Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4,5-Bis[bis(diethylamino)phosphino]-9,9-dimethylxanthene

David J Adams, John Fawcett, Eric G. Hope* and Alison M. Stuart

Department of Chemistry, University of Leicester, Leicester LE1 7RH, England

Correspondence e-mail: egh1@leicester.ac.uk

Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.050
wR factor = 0.139
Data-to-parameter ratio = 16.1

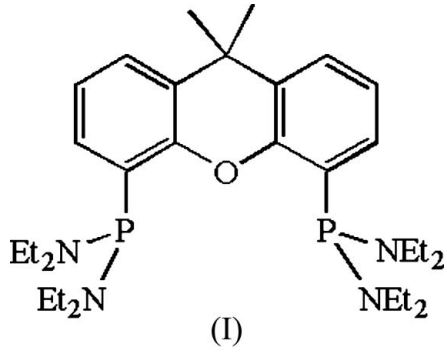
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The structure of the title compound, $C_{31}H_{52}N_4OP_2$, reveals a near-planar xanthene skeleton.

Received 24 June 2005
Accepted 2 August 2005
Online 6 August 2005

Comment

Xantphos, and related bidentate ligands with the xanthene backbone, have been shown to be remarkable ligands for the rhodium-catalysed hydroformylation of long-chain alkenes, giving exceptionally high selectivity to the industrially useful linear aldehyde (linear/branched ratio = 50:1) (Kranenburg *et al.*, 1995). The title compound, (I), was prepared according to the literature procedure of Goertz *et al.* (2001) as an intermediate in work directed towards the synthesis of perfluoroalkylated derivatives of Xantphos (Adams *et al.*, 2004). Crystals suitable for X-ray analysis were grown from hexane solution. The bond lengths and angles within the structure are unremarkable. The molecule has a pseudo-*C*2 axis of symmetry through O1 and C6, such that the lone pairs on phosphorus point either in front or behind an approximately planar xanthene skeleton.



(I)

Experimental

The title compound was prepared according to the literature procedure of Goertz *et al.* (2001), Crystals suitable for X-ray analysis were grown from a hexane solution.

Crystal data

$C_{31}H_{52}N_4OP_2$
M_r = 558.71
Monoclinic, $P2_1/c$
a = 18.799 (1) Å
b = 11.4968 (6) Å
c = 15.2179 (8) Å
β = 100.880 (1)°
V = 3229.9 (3) Å^3
Z = 4
D_x = 1.149 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6949 reflections
θ = 2.2–26.9°
μ = 0.16 mm^{-1}
T = 150 (2) K
Block, colourless
0.34 × 0.21 × 0.16 mm

Data collection

Bruker APEX CCD area-detector diffractometer	5685 independent reflections
φ and ω scans	4653 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$R_{int} = 0.034$
$T_{min} = 0.93$, $T_{max} = 0.96$	$\theta_{max} = 25.0^\circ$
22958 measured reflections	$h = -22 \rightarrow 22$
	$k = -13 \rightarrow 13$
	$l = -18 \rightarrow 18$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0817P)^2 + 0.3182P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.050$	$(\Delta/\sigma)_{max} = 0.002$
$wR(F^2) = 0.139$	$\Delta\rho_{max} = 0.53$ e Å^{-3}
$S = 1.06$	$\Delta\rho_{min} = -0.35$ e Å^{-3}
5685 reflections	
353 parameters	
H-atom parameters constrained	

All H atoms were included in calculated positions, riding on the bonded atom [C—H = 0.95 (CH), 0.98 (CH_3) and 0.99 Å (CH_2)], and with U_{iso}(H) values set at $1.5U_{eq}$ of the bonded C atom for methyl H atoms and at $1.2U_{eq}$ for all other H atoms.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2000); software used to prepare material for publication: *SHELXTL*.

The authors thank the EPSRC (DJA) and the Royal Society (AMS) for financial support.

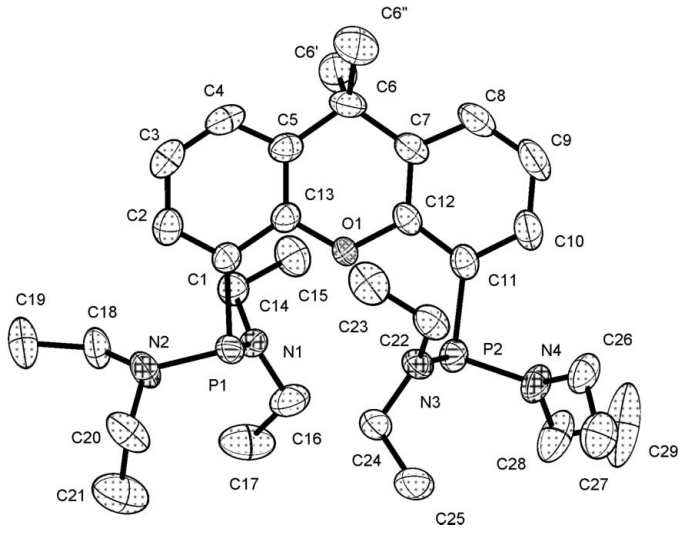


Figure 1
The molecular structure of (I), showing the atom-labelling scheme and 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

