Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Ethyl (2*Z*)-3-hydroxy-3-(4-nitrophenyl)-prop-2-enoate

Tania N. Hill* and Naadiya Patel

Molecular Sciences Institute, School of Chemistry, University of the Witwatersrand, PO WITS 2050, Johannesburg, South Africa
Correspondence e-mail: tania.hill@gmail.com

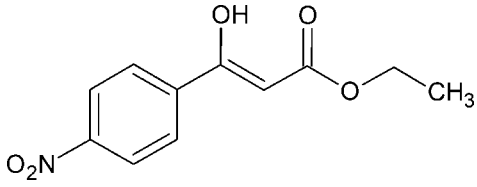
Received 14 April 2014; accepted 22 May 2014

Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.047; wR factor = 0.129; data-to-parameter ratio = 16.8.

The title compound, $C_{11}H_{11}NO_5$, is essentially planar, with an r.m.s. deviation of 0.06 Å. The molecular structure is stabilized by an intramolecular O—H···O hydrogen bond. In the crystal, molecules are linked by two pairs of C—H···O hydrogen bonds, forming sheets, lying parallel to (101), which enclose $R_4^4(26)$ ring motifs.

Related literature

For similar crystal structures, see: Caracelli *et al.* (2010); Yin *et al.* (2004); Syu *et al.* (2010). For geaph-set motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{11}H_{11}NO_5$
M_r = 237.21
Monoclinic, $P2_1/c$
a = 13.0495 (9) Å
b = 10.8363 (6) Å
c = 7.6723 (5) Å
β = 91.268 (4)°
V = 1084.66 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 173 K
0.34 × 0.21 × 0.17 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.962, T_{max} = 0.981
10438 measured reflections
2620 independent reflections
1476 reflections with $I > 2\sigma(I)$
R_{int} = 0.057

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.129
S = 1.03
2620 reflections
156 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 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—H1···O2	0.84	1.87	2.6028 (16)	146
C2—H2···O5^{i}	0.95	2.5	3.362 (2)	150
C8—H8···O2^{ii}	0.95	2.57	3.5050 (17)	170

Symmetry codes: (i) $-x, y+\frac{1}{2}, -z+\frac{5}{2}$; (ii) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT-Plus* (Bruker, 2004); data reduction: *SAINT-Plus* and *XPREP* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

The University of the Witwatersrand and the Molecular Sciences Institute are thanked for providing the infrastructure and financial support. Special thanks go to Dr Andreas Lemmerer of the University of the Witwatersrand for his contributions and insights toward this project.

Supporting information for this paper is available from the IUCr electronic archives (Reference: BX2458).

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supporting information

Acta Cryst. (2014). E**70**, o750 [https://doi.org/10.1107/S1600536814011891]

Ethyl (2*Z*)-3-hydroxy-3-(4-nitrophenyl)prop-2-enoate

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S1. Comment

The molecular structure of (I) is illustrated in Figure 1, and was obtained by recrystallization of the commercially available compound. The title compound, $C_{11}H_{11}NO_5$, consists of a hydroxy (O1) and a *p*-nitrophenyl substituted propenoate.The molecule is essentially planar with an *r.m.s.* deviation of 0.065Å, the larger *r.m.s.* value is as a result of the slight twisting of the substituents on the propenoate backbone, the dihedral angle of the planes of the subsitutents with the propenoate plane were found to be 3.69 (4) ° for the *p*-nitrophenyl and 3.3 (1) ° for the ethyl ester.

The propenoate backbone was observed in the enol tautomeric form with a typical hydrogen bond interaction between the hydroxy (O1) and the carbonyl (O2) with a distance of 2.603 (2) Å. The packing of (I) is seen as parallel sheets (Figure 2) when viewed along the *b*-axis. The crystal and molecular structure is stabilized by two weak C—H···O hydrogen bond interactions with graph-set motif $R_4^4(26)$ (Bernstein, *et al.*, 1995) and one O—H···O intramolecular hydrogen bond interaction respectively, Table 1

S2. Experimental

Ethyl 4-nitrobenzoylacetate was obtained commercially. (I) It was redissolved in warm MeOH and allowed to cool to room terperature. Yellow crystals suitable for single-crystal diffraction were obtained by slow evaporation over a few days.

S3. Refinement

All hydrogen atoms were positioned geometrically and refined using a riding model, with C—H = 0.95 Å U_{iso}(H)= 1.2 U_{eq}(C) for the aromatic H atoms, with C—H = 0.98 Å U_{iso}(H)= 1.5 U_{eq}(C) for methyl H atoms and with O—H = 0.84 Å U_{iso}(H)= 1.5 U_{eq}(O) for the hydroxyl H atoms. The methyl and hydroxyl groups were allowed to rotate with a fixed angle arround the C—C bond to best fit the experimental electron density [HFIX 137 and HFIX 147 in *SHELXL97* (Sheldrick, 2008)].

