Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-Amino-5-(2-ethoxyphenyl)-2,4-dihydro-2*H*-1,2,4-triazole-3-thione–triphenylphosphine oxide (1/1)

Xi-An Chen, Xiao-Bo Huang and Hua-Yue Wu*

School of Chemistry and Materials Engineering, Wenzhou University, Wenzhou 325027, People's Republic of China
Correspondence e-mail: hywu3@163.com

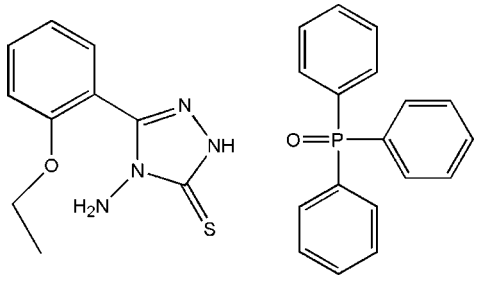
Received 27 April 2007; accepted 21 May 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.053; wR factor = 0.135; data-to-parameter ratio = 14.5.

In the title compound, $C_{18}H_{15}OP{\cdot}C_{10}H_{12}N_4OS$, the triazole ring makes a dihedral angle of 51.24 (7)° with the attached benzene ring. The triazole ring and its thione substituent are delocalized. There are intermolecular N—H···O and N—H···S hydrogen bonds, which also involve the cocrystallized triphenylphosphine oxide molecule.

Related literature

For related literature, see: Ding *et al.* (2004); Fresneda & Molina (2004); Huang *et al.* (2005); Jin *et al.* (2004); Xiong *et al.* (2005); Ding *et al.* (2005).



Experimental

Crystal data

$C_{18}H_{15}OP{\cdot}C_{10}H_{12}N_4OS$
M_r = 514.57
Monoclinic, $P2_1/c$
a = 10.0514 (7) Å
b = 18.3008 (12) Å
c = 15.285 (1) Å
β = 105.726 (1)°
V = 2706.4 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 298 (2) K
0.42 × 0.31 × 0.27 mm

Data collection

Bruker APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.917, T_{max} = 0.942
14188 measured reflections
4857 independent reflections
4255 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.135
S = 1.08
4857 reflections
335 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1	0.862 (18)	1.830 (19)	2.682 (2)	169 (3)
N4—H4A···S1	0.846 (18)	2.68 (3)	3.169 (2)	118 (3)
N4—H4B···O2	0.847 (18)	2.35 (3)	2.904 (3)	124 (3)
N4—H4B···S1^{i}	0.847 (18)	2.99 (3)	3.533 (2)	124 (3)

Symmetry code: (i) $-x, -y+2, -z$.

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. 2005 C31036).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CF2105).

References

Bruker (2002). *SADABS* (Version 2.03), *SAINT* (Version 6.02), *SMART* (Version 5.62) and *SHELXTL* (Version 6.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Ding, J.-C., Huang, X.-B., Wu, H.-Y., Liu, M.-C. & Hu, M.-L. (2005). *Acta Cryst.* E**61**, o1259–o1260.
Ding, M. W., Fu, B. Q. & Cheng, L. (2004). *Synthesis*, pp. 1067–1071.
Fresneda, P. M. & Molina, P. (2004). *Synlett*, pp. 1–17.
Huang, X.-B., Liu, M.-C., Wu, H.-Y., Ding, J.-C. & Hu, M.-L. (2005). *Acta Cryst.* E**61**, o280–o281.
Jin, Z.-M., Li, L., Li, M.-C., Hu, M.-L. & Shen, L. (2004). *Acta Cryst.* C**60**, o642–o643.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Xiong, J., Liu, M.-C. & Yuan, J.-X. (2005). *Acta Cryst.* E**61**, o2665–o2667.

supplementary materials

Acta Cryst. (2007). E**63**, o3191 [**doi:10.1107/S1600536807024774**]

4-Amino-5-(2-ethoxyphenyl)-2,4-dihydro-2*H*-1,2,4-triazole-3-thione-triphenylphosphine oxide (1/1)

X.-A. Chen, X.-B. Huang and H.-Y. Wu

Comment

Recently, iminophosphoranes have attracted increasing attention as useful building blocks for nitrogen-containing heterocycles (Fresneda & Molina, 2004). Many interesting crystal structures involving iminophosphorane groups have been published, including some recent reports from our laboratory (Ding *et al.*, 2005; Huang *et al.*, 2005). As an extension of our research, we report the synthesis and crystal structure of the title compound (I), in which an iminophosphorane is not formed.