References

Adams, D. J., Cole-Hamilton, D. J., Harding, D. A. J., Hope, E. G., Pogorzelec, P. & Stuart, A. M. (2004). *Tetrahedron*, **60**, 4079–4085.

Bruker (1997). *SMART*. Version 5.622. Bruker AXS Inc., Madison, Wisconsin, USA.

Bruker (1999). *SAINT*. Version 6.02. Bruker AXS Inc., Madison, Wisconsin, USA.

Goertz, W., Kamer, P. C. J., van Leeuwen, P. W. N. M. & Vogt, D. (2001). *Chem. Eur. J.* **7**, 1614–1618.

Kranenburg, M., van der Burgt, Y. E. M., Kamer, P. C. J., van Leeuwen, P. W. N. M. (1995). *Organometallics*, **14**, 3081–3089.

Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.

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

Sheldrick, G. M. (2000). *SHELXTL*. Version 6.10. Bruker AXS Inc., Madison, Wisconsin, USA.

supporting information

Acta Cryst. (2005). E**61**, o2831–o2832 [doi:10.1107/S1600536805024797]

4,5-Bis[bis(diethylamino)phosphino]-9,9-dimethylxanthene

David J Adams, John Fawcett, Eric G. Hope and Alison M. Stuart

S1. Comment

Xantphos, and related bidentate ligands with the xanthene backbone, have been shown to be remarkable ligands for the rhodium-catalyzed hydroformylation of long-chain alkenes, giving exceptionally high selectivity to the industrially useful linear aldehyde (linear/branched ratio = 50:1) (Kranenburg *et al.*, 1995). The title compound, (I), was prepared according to the literature procedure of Goertz *et al.* (2001) as an intermediate in work directed towards the synthesis of perfluoro-alkylated derivatives of Xantphos (Adams *et al.*, 2004). Crystals suitable for X-ray analysis were grown from hexane solution. The bond lengths and angles within the structure are unremarkable. The molecule, virtually, has a *C*2 axis of symmetry through O1 and C6 such that the lone pairs on phosphorus point either in front or behind an approximately planar xanthene skeleton.

S2. Experimental

The title compound was prepared according to the literature procedure of Goertz *et al.* (2001), Crystals suitable for X-ray analysis were grown from a hexane solution.

S3. Refinement

All H atoms were included in calculated positions with the atomic coordinates riding on the bonded atom [C—H = 0.95 (CH), 0.98 (CH_3) and 0.99 Å (CH_2)]] and with U_{iso}(H) values set at 1.5U_{eq} of the bonded C atom for methyl H atoms and at 1.2U_{eq} for all other H atoms.

