Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1,1′-Binaphthalene-2,2′-diyl hydrogen phosphate

Susanta K. Nayak,[a] S. Chandrasekhar[b] and T. N. Guru Row[a]*

[a]Solid State and Structural Chemistry Unit, Indian Institute of Science, Bangalore 560 012, Karnataka, India, and [b]Department of Organic Chemistry, Indian Institute of Science, Bangalore 560 012, India
Correspondence e-mail: ssctng@sscu.iisc.ernet.in

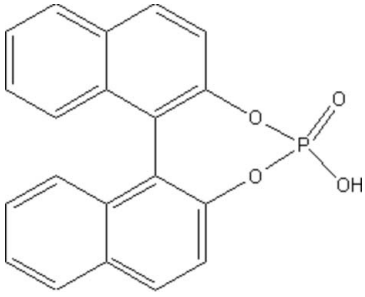
Received 22 November 2007; accepted 29 November 2007

Key indicators: single-crystal X-ray study; T = 292 K; mean σ(C–C) = 0.005 Å; R factor = 0.068; wR factor = 0.160; data-to-parameter ratio = 13.6.

The title compound, racemic bnppa, $C_{20}H_{13}O_4P$, crystallizes with four molecules in the asymmetric unit. Two independent centrosymmetric O—H···O hydrogen-bonded tetramers, each involving two of the symmetry-independent molecules in the asymmetric unit generate the packing motif in the crystal structure. The hydrophobic parts of the tetramers connect to their neighbours *via* C—H···π and π–π interactions [3.899 (2), 3.895 (2) and 3.803 (2) Å within the symmetric unit, 3.851 (2), 4.000 (2), 3.988 (2) and 3.905 (2) Å across centres of symmetry].

Related literature

For details on bnppa as an optical resolution agent of amines, see: Fujii & Hirayamma (2002); Arnold *et al.* (1983); Wilen *et al.* (1991). For the conglomerate nature of racemates, see: Jacques *et al.* (1981). For related literature, see: An *et al.* (1996); Bernstein *et al.* (1995); Etter & Baures (1988).



Experimental

Crystal data

$C_{20}H_{13}O_4P$
M_r = 348.27
Triclinic, $P\overline{1}$
a = 14.2152 (14) Å
b = 14.6480 (14) Å
c = 15.6397 (15) Å
α = 87.710 (2)°
β = 88.074 (2)°
γ = 83.952 (2)°
V = 3234.4 (5) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.19 mm^{-1}
T = 292 (2) K
0.31 × 0.07 × 0.06 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.939, T_{max} = 0.989
32848 measured reflections
12219 independent reflections
7419 reflections with $I > 2\sigma(I)$
R_{int} = 0.053

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.160
S = 1.06
12219 reflections
901 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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

*Cg*1 and *Cg*2 are the centroids of the C64–C69 and C12–C17 rings.

D—H···A	D—H	H···A	D···A	D—H···A
O4—H1O···O7	0.82	1.78	2.475 (4)	141
O8—H2O···O3^{i}	0.82	1.77	2.450 (4)	139
O16—H4O···O12^{ii}	0.82	1.99	2.441 (4)	114
O11—H30···O15	0.82	1.71	2.477 (4)	154
C22—H22···O11^{iii}	0.93	2.51	3.435 (4)	174
C59—H59···O7^{iv}	0.93	2.56	3.478 (4)	167
C27—H27···*Cg*1	0.93	2.75	3.567 (5)	147
C54—H54···*Cg*2^{v}	0.93	2.77	3.535 (5)	140

Symmetry codes: (i) $-x, -y+1, -z$; (ii) $-x+2, -y+1, -z+1$; (iii) $x-1, y, z$; (iv) $x+1, y, z$.

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *WinGX* (Farrugia, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1999) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *PLATON* (Spek, 2003).

The authors thank DST-IRHPA for the CCD X-ray facility at IISc and CSIR (SRF), India, for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LN2013).

References

An, J., Wilson, J. M., An, Y. Z. & Wiemer, D. W. (1996). *J. Org. Chem.* **61**, 4040–4045.
Arnold, W., Daly, J. J., Imhof, R. & Kyburz, E. (1983). *Tetrahedron Lett.* **24**, 343–346.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bruker (2004). *SMART* (Version 5.628) and *SAINT* (Version 6.45a). Bruker AXS Inc., Madison, Wisconsin, USA.
Etter, M. C. & Baures, P. W. (1988). *J. Am. Chem. Soc.* **110**, 639–640.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Fujii, I. & Hirayamma, N. (2002). *Helv. Chim. Acta*, **85**, 2946–2960.
Jacques, J., Collet, A. & Wilen, S. H. (1981). *Enantiomers, Racemates and Resolutions*, chs. 1–4. New York: John Wiley & Sons.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Watkin, D. M., Pearce, L. & Prout, C. K. (1993). *CAMERON*. Chemical Crystallography Laboratory, University of Oxford, England.
Wilen, S. H., Qi, J. Z. & Williard, P. G. (1991). *J. Org. Chem.* **56**, 485–487.

supporting information

Acta Cryst. (2008). E**64**, o256 [https://doi.org/10.1107/S1600536807064343]

1,1′-Binaphthalene-2,2′-diyl hydrogen phosphate

Susanta K. Nayak, S. Chandrasekhar and T. N. Guru Row

S1. Comment

We have been investigating the conglomerate nature of racemates in our laboratory, as very few structures show the features of conglomerates (Jacques *et al.*, 1981). Optically active 1,1′-binaphthalene-2,2′-diyl hydrogen phosphate (bnppa) has been used as a good optical resolution agent for amines (Fujii *et al.*, 2002; Arnold *et al.*, 1983; Wilen *et al.*, 1991). However, the procedure adopted in the preparation yields only racemic bnppa crystals (Scheme).

The compound crystallizes in a triclinic space group *P*-1, with *Z* = 8. Of the four molecules in the asymmetric unit (Fig. 1), pairs are connected through intermolecular O—H···O hydrogen bonds (Table 1) and two such motifs form independent centrosymmetric tetramers (Fig. 2) though additional O—H···O hydrogen bonds. The packing in the crystal structure is hence through tetrameric O—H···O hydrogen bonded systems [Etter's graph set symbol $R_4^4(16)$] (Bernstein *et al.*, 1995). Further, the hydrophobic parts of the symmetry-independent parts of the tetramers are linked *via* C—H···π interactions [C27—H27···π (C64/C65/C66/C67/C68/C69) = 2.75 Å, C54—H54···π (C12/C13/C14/C14/C15/C16/C17 = 2.77 Å)]. There are several π···π interactions within the symmetric unit [3.899 (2), 3.895 (2) and 3.803 (2) Å], as well as others across centres of symmetry [3.851 (2), 4.000 (2), 3.988 (2) and 3.905 (2) Å]. Table 1 lists all O—H···0 hydrogen bonds along with two C—H..O interactions which form as a consequence of tetramer formation.

S2. Experimental

Racemic binaphthylphosphoylchloride (29.2 g, 80 mmol) prepared by a known procedure (An *et al.*, 1996) was dissolved in 1.4 l. of 2% Na_2CO_3 solution and heated until a clear solution formed. The solution, on keeping at 273 K for 12 h, yields a grey precipitate. The precipitate was collected by filtration, washed with 100 ml of 2% Na_2CO_3 solution and suspended in 650 ml of water. 50 ml of conc. HCl was then added to this while stirring. The mixture was heated to 368 K for 5 minutes and was kept at 277 K for 14 h. The resulting white crystalline mass was separated by filtration followed by washing with cold water and dried to obtain bnppa in 59.2% yield (m.p. 621 K). Plate-like colourless crystals of bnppa were grown in acetonitrile and water (2:1 *v*/*v*) with triphenyphosphine oxide as an additive promotor in the ratio 50:50 (Etter *et al.*, 1988) by slow evaporation at room temperature.

S3. Refinement

All H atoms were positioned geometrically, (C—H = 0.93 Å, O—H = 0.82 Å) and refined using a riding model with U_{iso}(H)= 1.2 U_{eq}(C) or 1.5 U_{eq}(O).