The molecular structure of (I) is shown in Fig. 1. The triazole ring (C20, C19, N1—N3) is planar with an r.m.s. deviation of 0.0066 Å, and makes a dihedral angle of 51.24 (7)° with the attached benzene group. The C—N bond lengths, in the range 1.305 (3) Å-1.370 (3) Å, are longer than a typical C=N bond [*ca* 1.269 (2) Å], but short than a typical C—N bond length [*ca* 1.443 (4) Å], indicating electron delocalization in the triazole ring (Jin *et al.*, 2004).

The crystal packing (Fig. 2) is stabilized by an intramolecular N—H···O hydrogen bond, an intermolecular N—H···O hydrogen bond between the triazole ring and the triphenylphosphine oxide molecule, and intermolecular N—H···S hydrogen bonds, which can be described by the graph-set notation $R_2^2(8)$ (Xiong *et al.*, 2005).

Experimental

Triethylamine (0.024 mol, 2.42 g) was added to a mixture of triphenylphosphine (0.012 mol, 2.84 g), 4-amino-5-(2-ethoxyphenyl)-4*H*-1,2,4-triazole-3-thiol (0.008 mol) and dry acetonitrile (40 ml) at room temperature. The mixture was stirred for 5 h under reflux (Ding *et al.*, 2004). The white solid was filtered off and recrystallized from ethanol. Yield 58%, m. p. 436–438 K.

Refinement

H atoms attached to N atoms were located in a difference Fourier map, and refined with N—H distances restrained to 0.85 (1) Å with U_{iso} = 1.2U_{eq}(N). The other H atoms were positioned geometrically and allowed to ride on their parent atoms with C—H = 0.93–0.97 Å and U_{iso} = 1.2U_{eq}(C) or 1.5U_{eq}(methyl-C).

Figures

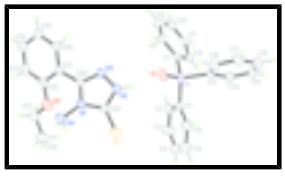


Fig. 1. The molecular structure of (I) with the atom numbering, showing displacement ellipsoids at the 30% probability level.

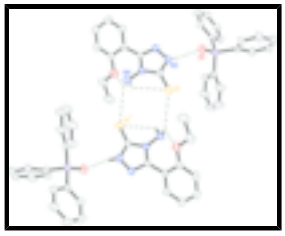


Fig. 2. Part of the crystal packing of (I). Hydrogen bonds are shown as dashed lines. [Symmetry code: (i) −*x*, 2 − *y*, −*z*.]

4-Amino-5-(2-ethoxyphenyl)-2,4-dihydro-2*H*-1,2,4-triazole-3-thione– triphenylphosphine oxide (1/1)

Crystal data

$C_{18}H_{15}OP·C_{10}H_{12}N_4OS$	F_{000} = 1080
M_r = 514.57	D_x = 1.263 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 5026 reflections
a = 10.0514 (7) Å	θ = 2.4–25.1º
b = 18.3008 (12) Å	μ = 0.21 mm^{-1}
c = 15.285 (1) Å	T = 298 (2) K
β = 105.726 (1)º	Block, colourless
V = 2706.4 (3) Å^3	0.42 × 0.31 × 0.27 mm
Z = 4	