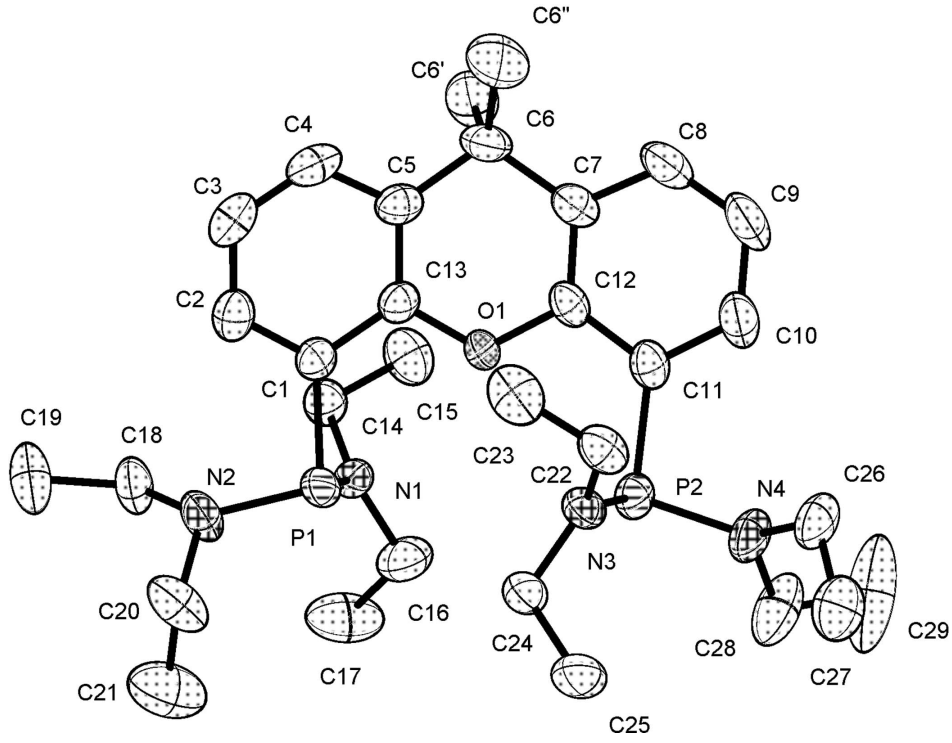


Figure 1

The molecular structure of (I), showing the atom-labelling scheme and 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

4,5-Bis[bis(diethylamino)phosphino]-9,9-dimethylxanthene

Crystal data

$C_{31}H_{52}N_4OP_2$
$M_r = 558.71$
Monoclinic, $P2_1/c$
$a = 18.799\ (1)$ Å
$b = 11.4968\ (6)$ Å
$c = 15.2179\ (8)$ Å
$\beta = 100.880\ (1)°$
$V = 3229.9\ (3)$ Å^3
$Z = 4$
$F(000) = 1216$
$D_x = 1.149$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6949 reflections
$\theta = 2.2–26.9°$
$\mu = 0.16$ mm^{-1}
$T = 150$ K
Block, colourless
0.34 × 0.21 × 0.16 mm

Data collection

Bruker APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.93$, $T_{max} = 0.96$
22958 measured reflections
5685 independent reflections
4653 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.1°$
$h = -22\rightarrow22$
$k = -13\rightarrow13$
$l = -18\rightarrow18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.139$
$S = 1.06$
5685 reflections
353 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0817P)^2 + 0.3182P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.53$ e Å^{-3}
$\Delta\rho_{min} = -0.35$ e Å^{-3}

Special details

Experimental. absorption correction based on 11566 reflections (*SADABS*); R_{int} 0.042 before and 0.029 after correction.
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 $F^2 > \sigma(F^2)$ 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}
P1	0.24064 (3)	0.79839 (4)	0.15134 (4)	0.03314 (17)
P2	0.34099 (3)	0.47707 (4)	0.26900 (4)	0.03378 (17)
O1	0.19529 (7)	0.58133 (11)	0.22875 (9)	0.0323 (3)
N1	0.25398 (9)	0.82334 (14)	0.26194 (12)	0.0335 (4)
N2	0.22386 (11)	0.92669 (15)	0.09521 (12)	0.0421 (5)
N3	0.32498 (9)	0.47476 (14)	0.15649 (12)	0.0341 (4)
N4	0.40281 (10)	0.37232 (15)	0.30858 (14)	0.0460 (5)
C1	0.14864 (10)	0.73710 (17)	0.13477 (13)	0.0299 (4)
C2	0.09075 (12)	0.78508 (19)	0.07518 (14)	0.0378 (5)
H2	0.0981	0.8535	0.0431	0.045*
C3	0.02307 (12)	0.7352 (2)	0.06197 (15)	0.0450 (6)
H3	−0.0154	0.7682	0.0199	0.054*
C4	0.01100 (12)	0.6380 (2)	0.10931 (15)	0.0431 (6)
H4	−0.0364	0.6061	0.1010	0.052*
C5	0.06652 (11)	0.58504 (18)	0.16925 (14)	0.0341 (5)
C6	0.05264 (12)	0.48505 (19)	0.22901 (17)	0.0436 (6)
C6′	0.02674 (16)	0.5366 (3)	0.3116 (2)	0.0691 (9)
H61	0.0167	0.4733	0.3506	0.104*
H62	−0.0175	0.5821	0.2918	0.104*
H63	0.0646	0.5872	0.3445	0.104*
C6"	−0.00587 (16)	0.4030 (3)	0.1799 (3)	0.0886 (11)
H64	0.0104	0.3692	0.1279	0.133*
H65	−0.0508	0.4467	0.1599	0.133*
H66	−0.0146	0.3406	0.2204	0.133*