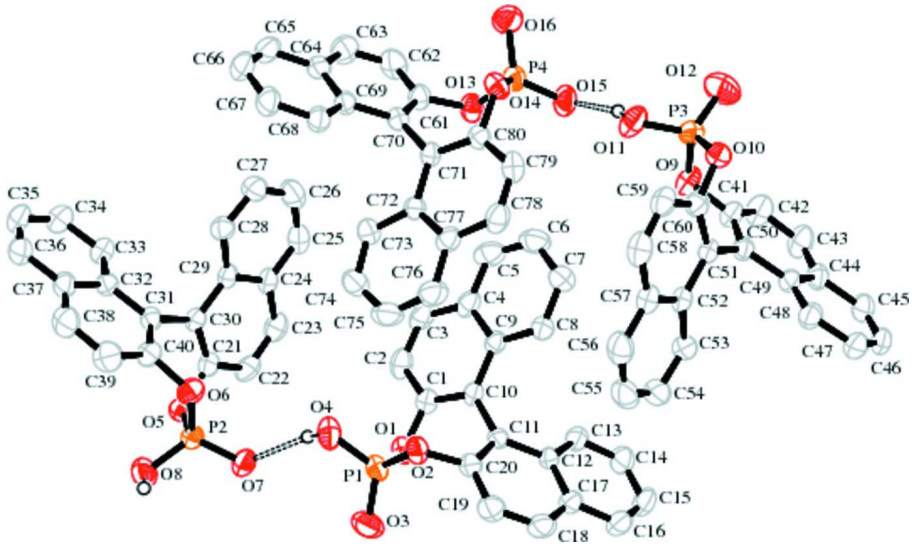


Figure 1

The four molecules in the asymmetric unit with O—H···O hydrogen bonds and 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

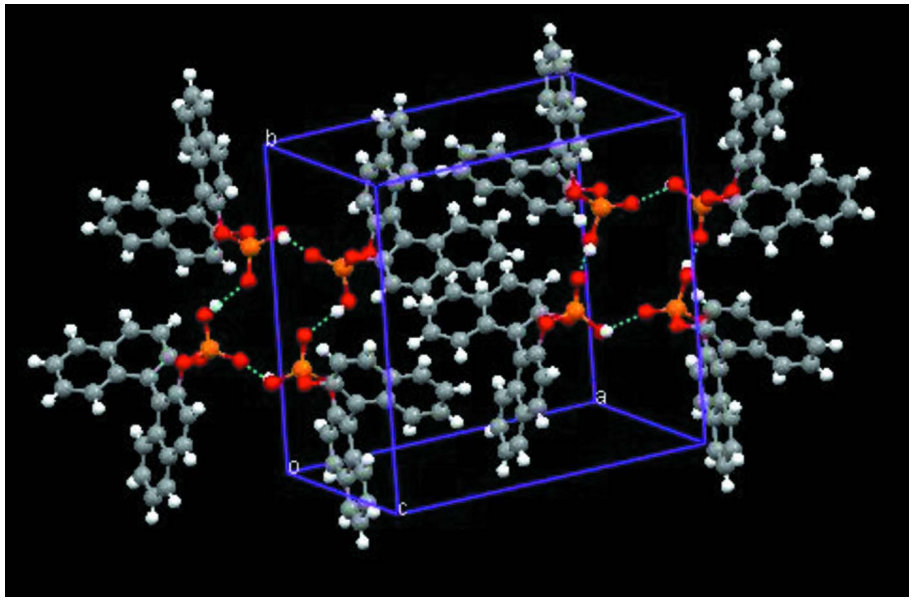


Figure 2

The molecular packing showing the O—H···O hydrogen bonds which create tetramers with the $R_4^4(16)$ motif.

1,1′-Binaphthalene-2,2′-diyl hydrogen phosphate

Crystal data

$C_{20}H_{13}O_4P$
$M_r = 348.27$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 14.2152\ (14)$ Å
$b = 14.6480\ (14)$ Å
$c = 15.6397\ (15)$ Å
$\alpha = 87.710\ (2)°$
$\beta = 88.074\ (2)°$
$\gamma = 83.952\ (2)°$
$V = 3234.4\ (5)$ Å^3
$Z = 8$

$F(000) = 1440$
$D_x = 1.430$ Mg m^{-3}
Melting point: 348 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 640 reflections
$\theta = 0.9$–$28.0°$
$\mu = 0.19$ mm^{-1}
$T = 292$ K
Needle, colourless
0.31 × 0.07 × 0.06 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.939$, $T_{max} = 0.989$
32848 measured reflections
12219 independent reflections
7419 reflections with $I > 2\sigma(I)$
$R_{int} = 0.053$
$\theta_{max} = 25.7°$, $\theta_{min} = 1.4°$
$h = -17 \rightarrow 17$
$k = -17 \rightarrow 17$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.068$
$wR(F^2) = 0.160$
$S = 1.06$
12219 reflections
901 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
$w = 1/[\sigma^2(F_o^2) + (0.0616P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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	0.18879 (7)	0.59609 (7)	0.09812 (7)	0.0433 (3)
P2	0.02314 (7)	0.32601 (7)	0.13354 (7)	0.0414 (3)
P3	0.94345 (8)	0.64658 (7)	0.34851 (7)	0.0479 (3)
P4	0.80353 (7)	0.37386 (7)	0.44392 (7)	0.0434 (3)
O1	0.18978 (16)	0.63722 (16)	0.18932 (15)	0.0429 (6)
O2	0.28908 (16)	0.61002 (16)	0.05549 (14)	0.0413 (6)
O3	0.10800 (18)	0.6437 (2)	0.05428 (18)	0.0664 (8)
O4	0.19492 (18)	0.49120 (17)	0.10530 (17)	0.0569 (8)
H1O	0.1483	0.4756	0.1320	0.085*
O5	−0.00382 (16)	0.30050 (15)	0.22946 (15)	0.0414 (6)
O6	0.11649 (17)	0.26132 (16)	0.11331 (15)	0.0428 (6)

O7	0.04349 (17)	0.42177 (16)	0.11774 (17)	0.0534 (7)
O8	−0.06052 (18)	0.29605 (18)	0.08799 (17)	0.0593 (8)
H2O	−0.0515	0.3017	0.0361	0.089*
O9	0.85273 (18)	0.71325 (15)	0.37247 (15)	0.0456 (7)
O10	0.97501 (16)	0.67963 (16)	0.25610 (16)	0.0458 (7)
O11	0.90470 (19)	0.55429 (16)	0.33839 (17)	0.0594 (8)
H30	0.8854	0.5353	0.3851	0.089*
O12	1.0212 (2)	0.6476 (2)	0.4053 (2)	0.0905 (11)
O13	0.69748 (17)	0.36199 (16)	0.47107 (15)	0.0442 (6)
O14	0.81529 (16)	0.33779 (16)	0.35017 (15)	0.0419 (6)
O15	0.81746 (19)	0.47098 (17)	0.45115 (16)	0.0535 (7)
O16	0.8698 (2)	0.30309 (18)	0.49275 (18)	0.0668 (9)
H4O	0.8642	0.3122	0.5441	0.100*
C1	0.2734 (2)	0.6182 (2)	0.2365 (2)	0.0357 (8)
C2	0.2703 (3)	0.5561 (3)	0.3067 (2)	0.0477 (10)
H2	0.2151	0.5297	0.3215	0.057*
C3	0.3490 (3)	0.5352 (3)	0.3527 (2)	0.0501 (11)
H3	0.3473	0.4946	0.3998	0.060*
C4	0.4334 (3)	0.5738 (2)	0.3308 (2)	0.0431 (9)
C5	0.5177 (3)	0.5497 (3)	0.3757 (2)	0.0558 (11)
H5	0.5170	0.5075	0.4218	0.067*
C6	0.5992 (3)	0.5859 (3)	0.3540 (3)	0.0594 (12)
H6	0.6534	0.5691	0.3850	0.071*
C7	0.6010 (3)	0.6487 (3)	0.2846 (3)	0.0564 (11)
H7	0.6569	0.6739	0.2693	0.068*
C8	0.5217 (3)	0.6737 (2)	0.2388 (2)	0.0433 (9)
H8	0.5247	0.7152	0.1925	0.052*
C9	0.4355 (2)	0.6380 (2)	0.2602 (2)	0.0339 (8)
C10	0.3506 (2)	0.6617 (2)	0.2128 (2)	0.0327 (8)
C11	0.3453 (2)	0.7269 (2)	0.1368 (2)	0.0319 (8)
C12	0.3617 (2)	0.8221 (2)	0.1423 (2)	0.0333 (8)
C13	0.3790 (2)	0.8617 (2)	0.2200 (2)	0.0408 (9)
H13	0.3837	0.8250	0.2699	0.049*
C14	0.3892 (3)	0.9527 (3)	0.2233 (3)	0.0516 (11)
H14	0.4005	0.9774	0.2755	0.062*
C15	0.3829 (3)	1.0098 (3)	0.1496 (3)	0.0590 (12)
H15	0.3909	1.0718	0.1526	0.071*
C16	0.3652 (3)	0.9746 (3)	0.0740 (3)	0.0546 (11)
H16	0.3604	1.0131	0.0252	0.066*
C17	0.3538 (2)	0.8803 (2)	0.0677 (2)	0.0414 (9)
C18	0.3306 (3)	0.8441 (3)	−0.0102 (3)	0.0518 (11)
H18	0.3286	0.8815	−0.0598	0.062*
C19	0.3109 (3)	0.7557 (3)	−0.0140 (2)	0.0476 (10)
H19	0.2940	0.7328	−0.0653	0.057*
C20	0.3166 (2)	0.6998 (2)	0.0604 (2)	0.0376 (9)
C21	0.0697 (2)	0.2935 (2)	0.2883 (2)	0.0355 (8)
C22	0.0694 (3)	0.3652 (2)	0.3445 (2)	0.0471 (10)
H22	0.0213	0.4133	0.3433	0.056*

