Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*Z*)-Ethyl 3-(1,3-benzodioxol-5-yl)-2-[(triphenylphosphoranylidene)amino]prop-2-enoate

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Received 6 December 2005
Accepted 3 January 2006

Key indicators

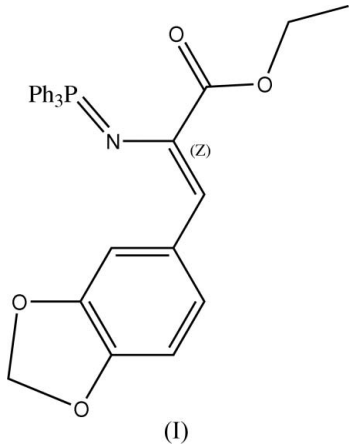
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.006 Å
R factor = 0.072
wR factor = 0.159
Data-to-parameter ratio = 13.7

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

The title compound, $C_{30}H_{26}NO_4P$, contains a planar bicyclic 1,3-benzodioxole system, which is almost coplanar with the CH═CCOOEt group. The molecule is a *Z* isomer, with the (triphenylphosphoranylidene)amino and 1,3-benzodioxole substituents on the same side of the double bond.

Comment

The use of the readily available iminophosphoranes provides a convenient synthetic route to nitrogen-containing heterocycles (Molina & Vilaplana, 1994; Fresneda & Molina, 2004). Thus, the title compound, (I), represents an intermediate in the preparation of imidazolinone (Ding *et al.*, 2003; Yang *et al.*, 2004). The structures of similar compounds with phenyl and *p*-methoxyphenyl groups in place of the benzodioxole substituent have been reported recently (Huang *et al.*, 2005; Ding *et al.*, 2005).



(I)

The molecule of (I) (Fig. 1) is a *Z* isomer, with the (triphenylphosphoranylidene)amino and benzodioxole substituents on the same side of the C8═C9 double bond; the C6—C8═C9—N1 torsion angle is 1.3 (5)°. The molecule of (I) contains an essentially planar bicyclic benzodioxole system, which is almost coplanar with the ethyl propenecarboxylate group (atoms C1, O1, O2, C2–C10, O4, O3 and C11 are coplanar to within 0.08 Å). The planes of the phenyl rings C13–C18 (*A*), C19–C24 (*B*), and C25–C30 (*C*) belonging to the triphenylphosphine group form dihedral angles of 83.9 (1) (*A*/*B*), 73.8 (2) (*A*/*C*) and 52.5 (1)° (*B*/*C*).

Experimental

A solution of (*Z*)-ethyl 2-azido-3-(benzo[*d*][1,3]dioxol-5-yl)prop-2-enoate (10.44 g, 0.04 mol) in dichloromethane (100 ml) was added

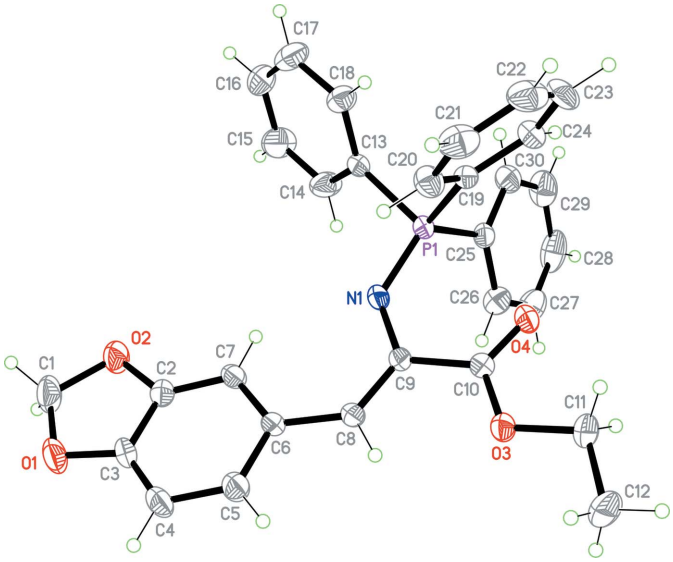


Figure 1
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

dropwise at room temperature under a nitrogen atmosphere to a solution of triphenylphosphine (10.48 g, 0.04 mol) in the same solvent (50 ml). The reaction mixture was stirred for 4 h and then the solvent was removed under reduced pressure. The residue was recrystallized from dichloromethane–petroleum ether (1:2, *v*/*v*) to give the title compound (13.08 g, 66%) (Molina *et al.*, 1993). Single crystals of (I) suitable for X-ray data collection (m.p. 434–435 K) were obtained by slow evaporation of an ethanol solution. Spectroscopic analysis: IR (KBr, ν, cm^{-1}): 2986, 1680, 1590, 1411, 1232; ^{1}H NMR (chloroform-*d*, δ, p.p.m.): 8.09–7.41 (*m*, 17H), 6.75–6.70 (*m*, 2H), 5.91 (*s*, 2H), 3.85 (*q*, 2H, J = 7.1 Hz), 0.99 (*t*, 3H, J = 7.1 Hz).

Crystal data

$C_{30}H_{26}NO_4P$
M_r = 495.49
Triclinic, $P\overline{1}$
a = 8.9346 (10) Å
b = 10.1613 (12) Å
c = 14.8002 (16) Å
α = 71.628 (2)°
β = 89.328 (2)°
γ = 79.940 (2)°
V = 1254.2 (2) Å^3
Z = 2
D_x = 1.312 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1645 reflections
θ = 2.3–24.2°
μ = 0.15 mm^{-1}
T = 298 (2) K
Block, colourless
0.19 × 0.14 × 0.12 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.970, T_{max} = 0.982
6745 measured reflections
4459 independent reflections
3371 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 25.2°
$h = -10 \rightarrow 10$
$k = -12 \rightarrow 9$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.072
$wR(F^2)$ = 0.159
S = 1.09
4459 reflections
326 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0518P)^2 + 0.6569P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

P1—N1	1.573 (3)	O2—C1	1.426 (4)
P1—C13	1.820 (3)	O2—C2	1.376 (4)
P1—C19	1.811 (3)	O3—C10	1.335 (4)
P1—C25	1.816 (3)	O3—C11	1.451 (4)
O1—C1	1.407 (5)	O4—C10	1.209 (3)
O1—C3	1.374 (4)	N1—C9	1.389 (4)
N1—P1—C19	115.60 (15)	C2—O2—C1	105.8 (3)
N1—P1—C25	116.52 (15)	C10—O3—C11	115.0 (3)
C19—P1—C25	111.67 (15)	C9—N1—P1	127.9 (2)
N1—P1—C13	104.93 (14)	O1—C1—O2	108.8 (3)
C19—P1—C13	104.30 (15)	C8—C9—N1	124.0 (3)
C25—P1—C13	101.70 (15)	C8—C9—C10	118.8 (3)
C3—O1—C1	105.8 (3)	N1—C9—C10	117.2 (3)

The H atoms were positioned geometrically and allowed to ride on their parent atoms at C—H distances of 0.93, 0.97 and 0.96 Å for aromatic, methylene and methyl H atoms, respectively, and with U_{iso}(H) = 1.2U_{eq}(C), or 1.5U_{eq}(C) for methyl H atoms.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); 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, 2002); software used to prepare material for publication: *SHELXL97*.

The authors thank the Commission of Science and Technology of Zhejiang Province (grant No. 2003 C24004) and the School of Chemistry and Materials Science, Wenzhou University, for supporting this work.

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