C7	0.12274 (12)	0.41934 (17)	0.26105 (14)	0.0356 (5)
C8	0.12244 (14)	0.30780 (19)	0.29698 (17)	0.0491 (6)
H8	0.0776	0.2709	0.2989	0.059*
C9	0.18592 (16)	0.2506 (2)	0.32963 (18)	0.0571 (7)
H9	0.1847	0.1759	0.3559	0.069*
C10	0.25162 (14)	0.30120 (19)	0.32450 (16)	0.0464 (6)
H10	0.2952	0.2611	0.3483	0.056*
C11	0.25522 (11)	0.40994 (17)	0.28504 (13)	0.0331 (5)
C12	0.18949 (11)	0.46837 (16)	0.25811 (13)	0.0301 (5)
C13	0.13532 (10)	0.63374 (17)	0.17769 (13)	0.0292 (4)
C14	0.19883 (12)	0.85861 (18)	0.31332 (14)	0.0355 (5)
H14A	0.1507	0.8556	0.2735	0.043*
H14B	0.2080	0.9403	0.3327	0.043*
C15	0.19678 (14)	0.7844 (2)	0.39480 (15)	0.0494 (6)
H15A	0.1927	0.7023	0.3773	0.074*
H15B	0.1549	0.8065	0.4210	0.074*
H15C	0.2414	0.7963	0.4390	0.074*
C16	0.32828 (12)	0.8252 (2)	0.31135 (18)	0.0513 (6)
H16A	0.3602	0.7907	0.2735	0.062*
H16B	0.3315	0.7758	0.3653	0.062*
C17	0.35593 (16)	0.9459 (3)	0.3400 (2)	0.0730 (9)
H17A	0.3533	0.9954	0.2871	0.110*
H17B	0.4063	0.9408	0.3718	0.110*
H17C	0.3260	0.9796	0.3798	0.110*
C18	0.18840 (14)	1.02350 (18)	0.13247 (16)	0.0449 (6)
H18A	0.2254	1.0670	0.1749	0.054*
H18B	0.1531	0.9914	0.1668	0.054*
C19	0.14921 (17)	1.1079 (2)	0.06274 (19)	0.0656 (8)
H19A	0.1838	1.1414	0.0291	0.098*
H19B	0.1273	1.1702	0.0926	0.098*
H19C	0.1112	1.0664	0.0216	0.098*
C20	0.27251 (16)	0.9574 (2)	0.03463 (18)	0.0596 (7)
H20A	0.2441	0.9963	−0.0186	0.072*
H20B	0.2928	0.8850	0.0141	0.072*
C21	0.33337 (19)	1.0348 (3)	0.0748 (2)	0.0846 (10)
H21A	0.3140	1.1095	0.0906	0.127*
H21B	0.3652	1.0476	0.0316	0.127*
H21C	0.3610	0.9983	0.1288	0.127*
C22	0.28363 (13)	0.38731 (18)	0.09829 (15)	0.0408 (5)
H22A	0.2685	0.3252	0.1359	0.049*
H22B	0.3155	0.3516	0.0609	0.049*
C23	0.21694 (14)	0.4359 (2)	0.03769 (16)	0.0546 (7)
H23A	0.1860	0.4742	0.0740	0.082*
H23B	0.1901	0.3725	0.0034	0.082*
H23C	0.2317	0.4926	−0.0036	0.082*
C24	0.36406 (12)	0.55852 (19)	0.11121 (16)	0.0407 (5)
H24A	0.3758	0.6276	0.1501	0.049*
H24B	0.3319	0.5843	0.0554	0.049*