C23	0.1402 (3)	0.3640 (2)	0.4009 (2)	0.0472 (10)
H23	0.1391	0.4103	0.4400	0.057*
C24	0.2155 (3)	0.2926 (2)	0.4005 (2)	0.0378 (9)
C25	0.2931 (3)	0.2927 (3)	0.4544 (2)	0.0496 (10)
H25	0.2934	0.3391	0.4933	0.059*
C26	0.3673 (3)	0.2266 (3)	0.4507 (3)	0.0574 (12)
H26	0.4177	0.2278	0.4869	0.069*
C27	0.3677 (3)	0.1566 (3)	0.3926 (3)	0.0522 (11)
H27	0.4194	0.1122	0.3892	0.063*
C28	0.2937 (2)	0.1526 (2)	0.3409 (2)	0.0412 (9)
H28	0.2951	0.1049	0.3033	0.049*
C29	0.2143 (2)	0.2193 (2)	0.3429 (2)	0.0347 (8)
C30	0.1362 (2)	0.2186 (2)	0.2876 (2)	0.0338 (8)
C31	0.1291 (2)	0.1453 (2)	0.2256 (2)	0.0343 (8)
C32	0.1270 (2)	0.0499 (2)	0.2507 (2)	0.0379 (9)
C33	0.1268 (3)	0.0182 (2)	0.3370 (3)	0.0486 (10)
H33	0.1286	0.0599	0.3801	0.058*
C34	0.1239 (3)	−0.0734 (3)	0.3582 (3)	0.0594 (12)
H34	0.1242	−0.0930	0.4155	0.071*
C35	0.1205 (3)	−0.1375 (3)	0.2948 (3)	0.0640 (13)
H35	0.1198	−0.1995	0.3098	0.077*
C36	0.1184 (3)	−0.1091 (3)	0.2121 (3)	0.0577 (12)
H36	0.1148	−0.1518	0.1702	0.069*
C37	0.1214 (3)	−0.0157 (2)	0.1874 (3)	0.0440 (10)
C38	0.1157 (3)	0.0147 (3)	0.1011 (3)	0.0561 (11)
H38	0.1127	−0.0282	0.0592	0.067*
C39	0.1143 (3)	0.1048 (3)	0.0774 (3)	0.0552 (11)
H39	0.1094	0.1241	0.0202	0.066*
C40	0.1203 (2)	0.1680 (2)	0.1408 (2)	0.0397 (9)
C41	0.8530 (3)	0.8082 (2)	0.3531 (2)	0.0399 (9)
C42	0.8586 (3)	0.8648 (3)	0.4221 (2)	0.0539 (11)
H42	0.8618	0.8403	0.4779	0.065*
C43	0.8590 (3)	0.9562 (3)	0.4057 (3)	0.0548 (11)
H43	0.8605	0.9950	0.4512	0.066*
C44	0.8573 (2)	0.9938 (2)	0.3217 (2)	0.0408 (9)
C45	0.8619 (3)	1.0890 (3)	0.3046 (3)	0.0493 (10)
H45	0.8640	1.1278	0.3501	0.059*
C46	0.8633 (3)	1.1242 (3)	0.2244 (3)	0.0510 (11)
H46	0.8651	1.1871	0.2146	0.061*
C47	0.8620 (3)	1.0669 (3)	0.1551 (3)	0.0489 (10)
H47	0.8649	1.0916	0.0995	0.059*
C48	0.8566 (2)	0.9748 (2)	0.1687 (2)	0.0430 (9)
H48	0.8554	0.9376	0.1220	0.052*
C49	0.8529 (2)	0.9352 (2)	0.2520 (2)	0.0353 (8)
C50	0.8474 (2)	0.8389 (2)	0.2694 (2)	0.0339 (8)
C51	0.8399 (2)	0.7725 (2)	0.2006 (2)	0.0310 (8)
C52	0.7632 (2)	0.7803 (2)	0.1420 (2)	0.0351 (8)
C53	0.6861 (2)	0.8493 (2)	0.1478 (2)	0.0425 (9)