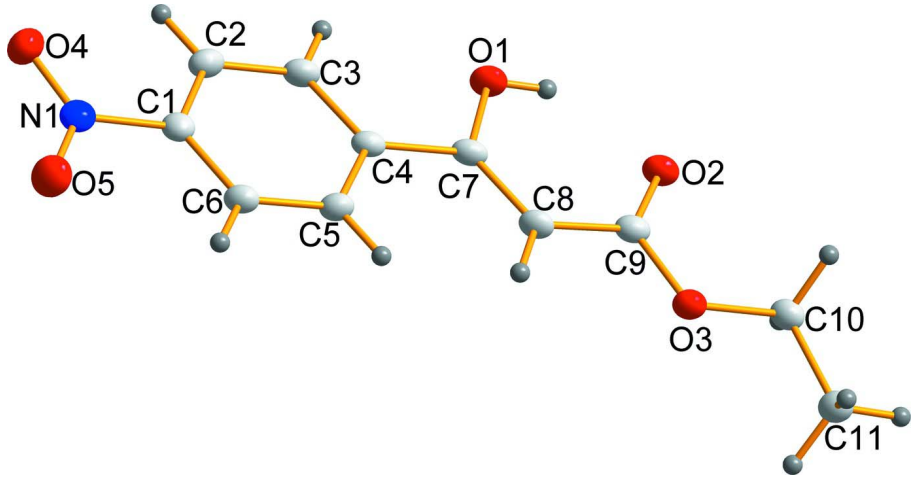

Figure 1

Molecular structure of (I), showing the atom labelling scheme and displacement ellipsoids at 20% probability level. (arbitrary spheres for the H atoms)

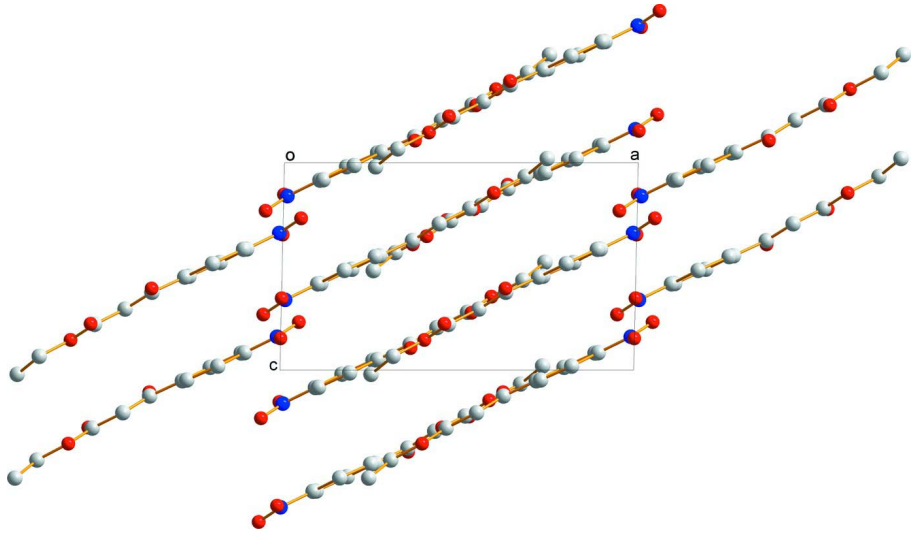

Figure 2

Packing of (I) viewed along the *b*-axis. Hydrogen atoms omitted for clarity.

Ethyl (2*Z*)-3-hydroxy-3-(4-nitrophenyl)prop-2-enoate

Crystal data

$C_{11}H_{11}NO_5$
$M_r = 237.21$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 13.0495\ (9)$ Å
$b = 10.8363\ (6)$ Å
$c = 7.6723\ (5)$ Å
$\beta = 91.268\ (4)°$
$V = 1084.66\ (12)$ Å^3
$Z = 4$

$F(000) = 496$
$D_x = 1.453$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2434 reflections
$\theta = 2.4–24.9°$
$\mu = 0.12$ mm^{-1}
$T = 173$ K
Cuboid, yellow
0.34 × 0.21 × 0.17 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 512 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.962, T_{max} = 0.981

10438 measured reflections
2620 independent reflections
1476 reflections with $I > 2\sigma(I)$
R_{int} = 0.057
θ_{max} = 28°, θ_{min} = 1.6°
h = −17→14
k = −14→12
l = −6→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.129
S = 1.03
2620 reflections
156 parameters