C25	0.43374 (14)	0.5114 (2)	0.0880 (2)	0.0588 (7)
H25A	0.4683	0.4941	0.1432	0.088*
H25B	0.4546	0.5695	0.0532	0.088*
H25C	0.4233	0.4401	0.0526	0.088*
C26	0.40659 (13)	0.2562 (2)	0.27059 (19)	0.0525 (7)
H26A	0.3605	0.2410	0.2286	0.063*
H26B	0.4105	0.1986	0.3196	0.063*
C27	0.46806 (16)	0.2363 (2)	0.2219 (2)	0.0665 (8)
H27A	0.4620	0.2868	0.1691	0.100*
H27B	0.4682	0.1548	0.2032	0.100*
H27C	0.5140	0.2544	0.2618	0.100*
C28	0.46724 (15)	0.4091 (2)	0.37006 (19)	0.0683 (9)
H28A	0.4594	0.4893	0.3900	0.082*
H28B	0.5080	0.4116	0.3373	0.082*
C29	0.4888 (3)	0.3344 (3)	0.4510 (2)	0.1249 (18)
H29A	0.4472	0.3240	0.4806	0.187*
H29B	0.5285	0.3717	0.4924	0.187*
H29C	0.5048	0.2583	0.4331	0.187*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0327 (3)	0.0226 (3)	0.0477 (4)	0.0019 (2)	0.0167 (3)	0.0019 (2)
P2	0.0341 (3)	0.0233 (3)	0.0428 (3)	0.0034 (2)	0.0045 (2)	−0.0032 (2)
O1	0.0293 (8)	0.0204 (7)	0.0463 (9)	−0.0018 (6)	0.0047 (6)	0.0051 (6)
N1	0.0250 (9)	0.0262 (9)	0.0489 (11)	0.0013 (7)	0.0062 (8)	0.0015 (8)
N2	0.0568 (12)	0.0292 (10)	0.0465 (11)	0.0027 (9)	0.0252 (9)	0.0080 (8)
N3	0.0348 (10)	0.0258 (9)	0.0436 (11)	−0.0028 (7)	0.0125 (8)	−0.0012 (7)
N4	0.0386 (11)	0.0278 (10)	0.0656 (13)	0.0065 (8)	−0.0051 (9)	−0.0067 (9)
C1	0.0307 (11)	0.0280 (10)	0.0331 (11)	0.0050 (8)	0.0114 (9)	−0.0011 (8)
C2	0.0434 (13)	0.0347 (12)	0.0358 (12)	0.0090 (10)	0.0087 (10)	0.0006 (9)
C3	0.0364 (13)	0.0523 (15)	0.0432 (13)	0.0134 (11)	−0.0007 (10)	−0.0033 (11)
C4	0.0260 (11)	0.0506 (14)	0.0525 (14)	0.0007 (10)	0.0070 (10)	−0.0117 (12)
C5	0.0289 (11)	0.0331 (11)	0.0418 (12)	−0.0006 (9)	0.0103 (9)	−0.0084 (9)
C6	0.0341 (12)	0.0368 (12)	0.0630 (16)	−0.0093 (10)	0.0174 (11)	−0.0005 (11)
C6′	0.0724 (19)	0.0618 (18)	0.090 (2)	0.0211 (15)	0.0569 (17)	0.0227 (15)
C6"	0.0591 (19)	0.0610 (19)	0.135 (3)	−0.0289 (16)	−0.0094 (19)	0.0131 (19)
C7	0.0428 (13)	0.0282 (11)	0.0401 (12)	−0.0048 (9)	0.0192 (10)	−0.0035 (9)
C8	0.0601 (16)	0.0325 (12)	0.0641 (16)	−0.0069 (11)	0.0362 (13)	0.0024 (11)
C9	0.080 (2)	0.0314 (13)	0.0695 (18)	0.0031 (13)	0.0380 (15)	0.0164 (12)
C10	0.0589 (16)	0.0330 (12)	0.0504 (14)	0.0099 (11)	0.0181 (12)	0.0119 (10)
C11	0.0433 (12)	0.0247 (10)	0.0328 (11)	0.0042 (9)	0.0113 (9)	−0.0006 (8)
C12	0.0416 (12)	0.0212 (10)	0.0297 (11)	−0.0012 (8)	0.0122 (9)	−0.0019 (8)
C13	0.0277 (11)	0.0273 (10)	0.0332 (11)	0.0032 (8)	0.0069 (8)	−0.0030 (8)
C14	0.0368 (12)	0.0313 (11)	0.0381 (12)	0.0019 (9)	0.0067 (9)	0.0003 (9)
C15	0.0621 (17)	0.0458 (14)	0.0399 (13)	−0.0022 (12)	0.0085 (12)	0.0050 (11)
C16	0.0311 (12)	0.0524 (15)	0.0677 (16)	−0.0042 (11)	0.0023 (11)	−0.0056 (13)
C17	0.0571 (18)	0.079 (2)	0.083 (2)	−0.0322 (16)	0.0133 (15)	−0.0303 (17)

C18	0.0589 (15)	0.0285 (12)	0.0488 (14)	0.0102 (10)	0.0141 (12)	0.0087 (10)
C19	0.088 (2)	0.0389 (14)	0.0653 (18)	0.0120 (14)	0.0036 (15)	0.0154 (13)
C20	0.082 (2)	0.0461 (14)	0.0577 (16)	−0.0171 (14)	0.0310 (15)	0.0000 (12)
C21	0.086 (2)	0.073 (2)	0.105 (3)	−0.0281 (18)	0.042 (2)	−0.0244 (19)
C22	0.0522 (14)	0.0318 (11)	0.0419 (13)	−0.0053 (10)	0.0183 (11)	−0.0057 (10)
C23	0.0701 (18)	0.0523 (15)	0.0380 (14)	−0.0116 (13)	0.0013 (12)	−0.0032 (11)
C24	0.0380 (12)	0.0297 (11)	0.0583 (15)	0.0004 (9)	0.0189 (11)	0.0027 (10)
C25	0.0481 (15)	0.0462 (14)	0.092 (2)	0.0016 (12)	0.0381 (15)	0.0040 (14)
C26	0.0425 (14)	0.0300 (12)	0.0789 (18)	0.0086 (10)	−0.0040 (12)	−0.0097 (12)
C27	0.072 (2)	0.0449 (15)	0.085 (2)	0.0168 (14)	0.0217 (16)	−0.0054 (14)
C28	0.0632 (18)	0.0544 (16)	0.0721 (19)	0.0192 (14)	−0.0260 (15)	−0.0176 (14)
C29	0.199 (5)	0.078 (2)	0.072 (2)	0.074 (3)	−0.039 (3)	−0.0197 (19)

