Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Diethyl 3-methyl-5-(triphenylphosphoranyleneamino)thiophene-2,4-dicarboxylate

Yuan-Hong Jiao,[a] Qian Zhang[a] and Seik Weng Ng[b]*

[a]School of Chemical and Materials Engineering, Huangshi Institute of Technology, Huangshi 435003, People's Republic of China, and [b]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

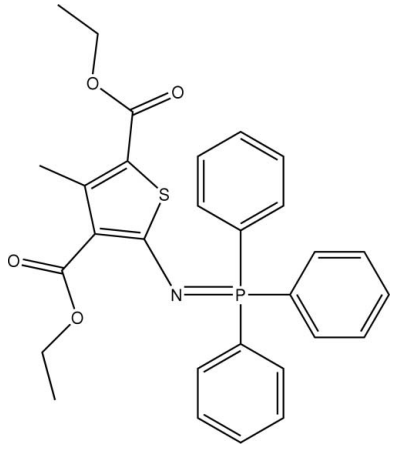
Received 17 October 2007; accepted 19 October 2007

Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.006 Å; R factor = 0.063; wR factor = 0.172; data-to-parameter ratio = 16.2.

The title iminophosphorane, $C_{29}H_{28}NO_4PS$, a thiophene compound with the sterically crowded –N=P$(C_6H_5)_3$ substituent in the α-position of the aromatic ring, features an N=P double bond of length 1.572 (3) Å. One of the phenyl rings is coplanar with the plane of the three *ipso*-C atoms, whereas the other two are approximately orthogonal.

Related literature

The synthesis of the amine precursor made use of the action of elemental sulfur on ethyl 2-cyanoacetate; see Gewald & Martin (1981). The iminophosphorane is a useful protecting group for primary amines; see Liu & Liu (1992). For a recent short account of the aza-Witting reaction, see: Palacios *et al.* (2007). For an older review of iminophosphoranes, see Wamhoff *et al.* (1995).



Experimental

Crystal data

$C_{29}H_{28}NO_4PS$
M_r = 517.55
Monoclinic, $P2_1/n$
a = 8.937 (1) Å
b = 22.593 (1) Å
c = 13.745 (2) Å
β = 105.411 (1)°
V = 2675.5 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 295 (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.949, T_{max} = 0.979
15880 measured reflections
4717 independent reflections
2798 reflections with $I > 2\sigma(I)$
R_{int} = 0.075

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.172
S = 0.94
4717 reflections
292 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2007).

We thank the Huangshi Institute of Technology (grant No. 07yjz18B) and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2341).

References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bruker (2001). *SAINT* and *SMART*. Bruker AXS Inc., Madison, Winconsin, USA.
Gewald, K. & Martin, A. (1981). *J. Prakt. Chem.* **323**, 843–846.
Liu, S.-T. & Liu, C.-Y. (1992). *J. Org. Chem.* **57**, 6079–6080.
Palacios, F., Alonso, C., Aparicio, D., Rubiales, G. & de los Santos, J. M. (2007). *Tetrahedron*, **63**, 523–575.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Wamhoff, H., Richardt, G. & Stölben, S. (1995). *Adv. Heterocycl. Chem.* **64**, 159–249.
Westrip, S. P. (2007). *publCIF*. In preparation.

  doi:10.1107/S1600536807051689

supplementary materials

Acta Cryst. (2007). E**63**, o4466 [**doi:10.1107/S1600536807051689**]

Diethyl 3-methyl-5-(triphenylphosphoranyleneamino)thiophene-2,4-dicarboxylate

Y.-H. Jiao, Q. Zhang and S. W. Ng

Experimental

Ethyl 3-oxobutanoate (1.30 g, 10 mmol), ethyl 2-cyanoacetate (1.13 g, 10 mmol) and sulfur (0.32 g, 10 mmol) in anhydrous ethanol (30 ml) were refluxed for 5 h. Water (200 ml) was added to precipitate a white product that was purified by re-crystallization from ethanol. Diethyl 2-amino-4-methylthiophene-3,5-dicarboxylate, m.p. 386–388 K was obtained in 40% yield. CH&N Elemental analysis. Calc. for $C_{11}H_{15}NO_4S$: C 51.35, H 5.88, N 5.44%. Found: C 51.20, H 5.92. N, 5.37%.