Data collection

Bruker APEX area-detector diffractometer	4857 independent reflections
Radiation source: fine-focus sealed tube	4255 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.022
T = 298(2) K	θ_{max} = 25.2º
φ and ω scans	θ_{min} = 1.8º
Absorption correction: multi-scan (SADABS; Bruker, 2002)	h = −11→12
T_{min} = 0.917, T_{max} = 0.942	k = −21→21
14188 measured reflections	l = −16→18

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.053	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.135	$w = 1/[\sigma^2(F_o^2) + (0.066P)^2 + 0.984P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.08	$(\Delta/\sigma)_{max}$ = 0.001
4857 reflections	$\Delta\rho_{max}$ = 0.27 e Å^{-3}

335 parameters
3 restraints
Primary atom site location: structure-invariant direct methods

$\Delta\rho_{min} = -0.27$ e Å^{-3}
Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	−0.05775 (9)	1.11318 (3)	0.05669 (4)	0.0669 (2)
P1	−0.47530 (6)	1.19485 (3)	0.18357 (4)	0.04240 (17)
O1	−0.32154 (16)	1.19058 (8)	0.21438 (12)	0.0549 (4)
O2	−0.00016 (19)	0.84581 (8)	0.21677 (11)	0.0632 (5)
N1	0.00551 (18)	1.00791 (9)	0.18675 (11)	0.0419 (4)
N2	−0.1418 (2)	1.08364 (10)	0.20819 (12)	0.0466 (4)
N3	−0.11670 (18)	1.03653 (10)	0.28068 (12)	0.0455 (4)
N4	0.1115 (2)	0.97627 (12)	0.15487 (15)	0.0583 (5)
C1	−0.5478 (2)	1.15002 (13)	0.07667 (15)	0.0503 (5)
C2	−0.6097 (3)	1.08207 (15)	0.06990 (18)	0.0673 (7)
H2	−0.6204	1.0591	0.1218	0.081*
C3	−0.6557 (3)	1.04802 (19)	−0.0136 (2)	0.0856 (9)
H3	−0.6986	1.0026	−0.0176	0.103*
C4	−0.6389 (4)	1.0803 (2)	−0.0896 (2)	0.0906 (10)
H4	−0.6699	1.0570	−0.1456	0.109*
C5	−0.5763 (4)	1.1470 (2)	−0.0841 (2)	0.0996 (12)
H5	−0.5644	1.1689	−0.1363	0.119*
C6	−0.5307 (4)	1.18205 (17)	−0.00140 (19)	0.0796 (9)
H6	−0.4882	1.2275	0.0018	0.096*
C7	−0.5512 (2)	1.15562 (11)	0.26723 (14)	0.0446 (5)
C8	−0.4618 (3)	1.13856 (15)	0.35082 (16)	0.0616 (6)
H8	−0.3673	1.1458	0.3607	0.074*
C9	−0.5120 (4)	1.11093 (17)	0.41956 (19)	0.0786 (9)
H9	−0.4513	1.0997	0.4756	0.094*
C10	−0.6507 (4)	1.10008 (15)	0.40541 (19)	0.0736 (8)
H10	−0.6842	1.0811	0.4518	0.088*
C11	−0.7405 (3)	1.11694 (14)	0.32346 (19)	0.0636 (7)
H11	−0.8348	1.1098	0.3145	0.076*
C12	−0.6922 (2)	1.14453 (13)	0.25385 (16)	0.0540 (6)