0 restraints
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0505P)^2 + 0.0768P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.10142 (14)	0.26271 (15)	1.0840 (2)	0.0434 (4)
C2	0.11032 (14)	0.38942 (15)	1.0826 (2)	0.0488 (5)
H2	0.0579	0.4402	1.128	0.059*
C3	0.19712 (15)	0.44047 (15)	1.0135 (2)	0.0466 (5)
H3	0.2041	0.5277	1.0101	0.056*
C4	0.27481 (13)	0.36667 (13)	0.9487 (2)	0.0393 (4)
C5	0.26256 (14)	0.23883 (14)	0.9526 (2)	0.0430 (4)
H5	0.3148	0.1872	0.9085	0.052*
C6	0.17583 (14)	0.18653 (14)	1.0193 (2)	0.0446 (4)
H6	0.1675	0.0994	1.0207	0.054*
C7	0.36660 (14)	0.42403 (13)	0.87532 (19)	0.0406 (4)
C8	0.44132 (14)	0.36221 (13)	0.7946 (2)	0.0427 (4)
H8	0.4378	0.2748	0.7861	0.051*
C9	0.52669 (14)	0.42678 (14)	0.7207 (2)	0.0419 (4)
C10	0.68104 (14)	0.41084 (14)	0.5666 (2)	0.0454 (4)
H10A	0.7235	0.4531	0.6569	0.055*
H10B	0.6575	0.4729	0.4799	0.055*
C11	0.74223 (15)	0.31324 (14)	0.4798 (2)	0.0517 (5)
H11A	0.7682	0.2546	0.5675	0.078*
H11B	0.8	0.3513	0.4206	0.078*
H11C	0.6987	0.2696	0.3942	0.078*
N1	0.01035 (12)	0.20690 (15)	1.16117 (19)	0.0539 (4)

O1	0.36768 (10)	0.54740 (9)	0.89365 (15)	0.0503 (4)
H1	0.421	0.5761	0.8495	0.075*
O2	0.53793 (9)	0.54001 (9)	0.72351 (14)	0.0481 (4)
O3	0.59352 (9)	0.35202 (9)	0.64570 (14)	0.0445 (3)
O4	−0.05048 (11)	0.27473 (14)	1.2318 (2)	0.0750 (5)
O5	0.00065 (11)	0.09522 (13)	1.15338 (18)	0.0731 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0507 (12)	0.0458 (10)	0.0334 (9)	−0.0012 (8)	−0.0024 (8)	−0.0002 (7)
C2	0.0536 (13)	0.0475 (10)	0.0451 (10)	0.0114 (9)	−0.0014 (9)	−0.0066 (8)
C3	0.0611 (13)	0.0334 (8)	0.0450 (10)	0.0044 (8)	−0.0037 (9)	−0.0036 (7)
C4	0.0516 (11)	0.0328 (8)	0.0334 (9)	0.0025 (7)	−0.0058 (8)	−0.0022 (6)
C5	0.0543 (12)	0.0355 (8)	0.0392 (10)	0.0044 (8)	0.0025 (8)	−0.0013 (7)
C6	0.0579 (12)	0.0355 (9)	0.0405 (10)	−0.0007 (8)	0.0006 (8)	−0.0012 (7)
C7	0.0579 (12)	0.0276 (8)	0.0358 (9)	0.0026 (7)	−0.0076 (8)	0.0003 (6)
C8	0.0572 (12)	0.0278 (8)	0.0430 (10)	−0.0013 (8)	−0.0022 (8)	0.0002 (7)
C9	0.0558 (12)	0.0342 (9)	0.0354 (10)	0.0021 (8)	−0.0065 (8)	0.0001 (7)
C10	0.0561 (12)	0.0384 (9)	0.0417 (10)	−0.0058 (8)	−0.0016 (8)	0.0028 (7)
C11	0.0601 (13)	0.0435 (9)	0.0519 (11)	−0.0023 (8)	0.0072 (9)	−0.0013 (8)
N1	0.0602 (12)	0.0604 (10)	0.0410 (9)	−0.0031 (8)	−0.0004 (8)	−0.0019 (7)
O1	0.0658 (10)	0.0291 (6)	0.0562 (8)	−0.0011 (5)	0.0053 (6)	−0.0010 (5)
O2	0.0628 (9)	0.0305 (6)	0.0510 (8)	−0.0022 (5)	0.0002 (6)	0.0005 (5)
O3	0.0546 (8)	0.0319 (6)	0.0471 (7)	−0.0002 (5)	0.0036 (6)	0.0002 (5)
O4	0.0628 (10)	0.0830 (10)	0.0799 (11)	0.0008 (8)	0.0175 (8)	−0.0198 (8)
O5	0.0831 (11)	0.0572 (9)	0.0796 (10)	−0.0103 (8)	0.0190 (8)	0.0101 (7)