Diethyl 2-amino-4-methylthiophene-3,5-dicarboxylate (2.57 g, 10 mmol), triphenylphosphine (5.3 g, 20 mmol) and hexachloroethane (4.7 g, 30 mmol) were dissolved in acetonitrile (20 ml). Triethylamine (3.0 g, 30 mmol) was added. The mixture was stirred for 4 h. The solvent was removed under reduced pressure and the residue quenched with cold water (300 ml). The organic product was extracted with dichloromethane (3× 30 ml). The solvent was evaporated and the residue was purified by recrystallization from ethanol to give the title compound in 90% yield. CH&N Elemental analysis, Calc. for $C_{29}H_{28}NO_4PS$: C 67.30, H 5.45, N 2.71%. Found: C 67.21, H 5.72, N, 2.77%.

Refinement

Owing to the small number of 'observeds', the three phenyl rings were refined as rigid hexagons of 1.39 A% sides. There is some disorder in the ethyl groups, and a tight restraint of restraining the carbon-carbon distance to 1.540±0.002 Å was used. The methyl groups were rotated to fit the electron density. All H-atoms were generated geometrically [C–H 0.93–0.97 Å], and were included in the refinement in the riding model approximation, with U(H) set to 1.2–1.5U_{eq}(C).

Figures

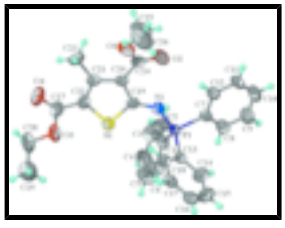


Fig. 1. Thermal ellipsoid plot $C_{29}H_{28}NO_4PS$; displacement ellipsoids are drawn at the 50% probability level, and H atoms as spheres of arbitrary radius.

Diethyl 3-methyl-5-(triphenylphosphoranyleneamino)thiophene-2,4-dicarboxylate

Crystal data

$C_{29}H_{28}NO_4PS$	F_{000} = 1088
M_r = 517.55	D_x = 1.285 Mg m^{-3}
Monoclinic, $P2_1/n$	Melting point: 438-440 K
Hall symbol: -P 2yn	Mo $K\alpha$ radiation

	λ = 0.71073 Å
a = 8.937 (1) Å	Cell parameters from 2244 reflections
b = 22.593 (1) Å	θ = 2.4–19.8º
c = 13.745 (2) Å	μ = 0.22 mm^{-1}
β = 105.411 (1)º	*T* = 295 (2) K
V = 2675.5 (5) Å^3	Block, colorless
Z = 4	0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART area-detector diffractometer	4717 independent reflections
Radiation source: fine-focus sealed tube	2798 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.075
T = 291(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 1.8º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −10→8
T_{min} = 0.949, T_{max} = 0.979	*k* = −26→26
15880 measured reflections	*l* = −16→16

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.063	H-atom parameters constrained
$wR(F^2)$ = 0.172	$w = 1/[\sigma^2(F_o^2) + (0.087P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.94	$(\Delta/\sigma)_{max} < 0.001$
4717 reflections	$\Delta\rho_{max}$ = 0.40 e Å^{-3}
292 parameters	$\Delta\rho_{min}$ = −0.27 e Å^{-3}
2 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.67049 (11)	0.34647 (4)	0.75632 (7)	0.0598 (3)
P1	0.64234 (10)	0.21622 (4)	0.87844 (6)	0.0449 (3)
O1	0.3029 (3)	0.42360 (13)	0.92798 (19)	0.0818 (9)
O2	0.4200 (3)	0.34519 (13)	1.0107 (2)	0.0806 (9)
O3	0.7395 (4)	0.43065 (13)	0.6249 (3)	0.1139 (13)
O4	0.6563 (4)	0.51282 (14)	0.6774 (3)	0.1179 (13)
N1	0.5676 (3)	0.27813 (13)	0.8885 (2)	0.0540 (8)
C1	0.8370 (2)	0.21594 (11)	0.86514 (18)	0.0513 (9)
C2	0.9530 (3)	0.23932 (11)	0.94401 (16)	0.0702 (11)