H53	0.6854	0.8930	0.1895	0.051*
C54	0.6131 (3)	0.8530 (3)	0.0937 (3)	0.0524 (11)
H54	0.5627	0.8984	0.0995	0.063*
C55	0.6125 (3)	0.7900 (3)	0.0294 (3)	0.0603 (12)
H55	0.5628	0.7945	−0.0082	0.072*
C56	0.6846 (3)	0.7221 (3)	0.0220 (3)	0.0565 (11)
H56	0.6837	0.6800	−0.0208	0.068*
C57	0.7612 (3)	0.7142 (2)	0.0785 (2)	0.0394 (9)
C58	0.8328 (3)	0.6398 (3)	0.0756 (2)	0.0476 (10)
H58	0.8324	0.5972	0.0331	0.057*
C59	0.9018 (3)	0.6292 (2)	0.1337 (2)	0.0444 (10)
H59	0.9474	0.5788	0.1325	0.053*
C60	0.9032 (2)	0.6951 (2)	0.1953 (2)	0.0371 (9)
C61	0.6665 (2)	0.2751 (2)	0.4561 (2)	0.0394 (9)
C62	0.6601 (3)	0.2145 (3)	0.5262 (2)	0.0548 (11)
H62	0.6711	0.2326	0.5810	0.066*
C63	0.6376 (3)	0.1284 (3)	0.5134 (3)	0.0579 (12)
H63	0.6330	0.0873	0.5599	0.070*
C64	0.6212 (2)	0.1006 (3)	0.4305 (2)	0.0425 (9)
C65	0.6033 (3)	0.0090 (3)	0.4153 (3)	0.0538 (11)
H65	0.6003	−0.0331	0.4611	0.065*
C66	0.5908 (3)	−0.0174 (3)	0.3355 (3)	0.0564 (11)
H66	0.5789	−0.0774	0.3265	0.068*
C67	0.5955 (3)	0.0449 (3)	0.2662 (3)	0.0498 (10)
H67	0.5867	0.0259	0.2113	0.060*
C68	0.6127 (2)	0.1327 (2)	0.2776 (2)	0.0401 (9)
H68	0.6162	0.1728	0.2303	0.048*
C69	0.6254 (2)	0.1641 (2)	0.3600 (2)	0.0349 (8)
C70	0.6473 (2)	0.2556 (2)	0.3742 (2)	0.0342 (8)
C71	0.6587 (2)	0.3251 (2)	0.3030 (2)	0.0317 (8)
C72	0.5837 (2)	0.3566 (2)	0.2458 (2)	0.0329 (8)
C73	0.4919 (2)	0.3289 (2)	0.2554 (2)	0.0428 (9)
H73	0.4783	0.2878	0.2998	0.051*
C74	0.4225 (3)	0.3615 (3)	0.2006 (3)	0.0566 (11)
H74	0.3622	0.3426	0.2080	0.068*
C75	0.4415 (3)	0.4232 (3)	0.1334 (3)	0.0613 (12)
H75	0.3940	0.4448	0.0959	0.074*
C76	0.5286 (3)	0.4517 (3)	0.1227 (3)	0.0582 (12)
H76	0.5402	0.4928	0.0776	0.070*
C77	0.6025 (3)	0.4205 (2)	0.1783 (2)	0.0407 (9)
C78	0.6919 (3)	0.4531 (3)	0.1695 (2)	0.0484 (10)
H78	0.7040	0.4940	0.1244	0.058*
C79	0.7611 (3)	0.4258 (2)	0.2260 (2)	0.0461 (10)
H79	0.8199	0.4482	0.2203	0.055*
C80	0.7419 (2)	0.3634 (2)	0.2928 (2)	0.0337 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0388 (6)	0.0449 (6)	0.0489 (6)	−0.0139 (5)	−0.0092 (5)	−0.0012 (5)
P2	0.0382 (6)	0.0351 (6)	0.0511 (7)	−0.0077 (5)	−0.0049 (5)	0.0076 (5)
P3	0.0505 (7)	0.0388 (6)	0.0553 (7)	−0.0066 (5)	−0.0177 (5)	0.0053 (5)
P4	0.0491 (7)	0.0386 (6)	0.0451 (6)	−0.0120 (5)	−0.0143 (5)	−0.0029 (5)
O1	0.0361 (14)	0.0458 (16)	0.0484 (16)	−0.0107 (12)	0.0010 (12)	−0.0065 (13)
O2	0.0435 (15)	0.0427 (15)	0.0398 (15)	−0.0125 (12)	−0.0025 (12)	−0.0048 (12)
O3	0.0464 (17)	0.084 (2)	0.069 (2)	−0.0095 (16)	−0.0190 (15)	0.0150 (17)
O4	0.0556 (18)	0.0504 (17)	0.0688 (19)	−0.0218 (14)	−0.0028 (14)	−0.0082 (15)
O5	0.0351 (14)	0.0406 (15)	0.0477 (16)	−0.0020 (12)	−0.0011 (12)	0.0017 (12)
O6	0.0469 (16)	0.0410 (15)	0.0397 (15)	−0.0039 (12)	0.0039 (12)	0.0036 (12)
O7	0.0502 (17)	0.0348 (15)	0.076 (2)	−0.0103 (12)	−0.0049 (14)	0.0083 (14)
O8	0.0580 (18)	0.0645 (19)	0.0598 (18)	−0.0282 (15)	−0.0136 (14)	0.0119 (15)
O9	0.0632 (18)	0.0291 (14)	0.0445 (15)	−0.0081 (13)	0.0040 (13)	0.0032 (12)
O10	0.0352 (14)	0.0408 (15)	0.0611 (18)	−0.0031 (12)	−0.0039 (13)	0.0034 (13)
O11	0.080 (2)	0.0325 (15)	0.0662 (19)	−0.0093 (14)	−0.0035 (16)	0.0024 (14)
O12	0.080 (2)	0.105 (3)	0.090 (3)	−0.016 (2)	−0.046 (2)	0.009 (2)
O13	0.0535 (17)	0.0446 (16)	0.0374 (15)	−0.0148 (13)	−0.0043 (12)	−0.0084 (12)
O14	0.0359 (14)	0.0492 (16)	0.0422 (15)	−0.0085 (12)	−0.0056 (12)	−0.0077 (12)
O15	0.0688 (19)	0.0420 (16)	0.0540 (17)	−0.0209 (14)	−0.0117 (14)	−0.0085 (13)
O16	0.081 (2)	0.0541 (18)	0.067 (2)	−0.0076 (16)	−0.0331 (17)	0.0049 (15)
C1	0.041 (2)	0.035 (2)	0.031 (2)	−0.0079 (17)	0.0027 (17)	−0.0035 (16)
C2	0.060 (3)	0.046 (2)	0.040 (2)	−0.022 (2)	0.010 (2)	−0.0007 (19)
C3	0.079 (3)	0.040 (2)	0.032 (2)	−0.013 (2)	−0.003 (2)	0.0105 (18)
C4	0.063 (3)	0.032 (2)	0.036 (2)	−0.0077 (19)	−0.0034 (19)	−0.0024 (17)
C5	0.075 (3)	0.053 (3)	0.038 (2)	0.004 (2)	−0.016 (2)	0.001 (2)
C6	0.064 (3)	0.058 (3)	0.056 (3)	0.006 (2)	−0.025 (2)	−0.005 (2)
C7	0.049 (3)	0.051 (3)	0.071 (3)	−0.006 (2)	−0.018 (2)	−0.002 (2)
C8	0.045 (2)	0.039 (2)	0.047 (2)	−0.0109 (18)	−0.0099 (19)	0.0059 (18)
C9	0.044 (2)	0.0285 (19)	0.030 (2)	−0.0070 (16)	−0.0034 (16)	−0.0016 (16)
C10	0.041 (2)	0.0271 (19)	0.031 (2)	−0.0087 (16)	0.0002 (16)	−0.0019 (15)
C11	0.0279 (19)	0.036 (2)	0.032 (2)	−0.0075 (15)	−0.0043 (15)	0.0060 (16)
C12	0.0274 (19)	0.030 (2)	0.042 (2)	−0.0055 (15)	−0.0010 (16)	0.0040 (17)
C13	0.046 (2)	0.034 (2)	0.043 (2)	−0.0069 (18)	−0.0033 (18)	−0.0003 (18)
C14	0.052 (3)	0.037 (2)	0.067 (3)	−0.0048 (19)	−0.006 (2)	−0.010 (2)
C15	0.061 (3)	0.027 (2)	0.089 (4)	−0.007 (2)	−0.004 (3)	0.004 (2)
C16	0.051 (3)	0.042 (3)	0.069 (3)	−0.005 (2)	−0.003 (2)	0.020 (2)
C17	0.037 (2)	0.038 (2)	0.048 (2)	−0.0051 (17)	−0.0030 (18)	0.0096 (19)
C18	0.051 (3)	0.057 (3)	0.046 (3)	−0.007 (2)	−0.008 (2)	0.020 (2)
C19	0.051 (3)	0.059 (3)	0.035 (2)	−0.012 (2)	−0.0053 (18)	0.006 (2)
C20	0.034 (2)	0.038 (2)	0.041 (2)	−0.0059 (17)	−0.0014 (17)	0.0030 (18)
C21	0.037 (2)	0.031 (2)	0.039 (2)	−0.0070 (17)	0.0047 (17)	−0.0022 (17)
C22	0.053 (3)	0.030 (2)	0.056 (3)	0.0039 (19)	0.008 (2)	−0.0044 (19)
C23	0.062 (3)	0.036 (2)	0.045 (2)	−0.008 (2)	0.008 (2)	−0.0097 (18)
C24	0.046 (2)	0.033 (2)	0.036 (2)	−0.0143 (18)	0.0021 (18)	−0.0007 (17)
C25	0.058 (3)	0.049 (3)	0.046 (2)	−0.023 (2)	0.000 (2)	−0.005 (2)