Geometric parameters (Å, º)

P1—N1	1.6789 (19)	C14—H14B	0.9900
P1—N2	1.7038 (18)	C15—H15A	0.9800
P1—C1	1.840 (2)	C15—H15B	0.9800
P2—N3	1.6816 (18)	C15—H15C	0.9800
P2—N4	1.7027 (18)	C16—C17	1.517 (4)
P2—C11	1.845 (2)	C16—H16A	0.9900
O1—C13	1.381 (2)	C16—H16B	0.9900
O1—C12	1.384 (2)	C17—H17A	0.9800
N1—C16	1.456 (3)	C17—H17B	0.9800
N1—C14	1.469 (3)	C17—H17C	0.9800
N2—C20	1.459 (3)	C18—C19	1.521 (3)
N2—C18	1.465 (3)	C18—H18A	0.9900
N3—C24	1.460 (3)	C18—H18B	0.9900
N3—C22	1.463 (3)	C19—H19A	0.9800
N4—C28	1.447 (3)	C19—H19B	0.9800
N4—C26	1.462 (3)	C19—H19C	0.9800
C1—C2	1.392 (3)	C20—C21	1.487 (4)
C1—C13	1.401 (3)	C20—H20A	0.9900
C2—C3	1.375 (3)	C20—H20B	0.9900
C2—H2	0.9500	C21—H21A	0.9800
C3—C4	1.371 (3)	C21—H21B	0.9800
C3—H3	0.9500	C21—H21C	0.9800
C4—C5	1.390 (3)	C22—C23	1.516 (3)
C4—H4	0.9500	C22—H22A	0.9900
C5—C13	1.393 (3)	C22—H22B	0.9900
C5—C6	1.519 (3)	C23—H23A	0.9800
C6—C7	1.517 (3)	C23—H23B	0.9800
C6—C6"	1.532 (3)	C23—H23C	0.9800
C6—C6′	1.549 (4)	C24—C25	1.520 (3)
C6′—H61	0.9800	C24—H24A	0.9900
C6′—H62	0.9800	C24—H24B	0.9900
C6′—H63	0.9800	C25—H25A	0.9800
C6"—H64	0.9800	C25—H25B	0.9800

C6"—H65	0.9800	C25—H25C	0.9800
C6"—H66	0.9800	C26—C27	1.503 (4)
C7—C12	1.384 (3)	C26—H26A	0.9900
C7—C8	1.394 (3)	C26—H26B	0.9900
C8—C9	1.371 (4)	C27—H27A	0.9800
C8—H8	0.9500	C27—H27B	0.9800
C9—C10	1.381 (4)	C27—H27C	0.9800
C9—H9	0.9500	C28—C29	1.494 (4)
C10—C11	1.394 (3)	C28—H28A	0.9900
C10—H10	0.9500	C28—H28B	0.9900
C11—C12	1.398 (3)	C29—H29A	0.9800
C14—C15	1.512 (3)	C29—H29B	0.9800
C14—H14A	0.9900	C29—H29C	0.9800

N1—P1—N2	109.46 (9)	N1—C16—C17	113.9 (2)
N1—P1—C1	99.29 (9)	N1—C16—H16A	108.8
N2—P1—C1	100.52 (9)	C17—C16—H16A	108.8
N3—P2—N4	109.15 (9)	N1—C16—H16B	108.8
N3—P2—C11	97.77 (9)	C17—C16—H16B	108.8
N4—P2—C11	102.27 (9)	H16A—C16—H16B	107.7
C13—O1—C12	119.03 (15)	C16—C17—H17A	109.5
C16—N1—C14	115.21 (18)	C16—C17—H17B	109.5
C16—N1—P1	117.86 (15)	H17A—C17—H17B	109.5
C14—N1—P1	126.45 (14)	C16—C17—H17C	109.5
C20—N2—C18	116.49 (18)	H17A—C17—H17C	109.5
C20—N2—P1	116.57 (16)	H17B—C17—H17C	109.5
C18—N2—P1	121.22 (14)	N2—C18—C19	114.2 (2)
C24—N3—C22	115.13 (17)	N2—C18—H18A	108.7
C24—N3—P2	117.43 (14)	C19—C18—H18A	108.7
C22—N3—P2	126.86 (14)	N2—C18—H18B	108.7
C28—N4—C26	115.18 (18)	C19—C18—H18B	108.7
C28—N4—P2	117.00 (15)	H18A—C18—H18B	107.6
C26—N4—P2	125.94 (16)	C18—C19—H19A	109.5
C2—C1—C13	116.95 (19)	C18—C19—H19B	109.5
C2—C1—P1	122.44 (16)	H19A—C19—H19B	109.5
C13—C1—P1	120.46 (15)	C18—C19—H19C	109.5
C3—C2—C1	121.2 (2)	H19A—C19—H19C	109.5
C3—C2—H2	119.4	H19B—C19—H19C	109.5
C1—C2—H2	119.4	N2—C20—C21	114.2 (2)
C4—C3—C2	120.2 (2)	N2—C20—H20A	108.7
C4—C3—H3	119.9	C21—C20—H20A	108.7
C2—C3—H3	119.9	N2—C20—H20B	108.7
C3—C4—C5	121.6 (2)	C21—C20—H20B	108.7
C3—C4—H4	119.2	H20A—C20—H20B	107.6
C5—C4—H4	119.2	C20—C21—H21A	109.5
C4—C5—C13	117.0 (2)	C20—C21—H21B	109.5
C4—C5—C6	122.15 (19)	H21A—C21—H21B	109.5
C13—C5—C6	120.70 (19)	C20—C21—H21C	109.5