H2	0.9288	0.2536	1.0014	0.084*
C3	1.1053 (2)	0.24131 (13)	0.9371 (2)	0.0854 (13)
H3	1.1830	0.2570	0.9899	0.102*
C4	1.1416 (2)	0.21992 (14)	0.8513 (2)	0.0908 (15)
H4	1.2435	0.2212	0.8467	0.109*
C5	1.0256 (3)	0.19653 (13)	0.77244 (19)	0.0962 (16)
H5	1.0498	0.1822	0.7150	0.115*
C6	0.8733 (3)	0.19454 (12)	0.77935 (16)	0.0716 (11)
H6	0.7957	0.1789	0.7266	0.086*
C7	0.6511 (3)	0.17827 (10)	0.99452 (13)	0.0466 (8)
C8	0.7473 (3)	0.12943 (11)	1.02329 (17)	0.0632 (10)
H8	0.8103	0.1169	0.9833	0.076*
C9	0.7494 (3)	0.09939 (10)	1.11189 (19)	0.0772 (12)
H9	0.8137	0.0667	1.1311	0.093*
C10	0.6553 (3)	0.11819 (12)	1.17173 (14)	0.0823 (13)
H10	0.6566	0.0981	1.2310	0.099*
C11	0.5591 (3)	0.16702 (12)	1.14297 (17)	0.0775 (13)
H11	0.4961	0.1796	1.1830	0.093*
C12	0.5570 (3)	0.19706 (10)	1.05437 (18)	0.0577 (10)
H12	0.4926	0.2297	1.0351	0.069*
C13	0.5240 (2)	0.17261 (9)	0.77717 (14)	0.0460 (8)
C14	0.5417 (3)	0.11152 (10)	0.77592 (17)	0.0771 (12)
H14	0.6200	0.0932	0.8249	0.093*
C15	0.4424 (3)	0.07785 (8)	0.7014 (2)	0.0902 (15)
H15	0.4543	0.0370	0.7006	0.108*
C16	0.3254 (3)	0.10527 (11)	0.62814 (16)	0.0715 (12)
H16	0.2590	0.0827	0.5783	0.086*
C17	0.3077 (2)	0.16637 (12)	0.62939 (15)	0.0611 (10)
H17	0.2294	0.1847	0.5804	0.073*
C18	0.4070 (3)	0.20003 (8)	0.70390 (16)	0.0533 (9)
H18	0.3951	0.2409	0.7047	0.064*
C19	0.5760 (4)	0.33193 (15)	0.8499 (2)	0.0462 (8)
C20	0.5090 (4)	0.38404 (15)	0.8752 (2)	0.0472 (8)
C21	0.5394 (4)	0.43465 (15)	0.8214 (3)	0.0520 (9)
C22	0.6268 (4)	0.42147 (15)	0.7558 (3)	0.0558 (9)
C23	0.4827 (5)	0.49637 (16)	0.8329 (3)	0.0773 (12)
H23A	0.5430	0.5244	0.8068	0.116*
H23B	0.4935	0.5044	0.9030	0.116*
H23C	0.3755	0.4997	0.7961	0.116*
C24	0.4114 (4)	0.38135 (17)	0.9452 (3)	0.0523 (9)
C25	0.1986 (5)	0.4243 (2)	0.9921 (3)	0.1076 (18)
H25A	0.2059	0.4619	1.0272	0.129*
H25B	0.2250	0.3929	1.0418	0.129*
C26	0.0342 (5)	0.4153 (3)	0.9243 (4)	0.143 (2)
H26A	−0.0398	0.4240	0.9617	0.215*
H26B	0.0219	0.3750	0.9014	0.215*
H26C	0.0174	0.4413	0.8671	0.215*
C27	0.6750 (5)	0.4608 (2)	0.6860 (3)	0.0719 (11)
C28	0.7881 (8)	0.4648 (2)	0.5496 (4)	0.134 (2)