H12	−0.7537	1.1557	0.1981	0.065*
C13	−0.5308 (2)	1.28893 (12)	0.17058 (15)	0.0446 (5)
C14	−0.6686 (3)	1.30798 (14)	0.13743 (17)	0.0586 (6)
H14	−0.7351	1.2720	0.1171	0.070*
C15	−0.7070 (3)	1.38043 (17)	0.1346 (2)	0.0727 (8)
H15	−0.7997	1.3931	0.1130	0.087*
C16	−0.6100 (4)	1.43355 (16)	0.1632 (2)	0.0786 (9)
H16	−0.6369	1.4823	0.1611	0.094*
C17	−0.4741 (4)	1.41562 (15)	0.1947 (2)	0.0786 (8)
H17	−0.4082	1.4522	0.2133	0.094*
C18	−0.4333 (3)	1.34315 (13)	0.19924 (18)	0.0602 (6)
H18	−0.3404	1.3310	0.2216	0.072*
C19	−0.0680 (2)	1.06811 (11)	0.15007 (14)	0.0442 (5)
C20	−0.0245 (2)	0.99114 (11)	0.26665 (14)	0.0400 (5)
C21	0.0393 (2)	0.93404 (11)	0.33202 (15)	0.0428 (5)
C22	0.0484 (2)	0.86112 (12)	0.30667 (16)	0.0489 (5)
C23	0.1019 (3)	0.80937 (13)	0.3740 (2)	0.0620 (7)
H23	0.1101	0.7609	0.3578	0.074*
C24	0.1424 (3)	0.82924 (16)	0.46318 (19)	0.0673 (7)
H24	0.1777	0.7941	0.5073	0.081*
C25	0.1316 (3)	0.90041 (15)	0.48875 (18)	0.0626 (7)
H25	0.1578	0.9133	0.5499	0.075*
C26	0.0816 (2)	0.95263 (13)	0.42319 (15)	0.0504 (5)
H26	0.0762	1.0010	0.4404	0.060*
C27	−0.0126 (4)	0.77064 (15)	0.1900 (2)	0.0777 (8)
H27A	−0.0690	0.7448	0.2223	0.093*
H27B	0.0780	0.7480	0.2048	0.093*
C28	−0.0768 (4)	0.76641 (18)	0.0916 (2)	0.0938 (11)
H28A	−0.1676	0.7875	0.0777	0.141*
H28B	−0.0834	0.7162	0.0727	0.141*
H28C	−0.0213	0.7928	0.0601	0.141*
H2A	−0.202 (3)	1.1183 (15)	0.203 (2)	0.113*
H4A	0.123 (4)	1.0041 (17)	0.113 (2)	0.113*
H4B	0.078 (4)	0.9357 (14)	0.133 (2)	0.113*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.1072 (6)	0.0476 (4)	0.0524 (4)	0.0064 (3)	0.0329 (4)	0.0079 (3)
P1	0.0379 (3)	0.0395 (3)	0.0503 (3)	0.0045 (2)	0.0128 (2)	−0.0017 (2)
O1	0.0421 (9)	0.0480 (9)	0.0748 (11)	0.0070 (7)	0.0162 (8)	−0.0034 (8)
O2	0.0910 (13)	0.0364 (9)	0.0617 (11)	0.0011 (8)	0.0197 (9)	−0.0026 (7)
N1	0.0504 (10)	0.0344 (9)	0.0447 (10)	0.0030 (7)	0.0196 (8)	−0.0039 (7)
N2	0.0535 (11)	0.0385 (10)	0.0504 (10)	0.0088 (8)	0.0187 (9)	0.0020 (8)
N3	0.0494 (11)	0.0413 (10)	0.0494 (10)	0.0056 (8)	0.0194 (8)	0.0027 (8)
N4	0.0686 (14)	0.0513 (12)	0.0663 (14)	0.0110 (10)	0.0374 (11)	−0.0013 (10)
C1	0.0493 (13)	0.0529 (14)	0.0502 (13)	0.0053 (10)	0.0162 (10)	−0.0052 (10)
C2	0.0782 (19)	0.0658 (17)	0.0614 (16)	−0.0144 (14)	0.0251 (14)	−0.0152 (13)