C5—C6—C7	109.42 (17)	H21A—C21—H21C	109.5
C5—C6—C6"	111.2 (2)	H21B—C21—H21C	109.5
C7—C6—C6"	110.5 (2)	N3—C22—C23	113.50 (18)
C5—C6—C6′	108.16 (19)	N3—C22—H22A	108.9
C7—C6—C6′	108.5 (2)	C23—C22—H22A	108.9
C6"—C6—C6′	109.0 (2)	N3—C22—H22B	108.9
C6—C6′—H61	109.5	C23—C22—H22B	108.9
C6—C6′—H62	109.5	H22A—C22—H22B	107.7
H61—C6′—H62	109.5	C22—C23—H23A	109.5
C6—C6′—H63	109.5	C22—C23—H23B	109.5
H61—C6′—H63	109.5	H23A—C23—H23B	109.5
H62—C6′—H63	109.5	C22—C23—H23C	109.5
C6—C6"—H64	109.5	H23A—C23—H23C	109.5
C6—C6"—H65	109.5	H23B—C23—H23C	109.5
H64—C6"—H65	109.5	N3—C24—C25	114.03 (18)
C6—C6"—H66	109.5	N3—C24—H24A	108.7
H64—C6"—H66	109.5	C25—C24—H24A	108.7
H65—C6"—H66	109.5	N3—C24—H24B	108.7
C12—C7—C8	117.3 (2)	C25—C24—H24B	108.7
C12—C7—C6	121.64 (18)	H24A—C24—H24B	107.6
C8—C7—C6	121.09 (19)	C24—C25—H25A	109.5
C9—C8—C7	121.0 (2)	C24—C25—H25B	109.5
C9—C8—H8	119.5	H25A—C25—H25B	109.5
C7—C8—H8	119.5	C24—C25—H25C	109.5
C8—C9—C10	120.3 (2)	H25A—C25—H25C	109.5
C8—C9—H9	119.9	H25B—C25—H25C	109.5
C10—C9—H9	119.9	N4—C26—C27	115.7 (2)
C9—C10—C11	121.2 (2)	N4—C26—H26A	108.4
C9—C10—H10	119.4	C27—C26—H26A	108.4
C11—C10—H10	119.4	N4—C26—H26B	108.4
C10—C11—C12	116.43 (19)	C27—C26—H26B	108.4
C10—C11—P2	123.15 (17)	H26A—C26—H26B	107.4
C12—C11—P2	120.41 (15)	C26—C27—H27A	109.5
C7—C12—O1	121.31 (18)	C26—C27—H27B	109.5
C7—C12—C11	123.46 (18)	H27A—C27—H27B	109.5
O1—C12—C11	115.23 (17)	C26—C27—H27C	109.5
O1—C13—C5	121.45 (18)	H27A—C27—H27C	109.5
O1—C13—C1	115.70 (17)	H27B—C27—H27C	109.5
C5—C13—C1	122.85 (18)	N4—C28—C29	115.4 (3)
N1—C14—C15	114.25 (18)	N4—C28—H28A	108.4
N1—C14—H14A	108.7	C29—C28—H28A	108.4
C15—C14—H14A	108.7	N4—C28—H28B	108.4
N1—C14—H14B	108.7	C29—C28—H28B	108.4
C15—C14—H14B	108.7	H28A—C28—H28B	107.5
H14A—C14—H14B	107.6	C28—C29—H29A	109.5
C14—C15—H15A	109.5	C28—C29—H29B	109.5
C14—C15—H15B	109.5	H29A—C29—H29B	109.5
H15A—C15—H15B	109.5	C28—C29—H29C	109.5

