Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

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

Xiao-Bo Huang,* Jin-Chang Ding, Miao-Chang Liu, Xi-Chang Zhang and Mao-Lin Hu

School of Chemistry and Materials Science, Wenzhou Normal College, Zhejiang, Wenzhou 325027, People's Republic of China

Correspondence e-mail:
xiaobhuang@hotmail.com

Received 29 March 2005
Accepted 4 April 2005
Online 9 April 2005

Key indicators

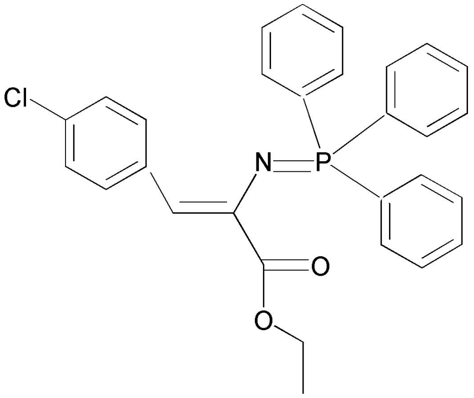
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.006 Å
R factor = 0.078
wR factor = 0.153
Data-to-parameter ratio = 14.7

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

The title compound, $C_{29}H_{25}ClNO_2P$, exists in the Z form. The short C···O [2.700 (4) Å] and C···N [2.963 (4) Å and 2.988 (5) Å] intramolecular contacts may indicate the presence of weak intramolecular hydrogen bonds.

Comment

The readily available iminophosphoranes have become useful building blocks in organic synthetic strategies directed towards the synthesis of nitrogen-containing heterocycles (Fresneda & Molina, 2004). The title compound, (I), is an intermediate in the preparation of imidazolinones (Ding *et al.* 2003), some of which exhibit fungicidal and herbicidal activities (Yang *et al.*, 2004).



(I)

The molecule of (I) contains four essentially planar phenyl rings, three of which, C12–C17 (A), C18–C23 (B) and C24–C29 (C), belong to the triphenylphosphine group (Fig. 1). The dihedral angles A/B, A/C and B/C are 56.2 (1), 78.8 (2) and 87.4 (1)°, respectively. Bond lengths and angles in the title compound (Table 1) are similar to those found in (Z)-ethyl 3-phenyl-2-[(triphenylphosphoranylidene)amino]prop-2-enoate (Huang *et al.*, 2005).

The short C···O and C···N intramolecular contacts (Table 2) may indicate the presence of weak intramolecular C—H···O and C—H···N hydrogen bonds.

Experimental

The title compound was synthesized in 70% yield by the Staudinger reaction of ethyl β-azidoacetate with triphenylphosphine at room temperature (Molina *et al.*, 1993). Single crystals of (I) suitable for X-ray data collection were obtained by slow evaporation of a solution in ethanol and trichloromethane (5:1, v/v; m.p. 453–455 K). IR (KBr, cm^{-1}): ν 2950, 1671, 1592, 1410, 1231; ^{1}H NMR (chloroform-d, p.p.m):

δ 8.12–7.20 (*m*, 19H), 6.67 (*d*, 1H, $J = 7.1$ Hz), 3.86 (*q*, 2H, $J = 7.1$ Hz), 1.00 (*t*, 3H, $J = 7.1$ Hz); ^{31}P NMR (chloroform-*d*, p.p.m): δ 8.60 (*s*).

Crystal data

$C_{29}H_{25}ClNO_2P$	$D_x = 1.279$ Mg m^{-3}
$M_r = 485.92$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 1467 reflections
$a = 10.4682$ (8) Å	$\theta = 2.2$–24.1°
$b = 17.7551$ (13) Å	$\mu = 0.24$ mm^{-1}
$c = 14.1264$ (10) Å	$T = 298$ (2) K
$\beta = 106.058$ (1)°	Block, colorless
$V = 2523.1$ (3) Å^3	0.23 × 0.19 × 0.08 mm
$Z = 4$	