C3	0.091 (2)	0.088 (2)	0.079 (2)	−0.0216 (18)	0.0246 (17)	−0.0317 (17)
C4	0.102 (3)	0.111 (3)	0.0597 (18)	−0.001 (2)	0.0222 (17)	−0.0295 (18)
C5	0.141 (3)	0.109 (3)	0.0576 (19)	−0.006 (3)	0.043 (2)	−0.0051 (18)
C6	0.112 (3)	0.075 (2)	0.0610 (17)	−0.0117 (17)	0.0393 (17)	−0.0036 (14)
C7	0.0491 (12)	0.0385 (11)	0.0462 (12)	0.0010 (9)	0.0131 (10)	−0.0054 (9)
C8	0.0617 (15)	0.0677 (16)	0.0505 (14)	−0.0018 (13)	0.0068 (12)	0.0051 (12)
C9	0.092 (2)	0.087 (2)	0.0507 (16)	−0.0075 (17)	0.0095 (15)	0.0084 (14)
C10	0.105 (3)	0.0665 (18)	0.0582 (17)	−0.0149 (16)	0.0380 (17)	−0.0036 (13)
C11	0.0674 (17)	0.0603 (16)	0.0705 (17)	−0.0121 (13)	0.0315 (14)	−0.0101 (13)
C12	0.0522 (14)	0.0577 (14)	0.0522 (13)	−0.0037 (11)	0.0143 (11)	−0.0035 (11)
C13	0.0477 (12)	0.0426 (11)	0.0477 (12)	0.0080 (9)	0.0203 (10)	0.0024 (9)
C14	0.0526 (14)	0.0602 (15)	0.0654 (16)	0.0129 (11)	0.0203 (12)	0.0118 (12)
C15	0.0731 (18)	0.074 (2)	0.0811 (19)	0.0350 (16)	0.0383 (15)	0.0259 (15)
C16	0.115 (3)	0.0485 (16)	0.087 (2)	0.0277 (17)	0.052 (2)	0.0133 (14)
C17	0.102 (2)	0.0459 (15)	0.094 (2)	−0.0025 (15)	0.0372 (19)	−0.0028 (14)
C18	0.0594 (15)	0.0482 (14)	0.0754 (17)	0.0023 (12)	0.0225 (13)	−0.0009 (12)
C19	0.0544 (13)	0.0338 (11)	0.0447 (12)	−0.0016 (9)	0.0138 (10)	−0.0036 (9)
C20	0.0432 (11)	0.0350 (11)	0.0437 (11)	−0.0020 (9)	0.0150 (9)	−0.0032 (8)
C21	0.0397 (11)	0.0391 (11)	0.0529 (13)	0.0015 (9)	0.0180 (9)	0.0043 (9)
C22	0.0482 (13)	0.0434 (12)	0.0569 (14)	0.0019 (10)	0.0173 (11)	0.0013 (10)
C23	0.0663 (16)	0.0409 (13)	0.0795 (18)	0.0082 (11)	0.0211 (14)	0.0110 (12)
C24	0.0645 (17)	0.0683 (18)	0.0660 (17)	0.0109 (13)	0.0124 (13)	0.0227 (14)
C25	0.0578 (15)	0.0746 (18)	0.0519 (14)	0.0040 (13)	0.0086 (12)	0.0092 (13)
C26	0.0473 (13)	0.0524 (13)	0.0515 (13)	0.0007 (10)	0.0135 (10)	0.0010 (10)
C27	0.102 (2)	0.0452 (15)	0.085 (2)	−0.0095 (15)	0.0244 (17)	−0.0081 (14)
C28	0.140 (3)	0.070 (2)	0.081 (2)	−0.016 (2)	0.046 (2)	−0.0201 (17)

Geometric parameters (Å, °)

S1—C19	1.676 (2)	C10—C11	1.366 (4)
P1—O1	1.4906 (16)	C10—H10	0.930
P1—C1	1.797 (2)	C11—C12	1.380 (3)
P1—C13	1.804 (2)	C11—H11	0.930
P1—C7	1.806 (2)	C12—H12	0.930
O2—C22	1.357 (3)	C13—C18	1.379 (3)
O2—C27	1.431 (3)	C13—C14	1.384 (3)
N1—C19	1.360 (3)	C14—C15	1.378 (4)
N1—C20	1.370 (3)	C14—H14	0.930
N1—N4	1.410 (3)	C15—C16	1.362 (5)
N2—C19	1.334 (3)	C15—H15	0.930
N2—N3	1.372 (2)	C16—C17	1.360 (5)
N2—H2A	0.862 (18)	C16—H16	0.930
N3—C20	1.305 (3)	C17—C18	1.384 (4)
N4—H4A	0.846 (18)	C17—H17	0.930
N4—H4B	0.847 (18)	C18—H18	0.930
C1—C6	1.381 (4)	C20—C21	1.468 (3)
C1—C2	1.382 (4)	C21—C26	1.385 (3)
C2—C3	1.383 (4)	C21—C22	1.399 (3)
C2—H2	0.930	C22—C23	1.394 (3)