H28A	0.8989	0.4722	0.5711	0.160*
H28B	0.7345	0.5026	0.5387	0.160*
C29	0.7469 (9)	0.4281 (2)	0.4527 (4)	0.162 (3)
H29A	0.7971	0.4446	0.4052	0.243*
H29B	0.6365	0.4286	0.4241	0.243*
H29C	0.7812	0.3881	0.4678	0.243*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0601 (6)	0.0574 (6)	0.0713 (7)	0.0094 (5)	0.0340 (5)	0.0071 (5)
P1	0.0398 (5)	0.0532 (6)	0.0436 (5)	0.0041 (4)	0.0147 (4)	0.0020 (4)
O1	0.0735 (19)	0.109 (2)	0.0703 (18)	0.0396 (18)	0.0328 (15)	0.0061 (16)
O2	0.077 (2)	0.100 (2)	0.0732 (19)	0.0247 (17)	0.0347 (16)	0.0135 (17)
O3	0.176 (4)	0.076 (2)	0.126 (3)	0.016 (2)	0.105 (3)	0.0294 (19)
O4	0.155 (4)	0.059 (2)	0.164 (3)	0.008 (2)	0.085 (3)	0.022 (2)
N1	0.0479 (18)	0.0565 (19)	0.0644 (18)	0.0074 (14)	0.0266 (15)	0.0002 (15)
C1	0.049 (2)	0.052 (2)	0.058 (2)	0.0057 (17)	0.0227 (19)	0.0087 (17)
C2	0.045 (2)	0.088 (3)	0.081 (3)	−0.003 (2)	0.022 (2)	−0.022 (2)
C3	0.050 (3)	0.106 (4)	0.101 (3)	−0.005 (2)	0.021 (3)	−0.007 (3)
C4	0.060 (3)	0.119 (4)	0.106 (4)	0.015 (3)	0.044 (3)	0.021 (3)
C5	0.075 (3)	0.147 (5)	0.081 (3)	0.028 (3)	0.047 (3)	0.007 (3)
C6	0.060 (3)	0.101 (3)	0.061 (2)	0.013 (2)	0.028 (2)	0.004 (2)
C7	0.0391 (19)	0.060 (2)	0.0406 (18)	−0.0060 (17)	0.0094 (16)	−0.0035 (16)
C8	0.059 (2)	0.077 (3)	0.050 (2)	0.004 (2)	0.0076 (19)	0.0101 (19)
C9	0.076 (3)	0.086 (3)	0.057 (2)	0.003 (2)	−0.003 (2)	0.023 (2)
C10	0.099 (4)	0.096 (3)	0.048 (2)	−0.021 (3)	0.011 (2)	0.015 (2)
C11	0.096 (3)	0.095 (3)	0.052 (2)	−0.021 (3)	0.038 (2)	−0.005 (2)
C12	0.056 (2)	0.066 (2)	0.052 (2)	−0.0089 (19)	0.0176 (19)	−0.0024 (18)
C13	0.044 (2)	0.052 (2)	0.0444 (19)	0.0036 (16)	0.0151 (16)	0.0041 (16)
C14	0.084 (3)	0.067 (3)	0.065 (3)	0.014 (2)	−0.006 (2)	−0.003 (2)
C15	0.117 (4)	0.065 (3)	0.071 (3)	0.001 (3)	−0.006 (3)	−0.012 (2)
C16	0.076 (3)	0.083 (3)	0.056 (2)	−0.018 (2)	0.017 (2)	−0.014 (2)
C17	0.050 (2)	0.085 (3)	0.048 (2)	0.002 (2)	0.0118 (18)	0.003 (2)
C18	0.048 (2)	0.063 (2)	0.049 (2)	0.0018 (18)	0.0140 (18)	0.0054 (18)
C19	0.0350 (19)	0.056 (2)	0.0456 (19)	0.0011 (16)	0.0066 (15)	−0.0026 (16)
C20	0.041 (2)	0.052 (2)	0.0447 (19)	0.0047 (17)	0.0040 (16)	−0.0086 (16)
C21	0.044 (2)	0.056 (2)	0.050 (2)	0.0051 (17)	0.0010 (17)	−0.0118 (17)
C22	0.053 (2)	0.050 (2)	0.062 (2)	−0.0040 (17)	0.0111 (19)	0.0037 (18)
C23	0.095 (3)	0.052 (2)	0.083 (3)	0.004 (2)	0.022 (3)	−0.015 (2)
C24	0.044 (2)	0.064 (2)	0.045 (2)	0.0091 (19)	0.0045 (17)	−0.0135 (18)
C25	0.093 (4)	0.159 (5)	0.084 (3)	0.046 (4)	0.047 (3)	−0.005 (3)
C26	0.100 (5)	0.211 (7)	0.136 (5)	−0.010 (5)	0.063 (4)	0.011 (5)
C27	0.068 (3)	0.063 (3)	0.088 (3)	0.002 (2)	0.026 (2)	0.006 (2)
C28	0.198 (7)	0.097 (4)	0.141 (5)	−0.003 (4)	0.106 (5)	0.039 (4)
C29	0.255 (9)	0.125 (5)	0.114 (5)	0.019 (5)	0.061 (6)	0.026 (4)

