Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

Xiao-Bo Huang,* Rong Xu, Jin-Chang Ding, Hua-Yue Wu, Xi-Chang Zhang, Miao-Chang Liu and Hong-Ping Xiao

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

Correspondence e-mail:
xiaobhuang@hotmail.com

Received 31 May 2005
Accepted 13 June 2005
Online 24 June 2005

Key indicators

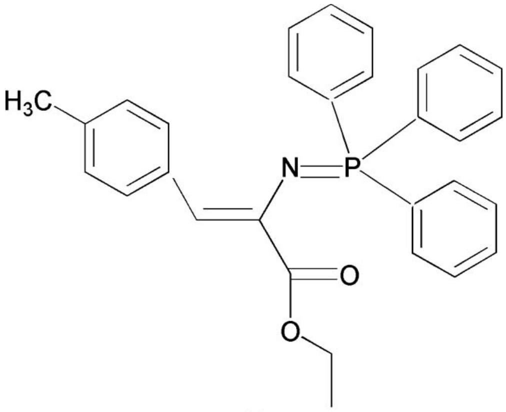
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.067
wR factor = 0.149
Data-to-parameter ratio = 14.8

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

The title compound, $C_{30}H_{28}NO_2P$, exists in the *Z* form.

Comment

Recently, 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 exhibiting various biological properties, for example fungicidal and herbicidal activities (Yang *et al.*, 2004). Some crystal structures involving iminophosphorane groups have been published, including recent reports from our laboratory (Ding *et al.*, 2005; Huang *et al.*, 2005).



(I)

Compound (I) (Fig. 1) exists in the *Z* configuration and contains four benzene rings, three of which, C1–C6 (ring *A*), C7–C12 (ring *B*) and C13–C18 (ring C), belong to the triphenylphosphine group. The dihedral angles between the planes of rings *A*/*B*, *A*/*C* and *B*/*C* are 76.4 (1), 86.8 (1) and 56.7 (1)°, respectively. Bond lengths and angles in (I) (Table 1) are unexceptional and compare well with those mentioned above from our laboratory.

Experimental

The title compound was readily synthesized in 60% yield by the Staudinger reaction of ethyl β-azidoacetate (0.01 mol) with triphenylphosphine (0.01 mol) at room temperature (Molina *et al.*, 1993). Single crystals of (I) suitable for X-ray data collection were obtained by slow evaporation of an ethanol solution (m.p. 403–405 K). Spectroscopic analysis: IR (KBr, ν, cm^{-1}): 2940, 1670, 1588, 1415, 1231; ^1H NMR (chloroform-*d*, δ, p.p.m.): 8.03–7.08 (*m*, 19H), 6.72 (*d*, 1H, J_{PH} = 6.8 Hz), 3.84 (*q*, 2H, J = 7.1 Hz), 2.32 (*s*, 3H), 0.99 (*t*,

doi:10.1107/S1600536805018647

3H, J = 7.1 Hz); ^{13}C NMR (chloroform-d, δ, p.p.m.): 168.04, 135.37, 133.81, 133.56, 133.27, 132.46, 132.33, 132.11, 131.98, 131.89, 130.87, 129.34, 128.65, 128.53,128.43, 128.37, 128.16, 127.99, 116.81, 77.45, 76.60, 60.58, 21.29, 14.02.

Crystal data

$C_{30}H_{28}NO_2P$
M_r = 465.50
Monoclinic, $P2_1/n$
a = 10.6177 (8) Å
b = 17.6052 (13) Å
c = 14.1076 (10) Å
β = 105.897 (2)°
V = 2536.2 (3) Å^3
Z = 4

D_x = 1.219 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2173 reflections
θ = 2.4–24.1°
μ = 0.14 mm^{-1}
T = 298 (2) K
Block, colourless
0.38 × 0.17 × 0.16 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.950, T_{max} = 0.979
13404 measured reflections

4560 independent reflections
3518 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 25.2°
$h = -12 \rightarrow 10$
$k = -20 \rightarrow 21$
$l = -14 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.150
S = 1.10
4560 reflections
309 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0583P)^2 + 1.0666P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

P1—N1	1.571 (2)	O1—C20	1.199 (3)
P1—C7	1.808 (3)	O2—C20	1.339 (3)
P1—C13	1.813 (3)	O2—C21	1.444 (4)
P1—C1	1.819 (3)	N1—C19	1.378 (3)
N1—P1—C7	115.43 (12)	C13—P1—C1	101.69 (12)
N1—P1—C13	116.77 (13)	C20—O2—C21	116.3 (2)
C7—P1—C13	109.93 (12)	C19—N1—P1	130.39 (19)
N1—P1—C1	105.35 (12)	O1—C20—O2	122.8 (3)
C7—P1—C1	106.00 (13)		

H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of Csp^2—H = 0.93 Å with U_{iso}(H) = 1.2U_{eq}(C), Csp^3—H = 0.96 Å with U_{iso}(H) = 1.5U_{eq}(C) for methyl groups, and Csp^3—H = 0.97 Å with U_{iso}(H) = 1.2U_{eq}(C) for the methylene group.

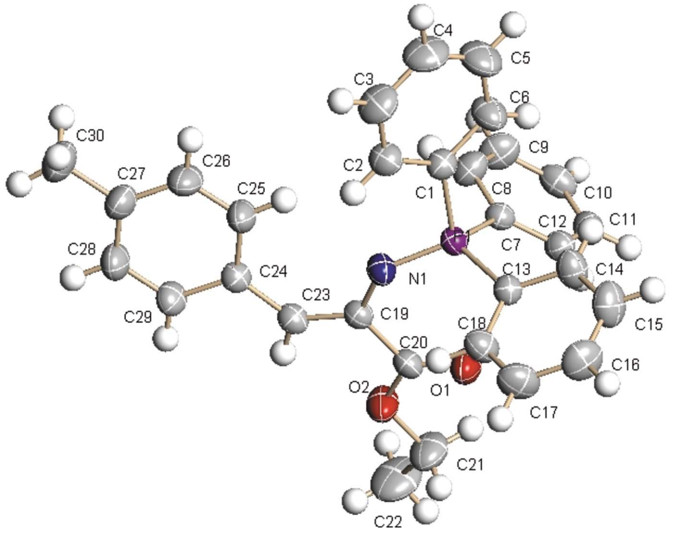


Figure 1
The molecular structure of (I). with the atom-numbering scheme, showing displacement ellipsoids at the 50% probability level.

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

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