C26	0.057 (3)	0.071 (3)	0.049 (3)	−0.025 (3)	−0.013 (2)	0.007 (2)
C27	0.042 (2)	0.050 (3)	0.063 (3)	−0.006 (2)	−0.005 (2)	0.009 (2)
C28	0.039 (2)	0.037 (2)	0.047 (2)	−0.0013 (18)	0.0019 (18)	−0.0049 (18)
C29	0.040 (2)	0.030 (2)	0.035 (2)	−0.0111 (16)	0.0025 (17)	0.0025 (16)
C30	0.039 (2)	0.0295 (19)	0.033 (2)	−0.0064 (16)	0.0014 (16)	0.0007 (16)
C31	0.031 (2)	0.034 (2)	0.038 (2)	−0.0039 (16)	0.0009 (16)	−0.0077 (17)
C32	0.033 (2)	0.032 (2)	0.049 (2)	−0.0020 (16)	−0.0029 (17)	−0.0050 (18)
C33	0.054 (3)	0.034 (2)	0.059 (3)	−0.0066 (19)	−0.007 (2)	0.000 (2)
C34	0.066 (3)	0.043 (3)	0.070 (3)	−0.011 (2)	−0.008 (2)	0.011 (2)
C35	0.064 (3)	0.029 (2)	0.101 (4)	−0.008 (2)	−0.013 (3)	−0.001 (3)
C36	0.053 (3)	0.039 (3)	0.082 (3)	−0.004 (2)	−0.016 (2)	−0.014 (2)
C37	0.039 (2)	0.035 (2)	0.059 (3)	−0.0034 (18)	−0.0042 (19)	−0.011 (2)
C38	0.062 (3)	0.050 (3)	0.058 (3)	−0.003 (2)	−0.004 (2)	−0.024 (2)
C39	0.065 (3)	0.056 (3)	0.045 (3)	−0.003 (2)	−0.003 (2)	−0.012 (2)
C40	0.043 (2)	0.036 (2)	0.040 (2)	−0.0024 (17)	0.0036 (18)	−0.0026 (18)
C41	0.052 (2)	0.030 (2)	0.038 (2)	−0.0067 (18)	−0.0003 (18)	0.0004 (17)
C42	0.082 (3)	0.050 (3)	0.033 (2)	−0.018 (2)	−0.005 (2)	−0.0039 (19)
C43	0.082 (3)	0.040 (2)	0.046 (3)	−0.017 (2)	−0.001 (2)	−0.014 (2)
C44	0.040 (2)	0.034 (2)	0.050 (2)	−0.0093 (17)	0.0007 (18)	−0.0066 (18)
C45	0.051 (3)	0.035 (2)	0.064 (3)	−0.0117 (19)	−0.007 (2)	−0.008 (2)
C46	0.044 (2)	0.030 (2)	0.080 (3)	−0.0124 (18)	−0.006 (2)	0.005 (2)
C47	0.046 (2)	0.044 (2)	0.056 (3)	−0.0094 (19)	0.000 (2)	0.013 (2)
C48	0.043 (2)	0.038 (2)	0.048 (2)	−0.0068 (18)	0.0031 (18)	0.0031 (19)
C49	0.035 (2)	0.0269 (19)	0.044 (2)	−0.0022 (16)	−0.0012 (17)	−0.0017 (17)
C50	0.033 (2)	0.030 (2)	0.039 (2)	−0.0049 (16)	0.0003 (16)	−0.0029 (16)
C51	0.035 (2)	0.0256 (18)	0.033 (2)	−0.0059 (15)	0.0042 (16)	−0.0011 (15)
C52	0.038 (2)	0.030 (2)	0.038 (2)	−0.0104 (16)	0.0033 (17)	0.0008 (16)
C53	0.039 (2)	0.040 (2)	0.047 (2)	−0.0041 (18)	−0.0020 (19)	0.0027 (18)
C54	0.043 (2)	0.056 (3)	0.058 (3)	−0.008 (2)	−0.005 (2)	0.011 (2)
C55	0.060 (3)	0.075 (3)	0.050 (3)	−0.025 (3)	−0.020 (2)	0.014 (2)
C56	0.074 (3)	0.059 (3)	0.041 (2)	−0.027 (3)	−0.010 (2)	−0.004 (2)
C57	0.049 (2)	0.034 (2)	0.036 (2)	−0.0119 (18)	0.0054 (18)	−0.0032 (17)
C58	0.067 (3)	0.042 (2)	0.037 (2)	−0.018 (2)	0.013 (2)	−0.0165 (19)
C59	0.048 (2)	0.032 (2)	0.053 (3)	−0.0029 (18)	0.012 (2)	−0.0079 (19)
C60	0.033 (2)	0.038 (2)	0.040 (2)	−0.0050 (17)	0.0038 (17)	−0.0003 (17)
C61	0.043 (2)	0.040 (2)	0.036 (2)	−0.0093 (18)	−0.0023 (17)	−0.0031 (18)
C62	0.065 (3)	0.070 (3)	0.033 (2)	−0.022 (2)	−0.012 (2)	0.004 (2)
C63	0.067 (3)	0.057 (3)	0.051 (3)	−0.016 (2)	−0.006 (2)	0.017 (2)
C64	0.039 (2)	0.042 (2)	0.047 (2)	−0.0062 (18)	−0.0060 (18)	0.0086 (19)
C65	0.053 (3)	0.039 (2)	0.069 (3)	−0.006 (2)	−0.001 (2)	0.015 (2)
C66	0.057 (3)	0.032 (2)	0.081 (3)	−0.006 (2)	−0.002 (2)	−0.007 (2)
C67	0.051 (3)	0.041 (2)	0.059 (3)	−0.008 (2)	−0.002 (2)	−0.014 (2)
C68	0.042 (2)	0.036 (2)	0.042 (2)	−0.0073 (18)	−0.0020 (18)	−0.0031 (18)
C69	0.032 (2)	0.035 (2)	0.038 (2)	−0.0061 (16)	−0.0011 (16)	−0.0022 (17)
C70	0.034 (2)	0.034 (2)	0.035 (2)	−0.0062 (16)	−0.0021 (16)	−0.0031 (16)
C71	0.037 (2)	0.0302 (19)	0.0291 (19)	−0.0092 (16)	0.0005 (16)	−0.0033 (15)
C72	0.035 (2)	0.0301 (19)	0.034 (2)	−0.0029 (16)	−0.0038 (16)	−0.0056 (16)
C73	0.041 (2)	0.042 (2)	0.047 (2)	−0.0089 (18)	−0.0074 (18)	−0.0053 (18)

C74	0.045 (3)	0.055 (3)	0.071 (3)	−0.005 (2)	−0.018 (2)	−0.009 (2)
C75	0.068 (3)	0.050 (3)	0.064 (3)	0.011 (2)	−0.033 (2)	−0.010 (2)
C76	0.075 (3)	0.048 (3)	0.052 (3)	−0.005 (2)	−0.023 (2)	0.006 (2)
C77	0.054 (2)	0.033 (2)	0.036 (2)	−0.0048 (18)	−0.0052 (18)	−0.0022 (17)
C78	0.066 (3)	0.043 (2)	0.038 (2)	−0.018 (2)	−0.002 (2)	0.0035 (19)
C79	0.047 (2)	0.046 (2)	0.048 (2)	−0.0181 (19)	0.004 (2)	−0.008 (2)
C80	0.036 (2)	0.033 (2)	0.033 (2)	−0.0040 (16)	−0.0006 (16)	−0.0088 (16)

Geometric parameters (Å, º)

P1—O3	1.457 (3)	C32—C37	1.417 (5)
P1—O4	1.529 (3)	C33—C34	1.374 (5)
P1—O1	1.572 (3)	C33—H33	0.9300
P1—O2	1.583 (2)	C34—C35	1.399 (6)
P2—O7	1.471 (2)	C34—H34	0.9300
P2—O8	1.519 (3)	C35—C36	1.344 (6)
P2—O5	1.576 (2)	C35—H35	0.9300
P2—O6	1.578 (2)	C36—C37	1.411 (5)
P3—O12	1.442 (3)	C36—H36	0.9300
P3—O11	1.528 (3)	C37—C38	1.406 (5)
P3—O10	1.572 (3)	C38—C39	1.356 (5)
P3—O9	1.578 (3)	C38—H38	0.9300
P4—O15	1.466 (2)	C39—C40	1.393 (5)
P4—O16	1.524 (3)	C39—H39	0.9300
P4—O14	1.576 (2)	C41—C50	1.369 (5)
P4—O13	1.578 (3)	C41—C42	1.396 (5)
O1—C1	1.416 (4)	C42—C43	1.354 (5)
O2—C20	1.416 (4)	C42—H42	0.9300
O4—H1O	0.8200	C43—C44	1.405 (5)
O5—C21	1.408 (4)	C43—H43	0.9300
O6—C40	1.413 (4)	C44—C45	1.418 (5)
O8—H2O	0.8200	C44—C49	1.421 (5)
O9—C41	1.412 (4)	C45—C46	1.338 (5)
O10—C60	1.413 (4)	C45—H45	0.9300
O11—H30	0.8200	C46—C47	1.398 (5)
O13—C61	1.420 (4)	C46—H46	0.9300
O14—C80	1.410 (4)	C47—C48	1.368 (5)
O16—H4O	0.8200	C47—H47	0.9300
C1—C10	1.360 (4)	C48—C49	1.405 (5)
C1—C2	1.400 (5)	C48—H48	0.9300
C2—C3	1.352 (5)	C49—C50	1.436 (4)
C2—H2	0.9300	C50—C51	1.493 (4)
C3—C4	1.405 (5)	C51—C60	1.373 (4)
C3—H3	0.9300	C51—C52	1.441 (4)
C4—C5	1.415 (5)	C52—C53	1.414 (5)
C4—C9	1.424 (5)	C52—C57	1.418 (5)
C5—C6	1.351 (6)	C53—C54	1.356 (5)
C5—H5	0.9300	C53—H53	0.9300