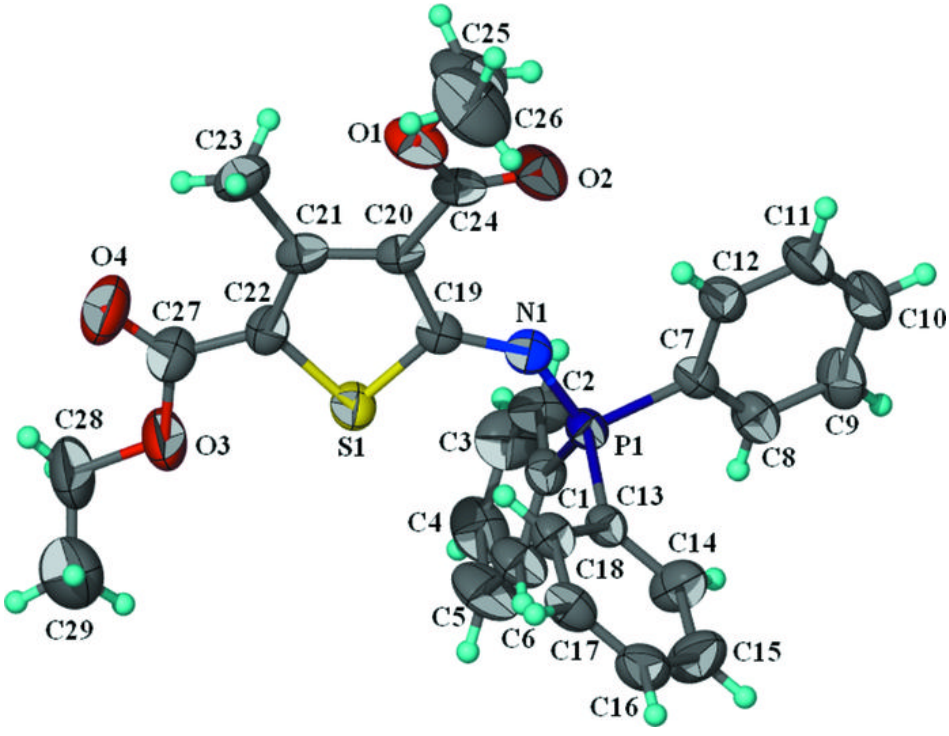
Geometric parameters (Å, °)

S1—C22	1.738 (4)	C12—H12	0.9300
S1—C19	1.747 (3)	C13—C14	1.3900
P1—N1	1.572 (3)	C13—C18	1.3900
P1—C7	1.7947 (18)	C14—C15	1.3900
P1—C1	1.7971 (19)	C14—H14	0.9300
P1—C13	1.8013 (18)	C15—C16	1.3900
O1—C24	1.336 (4)	C15—H15	0.9300
O1—C25	1.442 (5)	C16—C17	1.3900
O2—C24	1.203 (4)	C16—H16	0.9300
O3—C27	1.326 (5)	C17—C18	1.3900
O3—C28	1.448 (5)	C17—H17	0.9300
O4—C27	1.189 (5)	C18—H18	0.9300
N1—C19	1.336 (4)	C19—C20	1.406 (4)
C1—C2	1.3900	C20—C21	1.426 (5)
C1—C6	1.3900	C20—C24	1.462 (5)
C2—C3	1.3900	C21—C22	1.373 (5)
C2—H2	0.9300	C21—C23	1.506 (5)
C3—C4	1.3900	C22—C27	1.454 (5)
C3—H3	0.9300	C23—H23A	0.9600
C4—C5	1.3900	C23—H23B	0.9600
C4—H4	0.9300	C23—H23C	0.9600
C5—C6	1.3900	C25—C26	1.530 (7)
C5—H5	0.9300	C25—H25A	0.9700
C6—H6	0.9300	C25—H25B	0.9700
C7—C8	1.3900	C26—H26A	0.9600
C7—C12	1.3900	C26—H26B	0.9600
C8—C9	1.3900	C26—H26C	0.9600
C8—H8	0.9300	C28—C29	1.528 (7)
C9—C10	1.3900	C28—H28A	0.9700
C9—H9	0.9300	C28—H28B	0.9700
C10—C11	1.3900	C29—H29A	0.9600
C10—H10	0.9300	C29—H29B	0.9600
C11—C12	1.3900	C29—H29C	0.9600
C11—H11	0.9300		
C22—S1—C19	91.98 (17)	C17—C16—H16	120.0
N1—P1—C7	105.43 (13)	C15—C16—H16	120.0
N1—P1—C1	117.24 (14)	C16—C17—C18	120.0
C7—P1—C1	105.91 (11)	C16—C17—H17	120.0
N1—P1—C13	112.26 (14)	C18—C17—H17	120.0
C7—P1—C13	107.67 (11)	C17—C18—C13	120.0
C1—P1—C13	107.75 (12)	C17—C18—H18	120.0
C24—O1—C25	117.1 (3)	C13—C18—H18	120.0
C27—O3—C28	116.2 (4)	N1—C19—C20	126.2 (3)
C19—N1—P1	134.8 (2)	N1—C19—S1	123.5 (3)
C2—C1—C6	120.0	C20—C19—S1	110.2 (3)
C2—C1—P1	117.45 (15)	C19—C20—C21	113.0 (3)