Geometric parameters (Å, º)

C1—C6	1.376 (2)	C8—C9	1.442 (2)
C1—C2	1.378 (2)	C8—H8	0.95
C1—N1	1.469 (2)	C9—O2	1.2358 (17)
C2—C3	1.377 (2)	C9—O3	1.3304 (19)
C2—H2	0.95	C10—O3	1.4526 (19)
C3—C4	1.392 (2)	C10—C11	1.491 (2)
C3—H3	0.95	C10—H10A	0.99
C4—C5	1.395 (2)	C10—H10B	0.99
C4—C7	1.472 (2)	C11—H11A	0.98
C5—C6	1.375 (2)	C11—H11B	0.98
C5—H5	0.95	C11—H11C	0.98
C6—H6	0.95	N1—O4	1.2179 (18)
C7—O1	1.3443 (17)	N1—O5	1.2181 (18)
C7—C8	1.345 (2)	O1—H1	0.84
C6—C1—C2	122.32 (16)	C7—C8—H8	119.6
C6—C1—N1	118.82 (15)	C9—C8—H8	119.6
C2—C1—N1	118.84 (16)	O2—C9—O3	122.24 (16)

C3—C2—C1	118.27 (16)	O2—C9—C8	124.55 (16)
C3—C2—H2	120.9	O3—C9—C8	113.20 (13)
C1—C2—H2	120.9	O3—C10—C11	108.01 (12)
C2—C3—C4	121.23 (15)	O3—C10—H10A	110.1
C2—C3—H3	119.4	C11—C10—H10A	110.1
C4—C3—H3	119.4	O3—C10—H10B	110.1
C3—C4—C5	118.57 (16)	C11—C10—H10B	110.1
C3—C4—C7	119.95 (14)	H10A—C10—H10B	108.4
C5—C4—C7	121.47 (15)	C10—C11—H11A	109.5
C6—C5—C4	120.87 (15)	C10—C11—H11B	109.5
C6—C5—H5	119.6	H11A—C11—H11B	109.5
C4—C5—H5	119.6	C10—C11—H11C	109.5
C5—C6—C1	118.72 (15)	H11A—C11—H11C	109.5
C5—C6—H6	120.6	H11B—C11—H11C	109.5
C1—C6—H6	120.6	O4—N1—O5	123.60 (17)
O1—C7—C8	122.50 (15)	O4—N1—C1	118.15 (15)
O1—C7—C4	112.74 (14)	O5—N1—C1	118.24 (16)
C8—C7—C4	124.74 (14)	C7—O1—H1	109.5
C7—C8—C9	120.89 (14)	C9—O3—C10	116.26 (12)

C6—C1—C2—C3	0.0 (2)	C5—C4—C7—C8	5.6 (2)
N1—C1—C2—C3	178.54 (14)	O1—C7—C8—C9	−0.9 (2)
C1—C2—C3—C4	−0.8 (2)	C4—C7—C8—C9	177.59 (14)
C2—C3—C4—C5	0.9 (2)	C7—C8—C9—O2	−0.6 (2)
C2—C3—C4—C7	179.86 (14)	C7—C8—C9—O3	−179.97 (14)
C3—C4—C5—C6	−0.1 (2)	C6—C1—N1—O4	173.58 (15)
C7—C4—C5—C6	−179.12 (14)	C2—C1—N1—O4	−5.0 (2)
C4—C5—C6—C1	−0.6 (2)	C6—C1—N1—O5	−5.3 (2)
C2—C1—C6—C5	0.7 (2)	C2—C1—N1—O5	176.14 (15)
N1—C1—C6—C5	−177.83 (14)	O2—C9—O3—C10	−0.1 (2)
C3—C4—C7—O1	5.2 (2)	C8—C9—O3—C10	179.31 (12)
C5—C4—C7—O1	−175.83 (14)	C11—C10—O3—C9	−176.13 (13)
C3—C4—C7—C8	−173.38 (15)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2	0.84	1.87	2.6028 (16)	146
C2—H2···O5^{i}	0.95	2.5	3.362 (2)	150
C8—H8···O2^{ii}	0.95	2.57	3.5050 (17)	170

Symmetry codes: (i) −*x*, *y*+1/2, −*z*+5/2; (ii) −*x*+1, *y*−1/2, −*z*+3/2.