Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

(Z)-Ethyl 3-(4-methoxyphenyl)-2-[(triphenylphosphoranylidene)amino]prop-2-enoate

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Received 14 March 2005
Accepted 1 April 2005
Online 9 April 2005

The title compound, $C_{30}H_{28}NO_3P$, containing four planar ring systems, exists in the Z form. Short intramolecular C$\cdots$O [2.697 (3) Å] and C$\cdots$N [3.049 (4) Å] contacts may indicate the presence of weak intramolecular hydrogen bonds.

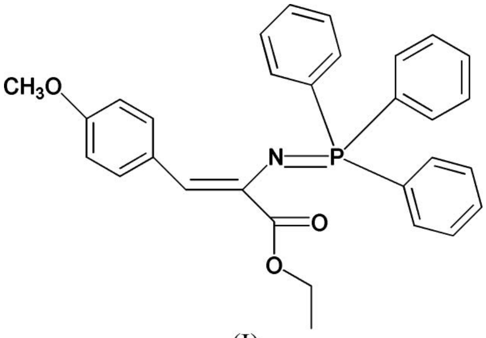
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.066
wR factor = 0.153
Data-to-parameter ratio = 14.8

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

Comment

The readily available iminophosphoranes have become useful building blocks in strategies directed towards the synthesis of nitrogen-containing heterocycles (Fresneda & Molina, 2004). For example, the title compound, (I), is an intermediate in the preparation of imidazolinone, which exhibits fungicidal and herbicidal activities (Yang *et al.*, 2004). More than 1200 crystal structures involving iminophosphorane groups have been published, including a recent report from our laboratory (Huang *et al.*, 2005).



(I)

Compound (I) contains four planar benzene rings, three of which, C13—C18 (A), C7—C12 (B) and C1—C6 (C), belong to the triphenylphosphine group. The dihedral angles A/B, A/C and B/C are 55.7 (1), 84.7 (1) and 78.6 (1)°, respectively.

Bond lengths and angles in the title compound (Table 1) are unexceptional and compare well with those in (Z)-ethyl 3-methoxyphenyl-2-[(triphenylphosphoranylidene)amino]prop-2-enoate (Huang, *et al.*, 2005). The short intramolecular contacts C$\cdots$O and C$\cdots$N (Table 2) may indicate the presence of weak intramolecular C—H$\cdots$O and C—H$\cdots$N hydrogen bonds.

Experimental

The title compound was synthesized in 76% yield by the Staudinger reaction of ethyl β-azidoacetate with triphenylphosphine at room temperature (Molina *et al.*, 1993). Single crystals suitable for X-ray data collection were obtained by slow evaporation of an ethanol solution (m.p. 405–407 K). IR (KBr): 2976, 1686, 1597, 1418, 1232 cm^{-1}; ^{1}H NMR (chloroform-d, p.p.m.): 8.19–6.85 (m, 19H), 6.79 (d, 1H, J = 7.0 Hz), 3.91 (q, 2H, J = 7.0 Hz), 3.85 (s, 3H), 1.03 (t, 3H, J =

doi:10.1107/S1600536805010275

7.0 Hz); ^{13}C NMR (chloroform-*d*, p.p.m.): 168.83, 158.40, 135.30, 135.21, 134.50, 133.14, 133.01, 131.86, 131.61, 131.57, 131.41, 128.91, 128.75, 117.37, 117.11, 113.88, 61.34, 55.92, 14.80.

Crystal data

$C_{30}H_{28}NO_3P$
$M_r = 481.50$
Monoclinic, $P2_1/c$
$a = 10.1565$ (11) Å
$b = 18.879$ (2) Å
$c = 14.2904$ (15) Å
$\beta = 107.548$ (2)°
$V = 2612.6$ (5) Å^3
$Z = 4$

$D_x = 1.224$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2681 reflections
$\theta = 2.4$–24.1°
$\mu = 0.14$ mm^{-1}
$T = 298$ (2) K
Block, colorless
0.33 × 0.27 × 0.16 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{\min} = 0.956$, $T_{\max} = 0.979$
13 808 measured reflections

4701 independent reflections
3736 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.034$
$\theta_{\max} = 25.2°$
$h = -12 \rightarrow 12$
$k = -22 \rightarrow 20$
$l = -17 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.066$
$wR(F^2) = 0.153$
$S = 1.12$
4701 reflections
318 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0599P)^2 + 1.1204P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.37$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

P1—N1	1.580 (2)	O2—C21	1.342 (3)
P1—C6	1.813 (3)	O2—C20	1.451 (4)
P1—C12	1.820 (3)	O3—C27	1.375 (3)
P1—C18	1.822 (3)	O3—C30	1.409 (4)
O1—C21	1.208 (3)	N1—C22	1.369 (3)
N1—P1—C6	106.08 (13)	C12—P1—C18	110.35 (12)
N1—P1—C12	116.58 (12)	C21—O2—C20	116.3 (2)
C6—P1—C12	103.49 (13)	C27—O3—C30	118.3 (2)
N1—P1—C18	115.95 (12)	C22—N1—P1	130.49 (19)
C6—P1—C18	102.43 (12)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C25—H25···N1	0.93	2.43	3.049 (4)	124
C23—H23···O2	0.93	2.25	2.697 (3)	109

The H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of Csp^2—H = 0.93 Å with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(parent atom), C(methylene)—H = 0.97 Å with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(parent atom), and C(methyl)—H = 0.96 Å with $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(parent atom).

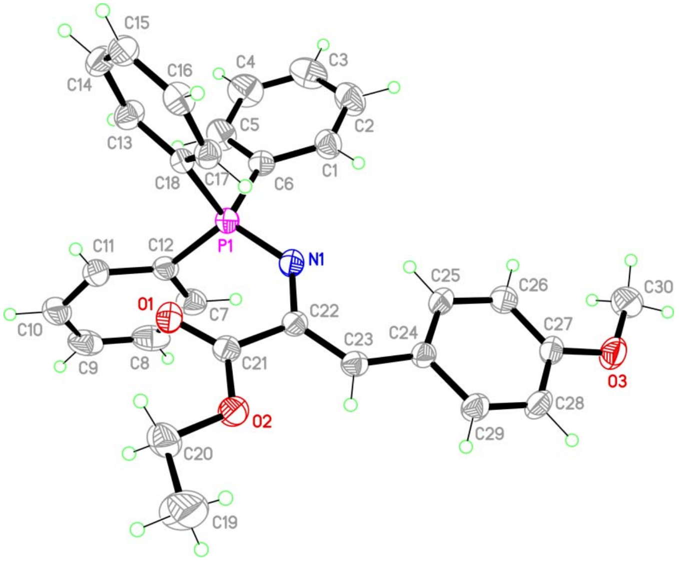


Figure 1
The molecular structure of (I), with the atom numbering scheme, showing displacement ellipsoids at the 50% probability level.

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 acknowledge financial support by the Wenzhou Bureau of Science and Technology of China (grant No. G 2004053).

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