C6—C1—P1	122.54 (15)	C19—C20—C24	119.7 (3)
C1—C2—C3	120.0	C21—C20—C24	127.1 (3)
C1—C2—H2	120.0	C22—C21—C20	112.7 (3)
C3—C2—H2	120.0	C22—C21—C23	122.4 (3)
C2—C3—C4	120.0	C20—C21—C23	124.9 (3)
C2—C3—H3	120.0	C21—C22—C27	128.4 (4)
C4—C3—H3	120.0	C21—C22—S1	112.0 (3)
C3—C4—C5	120.0	C27—C22—S1	119.5 (3)
C3—C4—H4	120.0	C21—C23—H23A	109.5
C5—C4—H4	120.0	C21—C23—H23B	109.5
C6—C5—C4	120.0	H23A—C23—H23B	109.5
C6—C5—H5	120.0	C21—C23—H23C	109.5
C4—C5—H5	120.0	H23A—C23—H23C	109.5
C5—C6—C1	120.0	H23B—C23—H23C	109.5
C5—C6—H6	120.0	O2—C24—O1	121.1 (3)
C1—C6—H6	120.0	O2—C24—C20	126.2 (3)
C8—C7—C12	120.0	O1—C24—C20	112.7 (3)
C8—C7—P1	120.64 (14)	O1—C25—C26	107.4 (4)
C12—C7—P1	119.33 (14)	O1—C25—H25A	110.2
C9—C8—C7	120.0	C26—C25—H25A	110.2
C9—C8—H8	120.0	O1—C25—H25B	110.2
C7—C8—H8	120.0	C26—C25—H25B	110.2
C8—C9—C10	120.0	H25A—C25—H25B	108.5
C8—C9—H9	120.0	C25—C26—H26A	109.5
C10—C9—H9	120.0	C25—C26—H26B	109.5
C11—C10—C9	120.0	H26A—C26—H26B	109.5
C11—C10—H10	120.0	C25—C26—H26C	109.5
C9—C10—H10	120.0	H26A—C26—H26C	109.5
C10—C11—C12	120.0	H26B—C26—H26C	109.5
C10—C11—H11	120.0	O4—C27—O3	121.2 (4)
C12—C11—H11	120.0	O4—C27—C22	127.7 (4)
C11—C12—C7	120.0	O3—C27—C22	111.0 (4)
C11—C12—H12	120.0	O3—C28—C29	106.6 (4)
C7—C12—H12	120.0	O3—C28—H28A	110.4
C14—C13—C18	120.0	C29—C28—H28A	110.4
C14—C13—P1	120.49 (14)	O3—C28—H28B	110.4
C18—C13—P1	119.35 (14)	C29—C28—H28B	110.4
C15—C14—C13	120.0	H28A—C28—H28B	108.6
C15—C14—H14	120.0	C28—C29—H29A	109.5
C13—C14—H14	120.0	C28—C29—H29B	109.5
C14—C15—C16	120.0	H29A—C29—H29B	109.5
C14—C15—H15	120.0	C28—C29—H29C	109.5
C16—C15—H15	120.0	H29A—C29—H29C	109.5
C17—C16—C15	120.0	H29B—C29—H29C	109.5
C7—P1—N1—C19	152.4 (3)	C13—C14—C15—C16	0.0
C1—P1—N1—C19	34.9 (4)	C14—C15—C16—C17	0.0
C13—P1—N1—C19	−90.7 (4)	C15—C16—C17—C18	0.0
N1—P1—C1—C2	61.91 (19)	C16—C17—C18—C13	0.0
C7—P1—C1—C2	−55.34 (17)	C14—C13—C18—C17	0.0