C3—C4	1.354 (5)	C23—C24	1.362 (4)
C3—H3	0.930	C23—H23	0.930
C4—C5	1.365 (5)	C24—C25	1.373 (4)
C4—H4	0.930	C24—H24	0.930
C5—C6	1.380 (4)	C25—C26	1.377 (3)
C5—H5	0.930	C25—H25	0.930
C6—H6	0.930	C26—H26	0.930
C7—C8	1.384 (3)	C27—C28	1.469 (4)
C7—C12	1.390 (3)	C27—H27A	0.970
C8—C9	1.379 (4)	C27—H27B	0.970
C8—H8	0.930	C28—H28A	0.960
C9—C10	1.367 (4)	C28—H28B	0.960
C9—H9	0.930	C28—H28C	0.960
O1—P1—C1	113.18 (10)	C18—C13—P1	118.67 (18)
O1—P1—C13	110.35 (10)	C14—C13—P1	121.96 (19)
C1—P1—C13	107.37 (11)	C15—C14—C13	119.8 (3)
O1—P1—C7	110.87 (10)	C15—C14—H14	120.1
C1—P1—C7	108.51 (10)	C13—C14—H14	120.1
C13—P1—C7	106.26 (10)	C16—C15—C14	120.4 (3)
C22—O2—C27	117.9 (2)	C16—C15—H15	119.8
C19—N1—C20	108.82 (17)	C14—C15—H15	119.8
C19—N1—N4	124.54 (18)	C17—C16—C15	120.3 (3)
C20—N1—N4	125.86 (18)	C17—C16—H16	119.9
C19—N2—N3	112.96 (17)	C15—C16—H16	119.9
C19—N2—H2A	126 (2)	C16—C17—C18	120.3 (3)
N3—N2—H2A	121 (2)	C16—C17—H17	119.9
C20—N3—N2	104.53 (16)	C18—C17—H17	119.9
N1—N4—H4A	106 (3)	C13—C18—C17	119.9 (3)
N1—N4—H4B	104 (3)	C13—C18—H18	120.1
H4A—N4—H4B	111 (3)	C17—C18—H18	120.1
C6—C1—C2	118.4 (2)	N2—C19—N1	103.67 (17)
C6—C1—P1	118.5 (2)	N2—C19—S1	129.52 (17)
C2—C1—P1	122.85 (19)	N1—C19—S1	126.73 (17)
C1—C2—C3	120.4 (3)	N3—C20—N1	109.98 (18)
C1—C2—H2	119.8	N3—C20—C21	122.72 (18)
C3—C2—H2	119.8	N1—C20—C21	127.22 (18)
C4—C3—C2	120.5 (3)	C26—C21—C22	119.2 (2)
C4—C3—H3	119.8	C26—C21—C20	117.93 (19)
C2—C3—H3	119.8	C22—C21—C20	122.7 (2)
C3—C4—C5	120.0 (3)	O2—C22—C23	124.6 (2)
C3—C4—H4	120.0	O2—C22—C21	116.40 (19)
C5—C4—H4	120.0	C23—C22—C21	119.0 (2)
C4—C5—C6	120.4 (3)	C24—C23—C22	120.5 (2)
C4—C5—H5	119.8	C24—C23—H23	119.7
C6—C5—H5	119.8	C22—C23—H23	119.7
C5—C6—C1	120.4 (3)	C23—C24—C25	120.9 (2)
C5—C6—H6	119.8	C23—C24—H24	119.6
C1—C6—H6	119.8	C25—C24—H24	119.6
C8—C7—C12	118.9 (2)	C24—C25—C26	119.5 (2)

