.0561 (8)	−0.0080 (6)	0.0244 (7)	−0.0092 (6)
C12	0.0677 (10)	0.0474 (8)	0.0927 (12)	−0.0124 (7)	0.0367 (9)	−0.0176 (8)

Geometric parameters (Å, °)

O1—C3	1.3615 (15)	C5—H5	0.9300
O1—H1X	0.853 (14)	C6—C7	1.4586 (16)
O2—C4	1.3696 (14)	C7—C8	1.3265 (17)
O2—H2X	0.840 (13)	C7—H7	0.9300
O3—C9	1.2192 (14)	C8—C9	1.4563 (17)
O4—C9	1.3295 (15)	C8—H8	0.9300
O4—C10	1.4482 (15)	C10—C11	1.4960 (19)
C1—C2	1.3813 (19)	C10—H10A	0.9700
C1—C6	1.3921 (17)	C10—H10B	0.9700
C1—H1	0.9300	C11—C12	1.516 (2)
C2—C3	1.3731 (18)	C11—H11A	0.9700
C2—H2	0.9300	C11—H11B	0.9700
C3—C4	1.3963 (17)	C12—H12A	0.9599
C4—C5	1.3779 (16)	C12—H12B	0.9599
C5—C6	1.3963 (16)	C12—H12C	0.9599
C3—O1—H1X	112.5 (13)	C7—C8—C9	123.82 (11)

C4—O2—H2X	107.6 (12)	C7—C8—H8	118.1
C9—O4—C10	117.25 (10)	C9—C8—H8	118.1
C2—C1—C6	121.17 (12)	O3—C9—O4	121.93 (11)
C2—C1—H1	119.4	O3—C9—C8	124.22 (11)
C6—C1—H1	119.4	O4—C9—C8	113.84 (10)
C3—C2—C1	120.06 (11)	O4—C10—C11	107.75 (11)
C3—C2—H2	120.0	O4—C10—H10A	110.2
C1—C2—H2	120.0	C11—C10—H10A	110.2
O1—C3—C2	118.95 (11)	O4—C10—H10B	110.2
O1—C3—C4	121.27 (11)	C11—C10—H10B	110.2
C2—C3—C4	119.77 (11)	H10A—C10—H10B	108.5
O2—C4—C5	123.59 (10)	C10—C11—C12	111.31 (13)
O2—C4—C3	116.31 (10)	C10—C11—H11A	109.4
C5—C4—C3	120.10 (11)	C12—C11—H11A	109.4
C4—C5—C6	120.62 (11)	C10—C11—H11B	109.4
C4—C5—H5	119.7	C12—C11—H11B	109.4
C6—C5—H5	119.7	H11A—C11—H11B	108.0
C1—C6—C5	118.29 (11)	C11—C12—H12A	109.5
C1—C6—C7	119.01 (11)	C11—C12—H12B	109.5
C5—C6—C7	122.69 (11)	H12A—C12—H12B	109.5
C8—C7—C6	127.19 (11)	C11—C12—H12C	109.5
C8—C7—H7	116.4	H12A—C12—H12C	109.5
C6—C7—H7	116.4	H12B—C12—H12C	109.5

C6—C1—C2—C3	0.1 (2)	C4—C5—C6—C1	−0.06 (18)
C1—C2—C3—O1	179.16 (12)	C4—C5—C6—C7	178.80 (11)
C1—C2—C3—C4	0.2 (2)	C1—C6—C7—C8	−176.34 (13)
O1—C3—C4—O2	0.75 (17)	C5—C6—C7—C8	4.8 (2)
C2—C3—C4—O2	179.66 (11)	C6—C7—C8—C9	−179.45 (12)
O1—C3—C4—C5	−179.33 (11)	C10—O4—C9—O3	−1.15 (18)
C2—C3—C4—C5	−0.42 (18)	C10—O4—C9—C8	179.58 (11)
O2—C4—C5—C6	−179.75 (11)	C7—C8—C9—O3	179.28 (13)
C3—C4—C5—C6	0.34 (18)	C7—C8—C9—O4	−1.47 (19)
C2—C1—C6—C5	−0.1 (2)	C9—O4—C10—C11	−176.71 (11)
C2—C1—C6—C7	−179.04 (12)	O4—C10—C11—C12	−179.36 (12)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1X···O2	0.853 (14)	2.298 (19)	2.7109 (14)	110.0 (15)
O1—H1X···O2$^{\rm i}$	0.853 (14)	2.057 (17)	2.7956 (14)	144.4 (18)
O2—H2X···O3$^{\rm ii}$	0.840 (13)	1.849 (14)	2.6878 (13)	175.9 (17)
C8—H8···O1$^{\rm iii}$	0.93	2.51	3.3979 (16)	160

Symmetry codes: (i) −*x*+1/2, −*y*+5/2, −*z*; (ii) −*x*+1/2, *y*+1/2, −*z*+1/2; (iii) *x*, −*y*+2, *z*+1/2.

Fig. 1

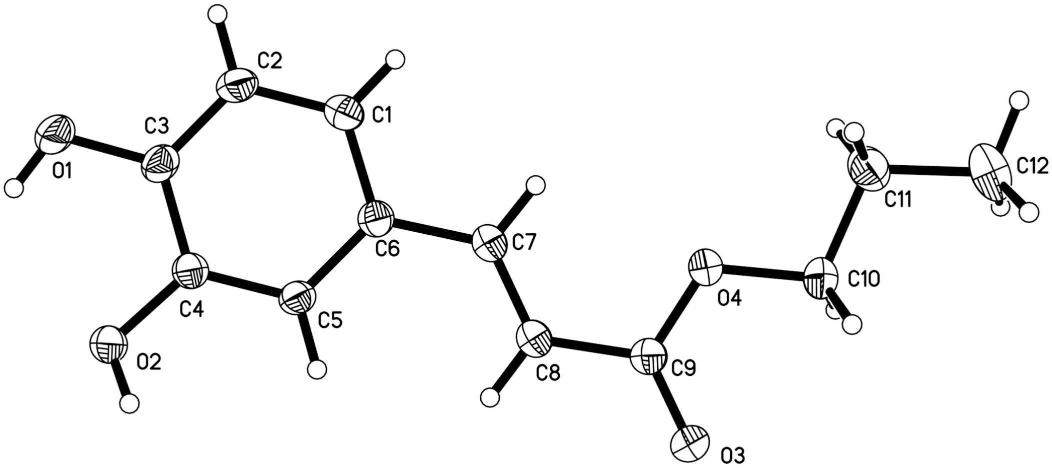

Fig. 2