C13—P1—C1—C2	−170.34 (14)	P1—C13—C18—C17	−175.43 (17)
N1—P1—C1—C6	−116.83 (18)	P1—N1—C19—C20	−174.2 (3)
C7—P1—C1—C6	125.92 (16)	P1—N1—C19—S1	7.6 (5)
C13—P1—C1—C6	10.92 (18)	C22—S1—C19—N1	−179.3 (3)
C6—C1—C2—C3	0.0	C22—S1—C19—C20	2.3 (3)
P1—C1—C2—C3	−178.77 (18)	N1—C19—C20—C21	179.6 (3)
C1—C2—C3—C4	0.0	S1—C19—C20—C21	−2.0 (3)
C2—C3—C4—C5	0.0	N1—C19—C20—C24	−4.1 (5)
C3—C4—C5—C6	0.0	S1—C19—C20—C24	174.3 (2)
C4—C5—C6—C1	0.0	C19—C20—C21—C22	0.5 (4)
C2—C1—C6—C5	0.0	C24—C20—C21—C22	−175.4 (3)
P1—C1—C6—C5	178.70 (19)	C19—C20—C21—C23	−180.0 (3)
N1—P1—C7—C8	−162.47 (16)	C24—C20—C21—C23	4.1 (5)
C1—P1—C7—C8	−37.55 (16)	C20—C21—C22—C27	177.3 (3)
C13—P1—C7—C8	77.50 (16)	C23—C21—C22—C27	−2.2 (6)
N1—P1—C7—C12	19.46 (18)	C20—C21—C22—S1	1.2 (4)
C1—P1—C7—C12	144.38 (14)	C23—C21—C22—S1	−178.3 (3)
C13—P1—C7—C12	−100.57 (15)	C19—S1—C22—C21	−2.0 (3)
C12—C7—C8—C9	0.0	C19—S1—C22—C27	−178.5 (3)
P1—C7—C8—C9	−178.06 (17)	C25—O1—C24—O2	0.8 (5)
C7—C8—C9—C10	0.0	C25—O1—C24—C20	179.1 (3)
C8—C9—C10—C11	0.0	C19—C20—C24—O2	27.2 (5)
C9—C10—C11—C12	0.0	C21—C20—C24—O2	−157.1 (4)
C10—C11—C12—C7	0.0	C19—C20—C24—O1	−150.9 (3)
C8—C7—C12—C11	0.0	C21—C20—C24—O1	24.8 (5)
P1—C7—C12—C11	178.08 (17)	C24—O1—C25—C26	−118.9 (4)
N1—P1—C13—C14	−159.09 (16)	C28—O3—C27—O4	1.2 (7)
C7—P1—C13—C14	−43.48 (16)	C28—O3—C27—C22	178.1 (4)
C1—P1—C13—C14	70.34 (16)	C21—C22—C27—O4	5.4 (7)
N1—P1—C13—C18	16.32 (19)	S1—C22—C27—O4	−178.7 (4)
C7—P1—C13—C18	131.93 (14)	C21—C22—C27—O3	−171.2 (4)
C1—P1—C13—C18	−114.25 (15)	S1—C22—C27—O3	4.6 (5)
C18—C13—C14—C15	0.0	C27—O3—C28—C29	−140.1 (5)
P1—C13—C14—C15	175.38 (17)		

Fig. 1


C25
C26
O1
O2
C23
C24
C21
C20
C11
C12
O4
C22
C19
N1
C10
C27
C7
C2
C28
O3
S1
C3
P1
C9
C8
C1
C13
C4
C14
C18
C5
C6
C17
C15
C29
C16