C8—C7—P1	116.74 (18)	C24—C25—H25	120.2
C12—C7—P1	124.27 (17)	C26—C25—H25	120.2
C9—C8—C7	120.4 (3)	C25—C26—C21	120.9 (2)
C9—C8—H8	119.8	C25—C26—H26	119.5
C7—C8—H8	119.8	C21—C26—H26	119.5
C10—C9—C8	120.1 (3)	O2—C27—C28	108.9 (2)
C10—C9—H9	120.0	O2—C27—H27A	109.9
C8—C9—H9	120.0	C28—C27—H27A	109.9
C11—C10—C9	120.3 (3)	O2—C27—H27B	109.9
C11—C10—H10	119.9	C28—C27—H27B	109.9
C9—C10—H10	119.9	H27A—C27—H27B	108.3
C10—C11—C12	120.5 (3)	C27—C28—H28A	109.5
C10—C11—H11	119.8	C27—C28—H28B	109.5
C12—C11—H11	119.8	H28A—C28—H28B	109.5
C11—C12—C7	119.8 (2)	C27—C28—H28C	109.5
C11—C12—H12	120.1	H28A—C28—H28C	109.5
C7—C12—H12	120.1	H28B—C28—H28C	109.5
C18—C13—C14	119.3 (2)		
C19—N2—N3—C20	0.2 (2)	C13—C14—C15—C16	0.8 (4)
O1—P1—C1—C6	−71.7 (2)	C14—C15—C16—C17	0.1 (4)
C13—P1—C1—C6	50.3 (2)	C15—C16—C17—C18	−0.9 (5)
C7—P1—C1—C6	164.8 (2)	C14—C13—C18—C17	0.1 (4)
O1—P1—C1—C2	102.7 (2)	P1—C13—C18—C17	−176.5 (2)
C13—P1—C1—C2	−135.2 (2)	C16—C17—C18—C13	0.8 (4)
C7—P1—C1—C2	−20.8 (2)	N3—N2—C19—N1	0.9 (2)
C6—C1—C2—C3	−1.3 (4)	N3—N2—C19—S1	−175.86 (17)
P1—C1—C2—C3	−175.8 (2)	C20—N1—C19—N2	−1.7 (2)
C1—C2—C3—C4	1.0 (5)	N4—N1—C19—N2	−172.0 (2)
C2—C3—C4—C5	−0.1 (6)	C20—N1—C19—S1	175.21 (16)
C3—C4—C5—C6	−0.4 (6)	N4—N1—C19—S1	4.9 (3)
C4—C5—C6—C1	0.0 (6)	N2—N3—C20—N1	−1.3 (2)
C2—C1—C6—C5	0.8 (5)	N2—N3—C20—C21	175.65 (19)
P1—C1—C6—C5	175.5 (3)	C19—N1—C20—N3	2.0 (2)
O1—P1—C7—C8	9.8 (2)	N4—N1—C20—N3	172.1 (2)
C1—P1—C7—C8	134.69 (19)	C19—N1—C20—C21	−174.8 (2)
C13—P1—C7—C8	−110.12 (19)	N4—N1—C20—C21	−4.7 (3)
O1—P1—C7—C12	−172.82 (18)	N3—C20—C21—C26	−43.7 (3)
C1—P1—C7—C12	−47.9 (2)	N1—C20—C21—C26	132.7 (2)
C13—P1—C7—C12	67.3 (2)	N3—C20—C21—C22	130.9 (2)
C12—C7—C8—C9	0.1 (4)	N1—C20—C21—C22	−52.7 (3)
P1—C7—C8—C9	177.6 (2)	C27—O2—C22—C23	7.7 (4)
C7—C8—C9—C10	0.1 (4)	C27—O2—C22—C21	−170.5 (2)
C8—C9—C10—C11	−0.4 (5)	C26—C21—C22—O2	177.25 (19)
C9—C10—C11—C12	0.5 (4)	C20—C21—C22—O2	2.7 (3)
C10—C11—C12—C7	−0.3 (4)	C26—C21—C22—C23	−1.1 (3)
C8—C7—C12—C11	0.0 (3)	C20—C21—C22—C23	−175.6 (2)
P1—C7—C12—C11	−177.30 (19)	O2—C22—C23—C24	−176.9 (2)
O1—P1—C13—C18	−7.5 (2)	C21—C22—C23—C24	1.3 (4)
C1—P1—C13—C18	−131.23 (19)	C22—C23—C24—C25	−0.1 (4)

