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Ethyl 3-(1,3-benzodioxol-5-yl)acrylate

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The title compound, $C_{12}H_{12}O_4$, was synthesized by the reaction of 1,3-benzodioxole-5-carbaldehyde with diethoxyphosphorylacetic acid ethyl ester in the presence of sodium hydride in tetrahydrofuran. The 1.3-benzodioxolane system is planar and subtends an angle of 0.82 (8)° with the acrylate unit.

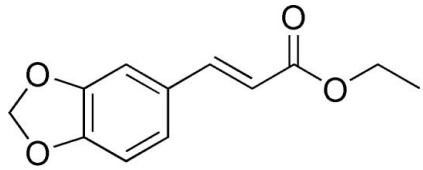
Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.002 Å
R factor = 0.038
wR factor = 0.114
Data-to-parameter ratio = 12.9

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

Comment

The title compound, (I), is an intermediate in the synthesis of selective antagonists of the endothelin receptor (Hoekstra & Maryanoff, 1996). A process has been developed recently to synthesize the pyrrolidine ester using compound (I) as an intermediate (Boyd & Mantei, 1999), and we report its crystal structure here (Fig. 1).



(I)

Both the 1.3-benzodioxolane system (r.m.s. deviation from the mean plane through the two rings is 0.0266 Å) and the acrylate unit (r.m.s. deviation 0.0389 Å from the plane through C8–C10/O3/O4/C11) are essentially planar. The molecule is almost flat, with a dihedral angle of 0.82 (8)° between these planes.

Experimental

A 60% suspension of sodium hydride in mineral oil (3.39 g, 84.6 mmol) was added to a solution of triethyl phosphonoacetate (19.0 g, 84.6 mmol) in tetrahydrofuran (150 ml). The mixture was stirred at room temperature for 20 min and then added dropwise at room temperature to a stirred mixture of 1,3-benzodioxole-5-carbaldehyde (11.4 g, 76.9 mmol) in tetrahydrofuran (15 ml). After the addition was complete, the reaction mixture was stirred at room

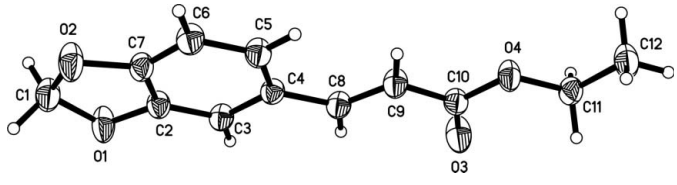


Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

temperature for a further 1 h. Water was added (15 ml) and the organic layer separated. The aqueous layer was extracted with ethyl acetate and the extract was washed with brine and dried over anhydrous magnesium sulfate. The filtrate was concentrated to obtain the product in high yield (88%). Colourless prisms of (I) were grown from ethyl acetate (m.p. 339 K).

Crystal data

$C_{12}H_{12}O_4$
$M_r = 220.22$
Triclinic, $P\overline{1}$
$a = 7.420\ (2)$ Å
$b = 7.673\ (3)$ Å
$c = 10.590\ (4)$ Å
$\alpha = 75.534\ (5)^\circ$
$\beta = 74.094\ (5)^\circ$
$\gamma = 70.637\ (6)^\circ$
$V = 538.6\ (3)$ Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 0.10$ mm^{-1}
$T = 294\ (2)$ K
$0.30 \times 0.28 \times 0.26$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1998)
$T_{min} = 0.970$, $T_{max} = 0.974$
2814 measured reflections
1897 independent reflections
1336 reflections with $I > 2\sigma(I)$
$R_{int} = 0.015$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.114$
$S = 1.05$
1897 reflections
147 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.14$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}

All H atoms bound to carbon were positioned geometrically and refined using a riding model, with C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for aromatic H, C—H = 0.97 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for CH_2, and C—H = 0.96 Å and $U_{iso}(H) = 1.5U_{eq}(C)$ for CH_3.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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