Data collection

Bruker APEX area-detector diffractometer	4535 independent reflections
φ and ω scans	3370 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)	$R_{int} = 0.057$
$T_{min} = 0.947$, $T_{max} = 0.981$	$\theta_{max} = 25.2°$
13 251 measured reflections	$h = -12 \rightarrow 12$
	$k = -17 \rightarrow 21$
	$l = -14 \rightarrow 16$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0467P)^2 + 1.2582P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.078$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.153$	$\Delta\rho_{max} = 0.38$ e Å^{-3}
$S = 1.14$	$\Delta\rho_{min} = -0.33$ e Å^{-3}
4535 reflections	Extinction correction: none
308 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Cl1—C1	1.745 (4)	O1—C10	1.452 (4)
P1—N1	1.576 (3)	O2—C9	1.198 (4)
P1—C29	1.811 (3)	N1—C8	1.369 (4)
P1—C17	1.811 (3)	C7—C8	1.353 (4)
P1—C23	1.812 (3)	C8—C9	1.505 (5)
O1—C9	1.343 (4)		
N1—P1—C29	104.92 (15)	C9—O1—C10	115.9 (3)
N1—P1—C17	115.30 (15)	C8—N1—P1	130.6 (2)
C29—P1—C17	106.53 (15)	C7—C8—N1	123.6 (3)
N1—P1—C23	117.03 (16)	N1—C8—C9	117.6 (3)
C29—P1—C23	101.32 (15)	O2—C9—O1	123.1 (3)
C17—P1—C23	110.04 (15)	O1—C9—C8	113.9 (3)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C28—H28···N1	0.93	2.59	2.988 (5)	106
C7—H7···O1	0.93	2.27	2.700 (4)	107
C3—H3···N1	0.93	2.35	2.963 (4)	123

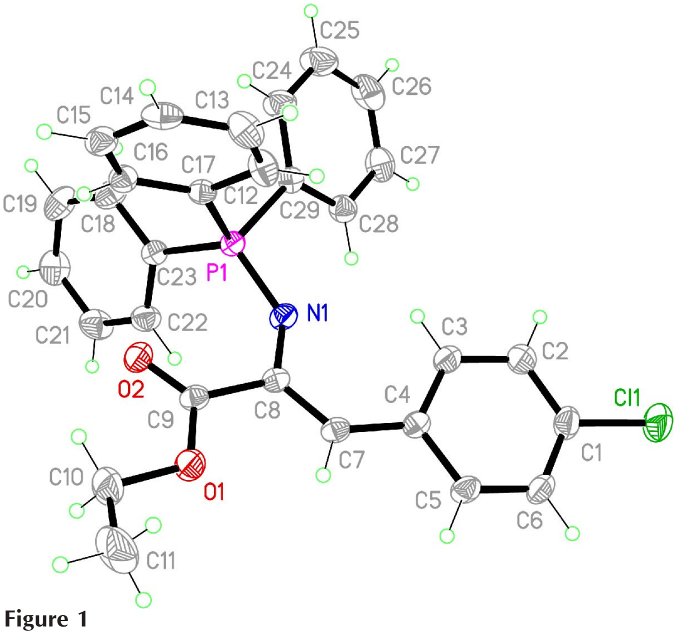


Figure 1
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of C—H = 0.93 Å (CH) and 0.97 Å (CH_2), with $U_{iso}(H) = 1.2U_{eq}(C)$, or Csp^3—H = 0.96 Å, with $U_{iso}(H) = 1.5U_{eq}(C)$.

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 Bureau of Science and Technology of Wenzhou (grant No. G 2004053).

References

Bruker (2002). *SADABS* (Version 2.03), *SAINT* (Version 6.02), *SMART* (Version 5.62) and *SHELXTL*. Bruker AXS Inc., Madison, Winsonsin, USA.

Ding, M. W., Sun, Y. & Liu, Z. J. (2003). *Synth. Commun.* **33**, 1267–1274.

Fresneda, P. M. & Molina, P. (2004). *Synlett*, **1**, 1–17.

Huang, X. B., Liu, M. C., Wu, H. Y., Ding, J. C. & Hu, M. L. (2005). *Acta Cryst.* E**61**, o280–o281.

Molina, P., Pastor, A. & Vilaplana, M. J. (1993). *Tetrahedron*, **49**, 7769–7778.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Yang, F. L., Liu, Z. J., Huang, X. B. & Ding, M. W. (2004). *J. Heterocycl. Chem.* **41**, 77–83.

o1262 Huang *et al.* • $C_{29}H_{25}ClNO_2P$ *Acta Cryst.* (2005). E**61**, o1261–o1262