C6—C7	1.398 (5)	C54—C55	1.391 (5)
C6—H6	0.9300	C54—H54	0.9300
C7—C8	1.365 (5)	C55—C56	1.356 (5)
C7—H7	0.9300	C55—H55	0.9300
C8—C9	1.407 (5)	C56—C57	1.417 (5)
C8—H8	0.9300	C56—H56	0.9300
C9—C10	1.441 (4)	C57—C58	1.412 (5)
C10—C11	1.493 (4)	C58—C59	1.351 (5)
C11—C20	1.363 (4)	C58—H58	0.9300
C11—C12	1.444 (4)	C59—C60	1.394 (5)
C12—C13	1.407 (5)	C59—H59	0.9300
C12—C17	1.419 (4)	C61—C70	1.367 (4)
C13—C14	1.359 (5)	C61—C62	1.390 (5)
C13—H13	0.9300	C62—C63	1.357 (5)
C14—C15	1.397 (5)	C62—H62	0.9300
C14—H14	0.9300	C63—C64	1.409 (5)
C15—C16	1.350 (5)	C63—H63	0.9300
C15—H15	0.9300	C64—C69	1.417 (5)
C16—C17	1.416 (5)	C64—C65	1.421 (5)
C16—H16	0.9300	C65—C66	1.345 (5)
C17—C18	1.410 (5)	C65—H65	0.9300
C18—C19	1.358 (5)	C66—C67	1.393 (5)
C18—H18	0.9300	C66—H66	0.9300
C19—C20	1.396 (5)	C67—C68	1.353 (5)
C19—H19	0.9300	C67—H67	0.9300
C21—C30	1.372 (4)	C68—C69	1.410 (5)
C21—C22	1.395 (5)	C68—H68	0.9300
C22—C23	1.358 (5)	C69—C70	1.436 (4)
C22—H22	0.9300	C70—C71	1.494 (4)
C23—C24	1.416 (5)	C71—C80	1.364 (4)
C23—H23	0.9300	C71—C72	1.441 (4)
C24—C25	1.411 (5)	C72—C73	1.408 (5)
C24—C29	1.431 (5)	C72—C77	1.420 (5)
C25—C26	1.356 (5)	C73—C74	1.364 (5)
C25—H25	0.9300	C73—H73	0.9300
C26—C27	1.397 (5)	C74—C75	1.396 (6)
C26—H26	0.9300	C74—H74	0.9300
C27—C28	1.356 (5)	C75—C76	1.351 (6)
C27—H27	0.9300	C75—H75	0.9300
C28—C29	1.412 (5)	C76—C77	1.413 (5)
C28—H28	0.9300	C76—H76	0.9300
C29—C30	1.431 (5)	C77—C78	1.404 (5)
C30—C31	1.488 (4)	C78—C79	1.362 (5)
C31—C40	1.361 (5)	C78—H78	0.9300
C31—C32	1.440 (5)	C79—C80	1.399 (5)
C32—C33	1.409 (5)	C79—H79	0.9300
O3—P1—O4	117.18 (16)	C36—C35—C34	119.7 (4)

O3—P1—O1	107.23 (16)	C36—C35—H35	120.2
O4—P1—O1	110.50 (14)	C34—C35—H35	120.2
O3—P1—O2	115.41 (15)	C35—C36—C37	121.4 (4)
O4—P1—O2	100.66 (14)	C35—C36—H36	119.3
O1—P1—O2	105.23 (13)	C37—C36—H36	119.3
O7—P2—O8	116.74 (15)	C38—C37—C36	121.4 (4)
O7—P2—O5	114.68 (15)	C38—C37—C32	118.8 (3)
O8—P2—O5	100.86 (14)	C36—C37—C32	119.7 (4)
O7—P2—O6	108.12 (14)	C39—C38—C37	121.6 (4)
O8—P2—O6	111.19 (15)	C39—C38—H38	119.2
O5—P2—O6	104.54 (13)	C37—C38—H38	119.2
O12—P3—O11	116.93 (18)	C38—C39—C40	118.5 (4)
O12—P3—O10	109.23 (18)	C38—C39—H39	120.8
O11—P3—O10	106.08 (15)	C40—C39—H39	120.8
O12—P3—O9	114.84 (19)	C31—C40—C39	124.4 (4)
O11—P3—O9	103.64 (14)	C31—C40—O6	119.2 (3)
O10—P3—O9	105.14 (13)	C39—C40—O6	116.4 (3)
O15—P4—O16	117.59 (16)	C50—C41—C42	124.4 (3)
O15—P4—O14	114.69 (15)	C50—C41—O9	118.9 (3)
O16—P4—O14	101.59 (15)	C42—C41—O9	116.7 (3)
O15—P4—O13	107.79 (15)	C43—C42—C41	118.2 (4)
O16—P4—O13	109.82 (15)	C43—C42—H42	120.9
O14—P4—O13	104.53 (13)	C41—C42—H42	120.9
C1—O1—P1	117.7 (2)	C42—C43—C44	121.6 (4)
C20—O2—P1	114.8 (2)	C42—C43—H43	119.2
P1—O4—H1O	109.5	C44—C43—H43	119.2
C21—O5—P2	116.8 (2)	C43—C44—C45	121.3 (4)
C40—O6—P2	118.2 (2)	C43—C44—C49	119.6 (3)
P2—O8—H2O	109.5	C45—C44—C49	119.1 (3)
C41—O9—P3	118.8 (2)	C46—C45—C44	121.2 (4)
C60—O10—P3	116.4 (2)	C46—C45—H45	119.4
P3—O11—H30	109.5	C44—C45—H45	119.4
C61—O13—P4	116.0 (2)	C45—C46—C47	120.3 (4)
C80—O14—P4	118.2 (2)	C45—C46—H46	119.9
P4—O16—H4O	109.5	C47—C46—H46	119.9
C10—C1—C2	123.8 (3)	C48—C47—C46	120.4 (4)
C10—C1—O1	119.1 (3)	C48—C47—H47	119.8
C2—C1—O1	117.1 (3)	C46—C47—H47	119.8
C3—C2—C1	118.9 (4)	C47—C48—C49	121.2 (4)
C3—C2—H2	120.6	C47—C48—H48	119.4
C1—C2—H2	120.6	C49—C48—H48	119.4
C2—C3—C4	121.3 (3)	C48—C49—C44	117.8 (3)
C2—C3—H3	119.3	C48—C49—C50	123.2 (3)
C4—C3—H3	119.3	C44—C49—C50	119.0 (3)
C3—C4—C5	122.2 (4)	C41—C50—C49	117.1 (3)
C3—C4—C9	119.4 (3)	C41—C50—C51	119.9 (3)
C5—C4—C9	118.4 (4)	C49—C50—C51	122.9 (3)
C6—C5—C4	122.2 (4)	C60—C51—C52	116.3 (3)

C6—C5—H5	118.9	C60—C51—C50	120.4 (3)
C4—C5—H5	118.9	C52—C51—C50	123.1 (3)
C5—C6—C7	119.3 (4)	C53—C52—C57	117.9 (3)
C5—C6—H6	120.4	C53—C52—C51	122.4 (3)
C7—C6—H6	120.4	C57—C52—C51	119.7 (3)
C8—C7—C6	120.7 (4)	C54—C53—C52	121.1 (4)
C8—C7—H7	119.7	C54—C53—H53	119.4
C6—C7—H7	119.7	C52—C53—H53	119.4
C7—C8—C9	121.5 (4)	C53—C54—C55	121.1 (4)
C7—C8—H8	119.2	C53—C54—H54	119.4
C9—C8—H8	119.2	C55—C54—H54	119.4
C8—C9—C4	117.9 (3)	C56—C55—C54	119.7 (4)
C8—C9—C10	123.2 (3)	C56—C55—H55	120.1
C4—C9—C10	118.9 (3)	C54—C55—H55	120.1
C1—C10—C9	117.6 (3)	C55—C56—C57	121.1 (4)
C1—C10—C11	119.0 (3)	C55—C56—H56	119.4
C9—C10—C11	123.4 (3)	C57—C56—H56	119.4
C20—C11—C12	117.2 (3)	C58—C57—C56	121.6 (4)
C20—C11—C10	120.2 (3)	C58—C57—C52	119.3 (3)
C12—C11—C10	122.3 (3)	C56—C57—C52	119.0 (4)
C13—C12—C17	117.9 (3)	C59—C58—C57	121.2 (3)
C13—C12—C11	122.9 (3)	C59—C58—H58	119.4
C17—C12—C11	119.1 (3)	C57—C58—H58	119.4
C14—C13—C12	121.2 (3)	C58—C59—C60	118.7 (4)
C14—C13—H13	119.4	C58—C59—H59	120.6
C12—C13—H13	119.4	C60—C59—H59	120.6
C13—C14—C15	120.9 (4)	C51—C60—C59	124.5 (3)
C13—C14—H14	119.6	C51—C60—O10	118.9 (3)
C15—C14—H14	119.6	C59—C60—O10	116.6 (3)
C16—C15—C14	119.8 (4)	C70—C61—C62	124.3 (3)
C16—C15—H15	120.1	C70—C61—O13	118.3 (3)
C14—C15—H15	120.1	C62—C61—O13	117.3 (3)
C15—C16—C17	121.2 (4)	C63—C62—C61	118.9 (4)
C15—C16—H16	119.4	C63—C62—H62	120.5
C17—C16—H16	119.4	C61—C62—H62	120.5
C18—C17—C16	121.4 (4)	C62—C63—C64	120.7 (4)
C18—C17—C12	119.5 (3)	C62—C63—H63	119.6
C16—C17—C12	119.0 (4)	C64—C63—H63	119.6
C19—C18—C17	121.0 (4)	C63—C64—C69	119.6 (3)
C19—C18—H18	119.5	C63—C64—C65	121.4 (4)
C17—C18—H18	119.5	C69—C64—C65	119.0 (4)
C18—C19—C20	118.8 (4)	C66—C65—C64	120.8 (4)
C18—C19—H19	120.6	C66—C65—H65	119.6
C20—C19—H19	120.6	C64—C65—H65	119.6
C11—C20—C19	124.0 (3)	C65—C66—C67	120.2 (4)
C11—C20—O2	118.7 (3)	C65—C66—H66	119.9
C19—C20—O2	117.2 (3)	C67—C66—H66	119.9
C30—C21—C22	124.0 (3)	C68—C67—C66	121.1 (4)

