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Methyl 2-[hydroxy(4-nitrophenyl)methyl]acrylate

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Key indicators

Single-crystal X-ray study
T = 183 K
Mean σ(C–C) = 0.003 Å
R factor = 0.043
wR factor = 0.106
Data-to-parameter ratio = 11.2

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

In the crystal structure of the title compound, $C_{11}H_{11}NO_5$, intermolecular O—H···O and C—H···O hydrogen bonds link the molecules into a three-dimensional network structure. An intramolecular O—H···O interaction is also present.

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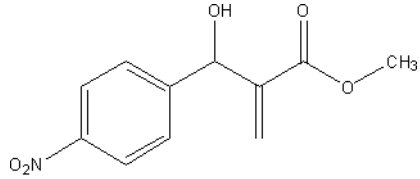
Comment

The title compound, (I), is a useful intermediate in organic synthesis (Basavaiah *et al.*, 1998; Cho *et al.*, 2004; Roy *et al.*, 2000). It was prepared by the Baylis–Hillman reaction, involving the coupling of an activated alkene with a carbon electrophile under the catalysis of a tertiary amine. Although it was synthesized and reported earlier (Cai *et al.*, 2002; Yu *et al.*, 2001), its crystal structure has not yet been published; it is described in this paper.



(I)

Selected geometric parameters of the title compound, (I), are listed in Table 1 and the molecular structure is illustrated in Fig. 1. Atoms C4, C2, C1, O3, C5, C3 and O2 are coplanar, the maximum deviation from the least-squares plane being 0.026 (1) Å for atom C4. The dihedral angle between this plane and the plane of the benzene ring is 77.0 (1)°. Although the molecule contains a chiral atom, C4, the compound crystallizes in a centrosymmetric space group and is thus racemic.

There is a strong O1—H1···O2^{i} [symmetry code: (i) $1-x$, $-y$, $1-z$] hydrogen bond (Table 2) linking the molecules into centrosymmetric dimers with a cyclic $R_2^2(12)$ pattern (Fig. 2). In addition, intermolecular C8—H8···O2^{ii} [symmetry code: (ii) x, $1+y$, z] interactions involve an aromatic CH group as donor and the keto O atom as acceptor. These intermolecular

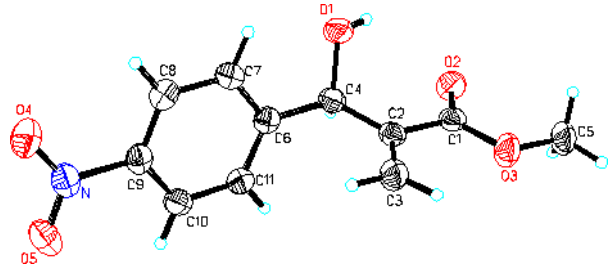


Figure 1
A view of the molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

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contacts play an important role in the packing of the crystal structure. One intramolecular O1—H1···O2 hydrogen bond is also present, forming an S(6) ring pattern.

Experimental

The title compound was synthesized according to a literature method (Cai *et al.*, 2002) using the Baylis–Hillman reaction. 50 mg of (I) were dissolved in $CHCl_3$ (2 ml) and the solution was allowed to evaporate at room temperature over a period of several days. Colorless crystals, suitable for single-crystal X-ray diffraction, were collected.

Crystal data

$C_{11}H_{11}NO_5$
M_r = 237.21
Triclinic, $P\overline{1}$
a = 6.817 (3) Å
b = 8.598 (4) Å
c = 10.457 (5) Å
α = 97.748 (6)°
β = 99.660 (6)°
γ = 110.765 (5)°
V = 552.3 (4) Å^3
Z = 2
D_x = 1.426 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2414 reflections
θ = 2.6–25.7°
μ = 0.11 mm^{-1}
T = 183 (2) K
Block, colorless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.967, T_{max} = 0.978
1930 measured reflections
1755 independent reflections
1284 reflections with $I > 2\sigma(I)$
R_{int} = 0.014
θ_{max} = 25.0°
$h = -7 \rightarrow 6$
$k = -10 \rightarrow 9$
$l = -12 \rightarrow 7$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.107
S = 0.96
1755 reflections
156 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0555P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C4	1.439 (2)	C1—C2	1.499 (3)
O2—C1	1.220 (2)	C2—C3	1.335 (3)
O3—C1	1.343 (2)	C4—C2	1.536 (3)
O3—C5	1.464 (2)	C6—C4	1.528 (3)
O1—C4—C2	110.45 (16)	C1—C2—C4	113.22 (18)
O1—C4—C6	108.33 (15)	C3—C2—C1	121.05 (19)
O2—C1—O3	123.08 (19)	C3—C2—C4	125.72 (19)
O2—C1—C2	122.91 (19)	C6—C4—C2	113.30 (16)
O3—C1—C2	113.97 (18)	C7—C6—C4	121.16 (17)
C1—O3—C5	115.72 (16)	C11—C6—C4	119.47 (17)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2	0.84	2.38	2.916 (2)	122
O1—H1···O2^{i}	0.84	2.17	2.961 (2)	158
C8—H8···O2^{ii}	0.95	2.46	3.286 (3)	145

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

H atoms were placed in calculated positions and allowed to ride on their parent atoms, with U_{iso}(H) values set equal to 1.5U_{eq}(parent atom) for the hydroxyl H atom and Csp^3-bound H atoms, and 1.2U_{eq}(parent atom) for Csp^2-bound H atoms. The O—H distance was fixed at 0.84 Å and the C—H distances were in the range 0.95–0.98 Å.

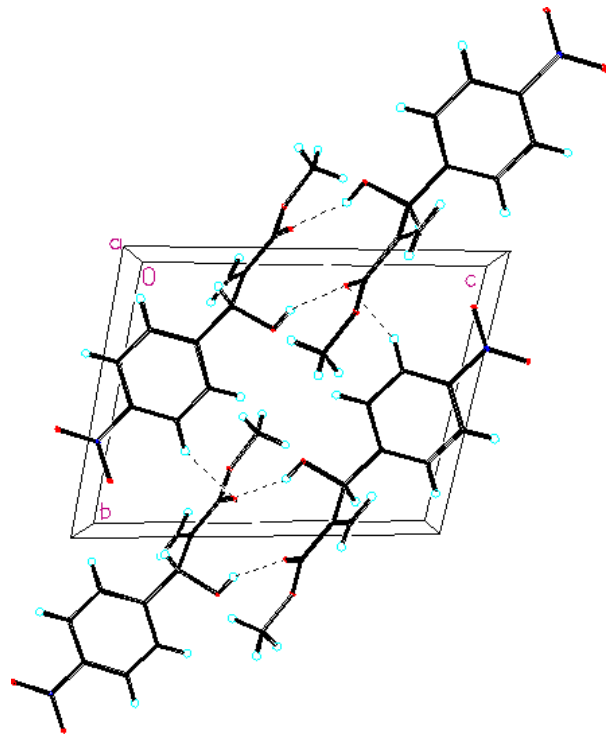

Figure 2
A packing diagram of the title compound, showing the hydrogen-bonded dimers. Hydrogen bonds are indicated by dashed lines.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT* and *SHELXTL* (Bruker, 2000); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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