C14—C15—H15C	109.5	H29A—C29—H29C	109.5
H15A—C15—H15C	109.5	H29B—C29—H29C	109.5
H15B—C15—H15C	109.5		
N2—P1—N1—C16	−101.02 (17)	C9—C10—C11—C12	5.2 (3)
C1—P1—N1—C16	154.27 (16)	C9—C10—C11—P2	−175.46 (19)
N2—P1—N1—C14	70.55 (18)	N3—P2—C11—C10	113.93 (19)
C1—P1—N1—C14	−34.16 (17)	N4—P2—C11—C10	2.3 (2)
N1—P1—N2—C20	122.28 (17)	N3—P2—C11—C12	−66.72 (17)
C1—P1—N2—C20	−133.86 (18)	N4—P2—C11—C12	−178.34 (16)
N1—P1—N2—C18	−30.1 (2)	C8—C7—C12—O1	−175.96 (18)
C1—P1—N2—C18	73.8 (2)	C6—C7—C12—O1	2.4 (3)
N4—P2—N3—C24	−97.65 (16)	C8—C7—C12—C11	3.2 (3)
C11—P2—N3—C24	156.43 (15)	C6—C7—C12—C11	−178.5 (2)
N4—P2—N3—C22	73.12 (19)	C13—O1—C12—C7	−19.3 (3)
C11—P2—N3—C22	−32.80 (18)	C13—O1—C12—C11	161.51 (17)
N3—P2—N4—C28	124.9 (2)	C10—C11—C12—C7	−6.3 (3)
C11—P2—N4—C28	−132.3 (2)	P2—C11—C12—C7	174.29 (16)
N3—P2—N4—C26	−38.7 (2)	C10—C11—C12—O1	172.85 (18)
C11—P2—N4—C26	64.1 (2)	P2—C11—C12—O1	−6.5 (2)
N1—P1—C1—C2	122.63 (17)	C12—O1—C13—C5	12.3 (3)
N2—P1—C1—C2	10.69 (19)	C12—O1—C13—C1	−166.96 (16)
N1—P1—C1—C13	−62.09 (17)	C4—C5—C13—O1	−173.69 (18)
N2—P1—C1—C13	−174.03 (16)	C6—C5—C13—O1	11.3 (3)
C13—C1—C2—C3	2.4 (3)	C4—C5—C13—C1	5.5 (3)
P1—C1—C2—C3	177.83 (16)	C6—C5—C13—C1	−169.50 (19)
C1—C2—C3—C4	1.6 (3)	C2—C1—C13—O1	173.18 (17)
C2—C3—C4—C5	−2.2 (3)	P1—C1—C13—O1	−2.4 (2)
C3—C4—C5—C13	−1.3 (3)	C2—C1—C13—C5	−6.1 (3)
C3—C4—C5—C6	173.6 (2)	P1—C1—C13—C5	178.41 (15)
C4—C5—C6—C7	160.1 (2)	C16—N1—C14—C15	−59.4 (2)
C13—C5—C6—C7	−25.1 (3)	P1—N1—C14—C15	128.82 (18)
C4—C5—C6—C6"	37.7 (3)	C14—N1—C16—C17	−65.7 (3)
C13—C5—C6—C6"	−147.5 (2)	P1—N1—C16—C17	106.8 (2)
C4—C5—C6—C6′	−81.9 (3)	C20—N2—C18—C19	52.3 (3)
C13—C5—C6—C6′	92.9 (2)	P1—N2—C18—C19	−155.28 (19)
C5—C6—C7—C12	18.5 (3)	C18—N2—C20—C21	58.6 (3)
C6"—C6—C7—C12	141.4 (2)	P1—N2—C20—C21	−95.2 (3)
C6′—C6—C7—C12	−99.3 (2)	C24—N3—C22—C23	−71.2 (2)
C5—C6—C7—C8	−163.2 (2)	P2—N3—C22—C23	117.85 (19)
C6"—C6—C7—C8	−40.4 (3)	C22—N3—C24—C25	−77.7 (3)
C6′—C6—C7—C8	79.0 (3)	P2—N3—C24—C25	94.1 (2)
C12—C7—C8—C9	1.3 (3)	C28—N4—C26—C27	−56.6 (3)
C6—C7—C8—C9	−177.0 (2)	P2—N4—C26—C27	107.2 (2)
C7—C8—C9—C10	−2.4 (4)	C26—N4—C28—C29	−60.1 (4)
C8—C9—C10—C11	−1.1 (4)	P2—N4—C28—C29	134.5 (2)