C30—C21—O5	119.4 (3)	C68—C67—H67	119.5
C22—C21—O5	116.6 (3)	C66—C67—H67	119.5
C23—C22—C21	119.5 (4)	C67—C68—C69	121.0 (4)
C23—C22—H22	120.3	C67—C68—H68	119.5
C21—C22—H22	120.3	C69—C68—H68	119.5
C22—C23—C24	120.3 (4)	C68—C69—C64	118.0 (3)
C22—C23—H23	119.8	C68—C69—C70	122.5 (3)
C24—C23—H23	119.8	C64—C69—C70	119.4 (3)
C25—C24—C23	121.6 (4)	C61—C70—C69	116.9 (3)
C25—C24—C29	118.9 (3)	C61—C70—C71	120.0 (3)
C23—C24—C29	119.5 (3)	C69—C70—C71	122.8 (3)
C26—C25—C24	121.4 (4)	C80—C71—C72	117.8 (3)
C26—C25—H25	119.3	C80—C71—C70	119.4 (3)
C24—C25—H25	119.3	C72—C71—C70	122.8 (3)
C25—C26—C27	119.9 (4)	C73—C72—C77	118.6 (3)
C25—C26—H26	120.1	C73—C72—C71	122.9 (3)
C27—C26—H26	120.1	C77—C72—C71	118.5 (3)
C28—C27—C26	120.7 (4)	C74—C73—C72	121.1 (4)
C28—C27—H27	119.6	C74—C73—H73	119.5
C26—C27—H27	119.6	C72—C73—H73	119.5
C27—C28—C29	121.6 (4)	C73—C74—C75	120.3 (4)
C27—C28—H28	119.2	C73—C74—H74	119.8
C29—C28—H28	119.2	C75—C74—H74	119.8
C28—C29—C24	117.5 (3)	C76—C75—C74	120.2 (4)
C28—C29—C30	123.0 (3)	C76—C75—H75	119.9
C24—C29—C30	119.4 (3)	C74—C75—H75	119.9
C21—C30—C29	117.1 (3)	C75—C76—C77	121.6 (4)
C21—C30—C31	119.6 (3)	C75—C76—H76	119.2
C29—C30—C31	123.1 (3)	C77—C76—H76	119.2
C40—C31—C32	116.8 (3)	C78—C77—C76	121.6 (4)
C40—C31—C30	119.8 (3)	C78—C77—C72	120.1 (3)
C32—C31—C30	123.3 (3)	C76—C77—C72	118.3 (4)
C33—C32—C37	117.5 (3)	C79—C78—C77	121.0 (3)
C33—C32—C31	122.7 (3)	C79—C78—H78	119.5
C37—C32—C31	119.7 (3)	C77—C78—H78	119.5
C34—C33—C32	120.9 (4)	C78—C79—C80	118.6 (3)
C34—C33—H33	119.6	C78—C79—H79	120.7
C32—C33—H33	119.6	C80—C79—H79	120.7
C33—C34—C35	120.8 (4)	C71—C80—C79	123.8 (3)
C33—C34—H34	119.6	C71—C80—O14	119.5 (3)
C35—C34—H34	119.6	C79—C80—O14	116.7 (3)

O3—P1—O1—C1	−165.3 (2)	C31—C32—C37—C36	−179.3 (3)
O4—P1—O1—C1	65.9 (3)	C36—C37—C38—C39	177.0 (4)
O2—P1—O1—C1	−41.9 (3)	C32—C37—C38—C39	−0.7 (6)
O3—P1—O2—C20	68.8 (3)	C37—C38—C39—C40	1.1 (6)
O4—P1—O2—C20	−164.0 (2)	C32—C31—C40—C39	−3.3 (5)
O1—P1—O2—C20	−49.1 (2)	C30—C31—C40—C39	179.2 (3)

O7—P2—O5—C21	−71.7 (3)	C32—C31—C40—O6	176.4 (3)
O8—P2—O5—C21	161.9 (2)	C30—C31—C40—O6	−1.1 (5)
O6—P2—O5—C21	46.5 (3)	C38—C39—C40—C31	1.1 (6)
O7—P2—O6—C40	166.8 (2)	C38—C39—C40—O6	−178.7 (3)
O8—P2—O6—C40	−63.8 (3)	P2—O6—C40—C31	−73.1 (4)
O5—P2—O6—C40	44.2 (3)	P2—O6—C40—C39	106.7 (3)
O12—P3—O9—C41	76.8 (3)	P3—O9—C41—C50	72.1 (4)
O11—P3—O9—C41	−154.4 (2)	P3—O9—C41—C42	−108.2 (3)
O10—P3—O9—C41	−43.3 (3)	C50—C41—C42—C43	−0.4 (6)
O12—P3—O10—C60	−171.3 (3)	O9—C41—C42—C43	180.0 (3)
O11—P3—O10—C60	61.8 (3)	C41—C42—C43—C44	−2.2 (6)
O9—P3—O10—C60	−47.6 (3)	C42—C43—C44—C45	−177.3 (4)
O15—P4—O13—C61	171.0 (2)	C42—C43—C44—C49	1.4 (6)
O16—P4—O13—C61	−59.8 (3)	C43—C44—C45—C46	177.9 (4)
O14—P4—O13—C61	48.5 (3)	C49—C44—C45—C46	−0.9 (6)
O15—P4—O14—C80	−75.0 (3)	C44—C45—C46—C47	−1.2 (6)
O16—P4—O14—C80	157.0 (2)	C45—C46—C47—C48	1.9 (6)
O13—P4—O14—C80	42.8 (3)	C46—C47—C48—C49	−0.4 (6)
P1—O1—C1—C10	73.7 (4)	C47—C48—C49—C44	−1.6 (5)
P1—O1—C1—C2	−107.5 (3)	C47—C48—C49—C50	−179.8 (3)
C10—C1—C2—C3	−2.1 (6)	C43—C44—C49—C48	−176.5 (3)
O1—C1—C2—C3	179.2 (3)	C45—C44—C49—C48	2.2 (5)
C1—C2—C3—C4	−0.7 (6)	C43—C44—C49—C50	1.8 (5)
C2—C3—C4—C5	−177.1 (4)	C45—C44—C49—C50	−179.5 (3)
C2—C3—C4—C9	1.7 (6)	C42—C41—C50—C49	3.5 (6)
C3—C4—C5—C6	179.1 (4)	O9—C41—C50—C49	−176.9 (3)
C9—C4—C5—C6	0.3 (6)	C42—C41—C50—C51	−178.8 (3)
C4—C5—C6—C7	−0.5 (6)	O9—C41—C50—C51	0.8 (5)
C5—C6—C7—C8	0.0 (6)	C48—C49—C50—C41	174.1 (3)
C6—C7—C8—C9	0.6 (6)	C44—C49—C50—C41	−4.1 (5)
C7—C8—C9—C4	−0.7 (5)	C48—C49—C50—C51	−3.4 (5)
C7—C8—C9—C10	−179.1 (3)	C44—C49—C50—C51	178.3 (3)
C3—C4—C9—C8	−178.6 (3)	C41—C50—C51—C60	−51.4 (5)
C5—C4—C9—C8	0.2 (5)	C49—C50—C51—C60	126.1 (4)
C3—C4—C9—C10	−0.1 (5)	C41—C50—C51—C52	122.8 (4)
C5—C4—C9—C10	178.7 (3)	C49—C50—C51—C52	−59.7 (5)
C2—C1—C10—C9	3.6 (5)	C60—C51—C52—C53	171.2 (3)
O1—C1—C10—C9	−177.7 (3)	C50—C51—C52—C53	−3.2 (5)
C2—C1—C10—C11	−179.7 (3)	C60—C51—C52—C57	−5.5 (5)
O1—C1—C10—C11	−1.0 (5)	C50—C51—C52—C57	−179.9 (3)
C8—C9—C10—C1	176.0 (3)	C57—C52—C53—C54	−0.8 (5)
C4—C9—C10—C1	−2.4 (5)	C51—C52—C53—C54	−177.6 (3)
C8—C9—C10—C11	−0.6 (5)	C52—C53—C54—C55	−1.3 (6)
C4—C9—C10—C11	−178.9 (3)	C53—C54—C55—C56	1.8 (6)
C1—C10—C11—C20	−52.8 (5)	C54—C55—C56—C57	−0.3 (6)
C9—C10—C11—C20	123.7 (4)	C55—C56—C57—C58	175.0 (4)
C1—C10—C11—C12	121.6 (4)	C55—C56—C57—C52	−1.8 (6)
C9—C10—C11—C12	−61.9 (5)	C53—C52—C57—C58	−174.5 (3)

