Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Ethyl 5-(triphenylphosphoranylideneamino)-1*H*-1,2,3-triazole-4-carboxylate

Ming-Guo Liu* and Quan-Bin Liao

College of Chemistry and Life Science, China Three Gorges University, Yichang 443002, People's Republic of China

Correspondence e-mail:
mgliu0427@yahoo.com.cn

Received 19 April 2006
Accepted 2 May 2006

Key indicators

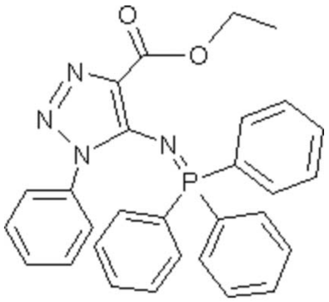
Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.056
wR factor = 0.189
Data-to-parameter ratio = 17.0

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

Molecules of the title compound, $C_{29}H_{25}N_4O_2P$, form a supramolecular structure *via* intermolecular C—H$\cdots\pi$ interactions.

Comment

We have recently focused on the synthesis of fused heterocyclic systems containing the pyrimidone group using aza-Wittig reactions (Liu *et al.*, 2006). Iminophosphoranes are key intermediates, which can be converted into a wide range of pyrimidiones by reaction with various isocyanates and subsequent reaction with a secondary amine at room temperature. In this paper, we report the structure of the title compound, (I) (Fig. 1).



(I)

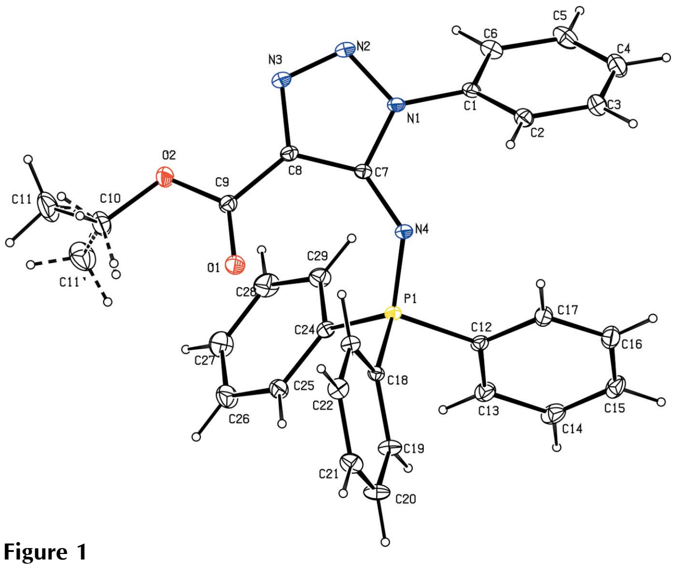


Figure 1
A view of the molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. Both disorder components are shown.

doi:10.1107/S1600536806016096

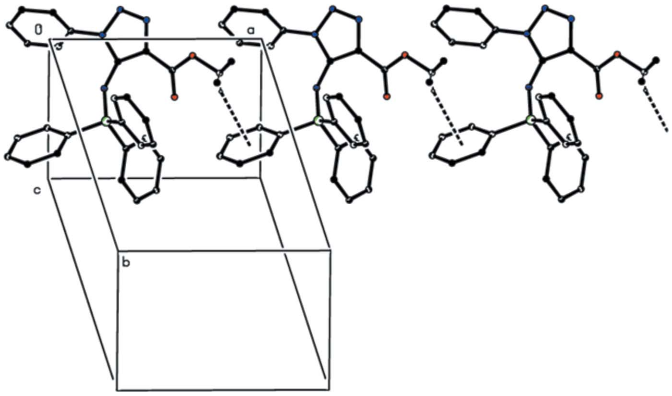


Figure 2
A partial packing diagram of (I), showing the C–H$\cdots\pi$ interactions as dashed lines. H atoms, except those involved in C—H$\cdots\pi$ interactions, have been omitted for clarity. One disorder component has been omitted for clarity.

The five-membered triazole ring is planar, with a maximum deviation of 0.003 (2) Å for atom N3. The phenyl ring C1–C6 is twisted with respect to the triazole ring, with a dihedral angle of 50.66 (12)°. A packing diagram is shown in Fig. 2. There exists a weak intermolecular C—H$\cdots\pi$ interaction between the ethyl group and phenyl ring C12–C17 (Table 1).

Experimental

To a solution of 5-amino-1-benzyl-4-ethoxycarbonyl-1,2,3-triazole (5 mmol) in anhydrous acetonitrile (15 ml) were added triphenylphosphine (15 mmol), hexachloroethane (15 mmol) and triethylamine (30 mmol). After the reaction mixture had been allowed to stand for 5 h at room temperature, the solution was concentrated under reduced pressure and the residue recrystallized from ethanol to afford the title compound, (I). The product was recrystallized from ethanol/dichloromethane (3:1) at room temperature to give single crystals suitable for X-ray diffraction analysis.

Crystal data

$C_{29}H_{25}N_4O_2P$
$M_r = 492.50$
Triclinic, $P\overline{1}$
$a = 9.2719$ (9) Å
$b = 10.1752$ (9) Å
$c = 14.3413$ (13) Å
$\alpha = 83.580$ (1)°
$\beta = 89.418$ (2)°
$\gamma = 73.251$ (2)°
$V = 1287.1$ (2) Å^3
$Z = 2$
$D_x = 1.271$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.14$ mm^{-1}
$T = 292$ (2) K
Plate, green
0.36 × 0.30 × 0.10 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{\min} = 0.951$, $T_{\max} = 0.986$
11400 measured reflections
5723 independent reflections
4468 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.090$
$\theta_{\max} = 27.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.189$
$S = 1.05$
5723 reflections
337 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1112P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.66$ e Å^{-3}
$\Delta\rho_{\min} = -0.45$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*1 is the centroid of the C12–C17 ring.

D—H$\cdots$*A*	*D*—H	H$\cdots$*A*	*D*$\cdots$*A*	*D*—H$\cdots$*A*
C10—H10*D*$\cdots$*Cg*1$^{\rm i}$	0.97	2.86	3.758 (2)	155

Symmetry code: (i) $x + 1, y, z$.

H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and constrained to ride on their parent atoms, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ or $1.5U_{\rm eq}$(methyl C). The ethyl group C10—C11 is disordered over two sites. Refined occupancy factors converged to almost equal values of 0.520 (15) and 0.480 (15), and were then fixed at 0.5 in the final refinement.

Data collection: *SMART* (Bruker, 2001); 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: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2001).

References

Bruker (2001). *SMART* (Version 5.628) and *SAINT* (Version 6.45). Bruker AXS Inc., Madison, Wisconsin, USA.
Liu, M.-G., Yuan, J.-Z., Hu, Y.-G. & Xu, S.-Z. (2006). *Acta Cryst.* E**62**, o147–o149.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Sheldrick, G. M. (2001). *SHELXTL*. Version 5.0. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2003). *SADABS*. Version 2.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.