C7—P1—C13—C18	112.81 (19)	C23—C24—C25—C26	−1.2 (4)
O1—P1—C13—C14	176.10 (19)	C24—C25—C26—C21	1.4 (4)
C1—P1—C13—C14	52.3 (2)	C22—C21—C26—C25	−0.3 (3)
C7—P1—C13—C14	−63.6 (2)	C20—C21—C26—C25	174.5 (2)
C18—C13—C14—C15	−0.9 (4)	C22—O2—C27—C28	175.6 (2)
P1—C13—C14—C15	175.5 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···O1	0.862 (18)	1.830 (19)	2.682 (2)	169 (3)
N4—H4A···S1	0.846 (18)	2.68 (3)	3.169 (2)	118 (3)
N4—H4B···O2	0.847 (18)	2.35 (3)	2.904 (3)	124 (3)
N4—H4B···S1^{i}	0.847 (18)	2.99 (3)	3.533 (2)	124 (3)

Symmetry codes: (i) −*x*, −*y*+2, −*z*.

Fig. 1

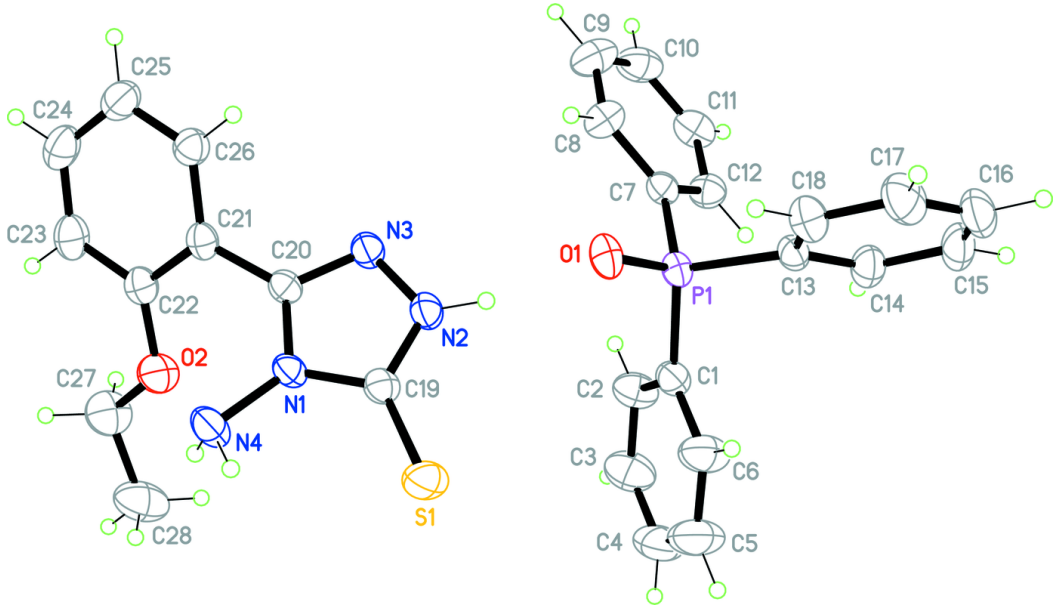

C25
C24
C26
C23
C21
C20
N3
C22
N2
O2
C27
N1
C19
N4
S1
C28
C9
C10
C11
C8
C12
C7
C18
C17
C16
O1
P1
C13
C14
C15
C1
C2
C3
C6
C4
C5

Fig. 2