C20—C11—C12—C13	171.2 (3)	C51—C52—C57—C58	2.3 (5)
C10—C11—C12—C13	−3.3 (5)	C53—C52—C57—C56	2.3 (5)
C20—C11—C12—C17	−4.4 (5)	C51—C52—C57—C56	179.2 (3)
C10—C11—C12—C17	−179.0 (3)	C56—C57—C58—C59	−175.1 (3)
C17—C12—C13—C14	−0.8 (5)	C52—C57—C58—C59	1.6 (5)
C11—C12—C13—C14	−176.5 (3)	C57—C58—C59—C60	−2.1 (5)
C12—C13—C14—C15	−0.2 (6)	C52—C51—C60—C59	5.3 (5)
C13—C14—C15—C16	0.9 (6)	C50—C51—C60—C59	179.9 (3)
C14—C15—C16—C17	−0.7 (6)	C52—C51—C60—O10	−174.4 (3)
C15—C16—C17—C18	176.7 (4)	C50—C51—C60—O10	0.1 (5)
C15—C16—C17—C12	−0.3 (6)	C58—C59—C60—C51	−1.5 (5)
C13—C12—C17—C18	−176.1 (3)	C58—C59—C60—O10	178.2 (3)
C11—C12—C17—C18	−0.2 (5)	P3—O10—C60—C51	74.6 (3)
C13—C12—C17—C16	1.0 (5)	P3—O10—C60—C59	−105.1 (3)
C11—C12—C17—C16	176.9 (3)	P4—O13—C61—C70	−75.9 (4)
C16—C17—C18—C19	−173.7 (4)	P4—O13—C61—C62	102.5 (3)
C12—C17—C18—C19	3.3 (6)	C70—C61—C62—C63	3.2 (6)
C17—C18—C19—C20	−1.7 (6)	O13—C61—C62—C63	−175.1 (3)
C12—C11—C20—C19	6.4 (5)	C61—C62—C63—C64	0.2 (6)
C10—C11—C20—C19	−179.0 (3)	C62—C63—C64—C69	−1.9 (6)
C12—C11—C20—O2	−172.6 (3)	C62—C63—C64—C65	176.1 (4)
C10—C11—C20—O2	2.0 (5)	C63—C64—C65—C66	−177.9 (4)
C18—C19—C20—C11	−3.4 (6)	C69—C64—C65—C66	0.1 (6)
C18—C19—C20—O2	175.6 (3)	C64—C65—C66—C67	0.3 (6)
P1—O2—C20—C11	74.7 (3)	C65—C66—C67—C68	0.0 (6)
P1—O2—C20—C19	−104.3 (3)	C66—C67—C68—C69	−0.7 (6)
P2—O5—C21—C30	−74.2 (3)	C67—C68—C69—C64	1.0 (5)
P2—O5—C21—C22	105.9 (3)	C67—C68—C69—C70	177.6 (3)
C30—C21—C22—C23	2.2 (6)	C63—C64—C69—C68	177.3 (3)
O5—C21—C22—C23	−177.9 (3)	C65—C64—C69—C68	−0.8 (5)
C21—C22—C23—C24	2.4 (6)	C63—C64—C69—C70	0.6 (5)
C22—C23—C24—C25	175.7 (3)	C65—C64—C69—C70	−177.4 (3)
C22—C23—C24—C29	−2.9 (5)	C62—C61—C70—C69	−4.4 (5)
C23—C24—C25—C26	−176.6 (4)	O13—C61—C70—C69	173.9 (3)
C29—C24—C25—C26	2.0 (5)	C62—C61—C70—C71	−178.5 (3)
C24—C25—C26—C27	0.1 (6)	O13—C61—C70—C71	−0.2 (5)
C25—C26—C27—C28	−1.6 (6)	C68—C69—C70—C61	−174.1 (3)
C26—C27—C28—C29	0.9 (6)	C64—C69—C70—C61	2.4 (5)
C27—C28—C29—C24	1.1 (5)	C68—C69—C70—C71	−0.1 (5)
C27—C28—C29—C30	178.1 (3)	C64—C69—C70—C71	176.4 (3)
C25—C24—C29—C28	−2.5 (5)	C61—C70—C71—C80	52.1 (5)
C23—C24—C29—C28	176.1 (3)	C69—C70—C71—C80	−121.7 (4)
C25—C24—C29—C30	−179.7 (3)	C61—C70—C71—C72	−125.5 (4)
C23—C24—C29—C30	−1.0 (5)	C69—C70—C71—C72	60.7 (5)
C22—C21—C30—C29	−5.9 (5)	C80—C71—C72—C73	−173.5 (3)
O5—C21—C30—C29	174.1 (3)	C70—C71—C72—C73	4.0 (5)
C22—C21—C30—C31	179.1 (3)	C80—C71—C72—C77	4.5 (5)
O5—C21—C30—C31	−0.8 (5)	C70—C71—C72—C77	−177.9 (3)

C28—C29—C30—C21	−171.8 (3)	C77—C72—C73—C74	0.9 (5)
C24—C29—C30—C21	5.2 (5)	C71—C72—C73—C74	178.9 (3)
C28—C29—C30—C31	3.0 (5)	C72—C73—C74—C75	0.1 (6)
C24—C29—C30—C31	179.9 (3)	C73—C74—C75—C76	−0.6 (6)
C21—C30—C31—C40	52.2 (5)	C74—C75—C76—C77	0.0 (6)
C29—C30—C31—C40	−122.4 (4)	C75—C76—C77—C78	−177.1 (4)
C21—C30—C31—C32	−125.1 (4)	C75—C76—C77—C72	1.0 (6)
C29—C30—C31—C32	60.3 (5)	C73—C72—C77—C78	176.8 (3)
C40—C31—C32—C33	−174.1 (3)	C71—C72—C77—C78	−1.4 (5)
C30—C31—C32—C33	3.3 (5)	C73—C72—C77—C76	−1.4 (5)
C40—C31—C32—C37	3.5 (5)	C71—C72—C77—C76	−179.6 (3)
C30—C31—C32—C37	−179.1 (3)	C76—C77—C78—C79	176.8 (4)
C37—C32—C33—C34	1.8 (5)	C72—C77—C78—C79	−1.3 (6)
C31—C32—C33—C34	179.4 (3)	C77—C78—C79—C80	0.8 (6)
C32—C33—C34—C35	−0.4 (6)	C72—C71—C80—C79	−5.3 (5)
C33—C34—C35—C36	−1.2 (7)	C70—C71—C80—C79	177.0 (3)
C34—C35—C36—C37	1.3 (6)	C72—C71—C80—O14	176.8 (3)
C35—C36—C37—C38	−177.6 (4)	C70—C71—C80—O14	−0.8 (5)
C35—C36—C37—C32	0.1 (6)	C78—C79—C80—C71	2.7 (5)
C33—C32—C37—C38	176.2 (3)	C78—C79—C80—O14	−179.4 (3)
C31—C32—C37—C38	−1.6 (5)	P4—O14—C80—C71	−72.6 (4)
C33—C32—C37—C36	−1.6 (5)	P4—O14—C80—C79	109.4 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H1*O*···O7	0.82	1.78	2.475 (4)	141
O8—H2*O*···O3^{i}	0.82	1.77	2.450 (4)	139
O16—H4*O*···O12^{ii}	0.82	1.99	2.441 (4)	114
O11—H30···O15	0.82	1.71	2.477 (4)	154
C22—H22···O11^{iii}	0.93	2.51	3.435 (4)	174
C59—H59···O7^{iv}	0.93	2.56	3.478 (4)	167
C27—H27···*Cg*1	0.93	2.75	3.567 (5)	147
C54—H54···*Cg*2^{v}	0.93	2.77	3.535 (5)	140

Symmetry codes: (i) −*x*, −*y*+1, −*z*; (ii) −*x*+2, −*y*+1, −*z*+1; (iii) *x*−1, *y*, *z*; (iv) *x*+1, *y*, *z*; (v) −*x*+1, −*y*+1, −*z*+1.