Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1,1,1-Tris[(diphenylphosphoryl)methyl]-propane monohydrate

Mikhail S. Grigoriev,[a]* Svetlana A. Pisareva,[b] Alfiya M. Safiulina[a] and Eduard E. Nifant'ev[b]

[a]A. N. Frumkin Institute of Physical Chemistry and Electrochemistry, Russian Academy of Sciences, 31 Leninsky prospekt, 119991 Moscow, Russian Federation, and [b]A. N. Nesmeyanov Institute of Organoelement Compounds, Russian Academy of Sciences, 28 Vavilova street, 119991 Moscow, Russian Federation
Correspondence e-mail: grigoriev@ipc.rssi.ru

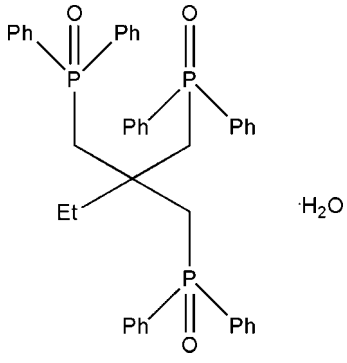
Received 21 June 2007; accepted 4 July 2007

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.035; wR factor = 0.068; data-to-parameter ratio = 23.2.

The arrangement of phosphoryl groups and phenyl rings in the title compound, $C_2H_5C[CH_2P(O)Ph_2]_3\cdot H_2O$ or $C_{42}H_{41}O_3P_3\cdot H_2O$, is far from threefold symmetry. The P═O bond lengths are 1.4899 (12), 1.4926 (12) and 1.4927 (12) Å. The O═P—C(methylene) angles differ significantly from ideal tetrahedral values, the maximum value being 117.08 (7)°. The C—C—C angles around the central C atom vary from 107.92 (12) to 111.91 (12)°. In the crystal structure, hydrogen bonds in which water molecules act as proton donors link organic molecules into chains along the [100] direction.

Related literature

The synthesis of the title compound was described by Pisareva *et al.* (2005). A structure of a solid solution of $C_2H_5C(CH_2PPh_2)_3$ and its partially oxygenated derivatives was described by Chekhlov (2000). Lees & Platt (2005) reported the structure of La and Pr nitrate complexes with $CH_3C[CH_2P(O)Ph_2]$, in which tridentate organic ligands are linked by phosphoryl O atoms to two Ln atoms, to one atom in a bidentate–chelating fashion and to the other in a monodentate fashion.



Experimental

Crystal data

$C_{42}H_{41}O_3P_3\cdot H_2O$
M_r = 704.67
Monoclinic, Pc
a = 9.5303 (2) Å
b = 10.4028 (2) Å
c = 18.0951 (4) Å
β = 90.1433 (14)°
V = 1793.98 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 100 (2) K
0.18 × 0.16 × 0.10 mm

Data collection

Bruker Kappa APEXII area-detector diffractometer
Absorption correction: none
41040 measured reflections
10441 independent reflections
8909 reflections with $I > 2\sigma(I)$
R_{int} = 0.059

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.068
S = 0.95
10441 reflections
450 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}
Absolute structure: Flack (1983), with 5207 Friedel pairs
Flack parameter: −0.04 (4)

Table 1
Selected geometric parameters (Å, °).

P1—O1	1.4927 (12)	P2—C41	1.8109 (16)
P1—C21	1.8057 (17)	P2—C3	1.8165 (16)
P1—C11	1.8175 (16)	P3—O3	1.4926 (12)
P1—C2	1.8257 (15)	P3—C4	1.8081 (16)
P2—O2	1.4899 (12)	P3—C51	1.8081 (16)
P2—C31	1.8060 (16)	P3—C61	1.8128 (16)
O1—P1—C2	115.31 (7)	C3—P2—C31	103.21 (7)
O1—P1—C11	109.75 (7)	C3—P2—C41	109.94 (7)
O1—P1—C21	111.79 (7)	C31—P2—C41	105.59 (7)
C2—P1—C11	107.97 (7)	O3—P3—C4	117.08 (7)
C2—P1—C21	103.67 (7)	O3—P3—C51	111.42 (7)
C11—P1—C21	107.96 (7)	O3—P3—C61	111.26 (7)
O2—P2—C3	114.00 (7)	C4—P3—C51	102.51 (7)
O2—P2—C31	112.73 (7)	C4—P3—C61	108.37 (7)
O2—P2—C41	110.81 (7)	C51—P3—C61	105.26 (7)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4C···O1	0.75 (3)	2.16 (3)	2.912 (2)	179 (3)
O4—H4D···O3^{i}	0.86 (3)	1.98 (3)	2.8413 (19)	176 (3)

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

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT-Plus* (Bruker, 1998); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

This work was supported by RFBR (project No. 05-03-08017-ofi_e) and program No. 8 of basic research of the Presidium of the Russian Academy of Sciences, program

'Development of methods of preparation of chemical substances and creation of new materials', subprogram 'Development of organic synthesis methodology and formation of new organic compounds with valuable practical properties'. The project involving the title compound is 'Development of methods of synthesis and study of properties of new mono- and polydentate organophosphorus compounds as applied to radioactive waste fractionation processes'.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2120).

References

Bruker (1998). *SAINT-Plus*. Version 6.01. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2006). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chekhlov, A. N. (2000). *Zh. Strukt. Khim.* **41**, 788–794.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Lees, A. M. J. & Platt, A. W. G. (2005). *Polyhedron*, **24**, 427–433.
Pisareva, S. A., Petrovskii, P. V., Lyssenko, K. A., Antipin, M. Yu. & Nifant'ev, E. E. (2005). *Russ. Chem. Bull. Int. Ed.* **53**, 2008–20012.
Sheldrick, G. M. (1997*a*). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.

supplementary materials

Acta Cryst. (2007). E**63**, o3441-o3442 [**doi:10.1107/S1600536807032527**]

1,1,1-Tris[(diphenylphosphoryl)methyl]propane monohydrate

M. S. Grigoriev, S. A. Pisareva, A. M. Safiulina and E. E. Nifant'ev

Comment

Organophosphorus ligands with three phosphoryl groups are considered as promising agents to ensure selectivity of separation of metal mixtures by extraction (Pisareva *et al.*, 2005).

The arrangement of phosphoryl groups and phenyl rings in the title compound, (I, Fig. 1), is far from a threefold symmetry. Selected bond lengths and angles in (I) are given in Table 1. The P═O distances range from 1.4899 (12) to 1.4927 (12) Å. The O═P—C angles with C atoms of CH_2 groups differ significantly from ideal tetrahedral values, the maximum value being 117.08 (7)° (Table 1). The C—C—C angles around the central atom C1 vary from 107.92 (12) to 111.91 (12)°.

The same behaviour of the O═P—C angles can be found in Ln nitrate complexes with $CH_3C[CH_2P(O)Ph_2]$ (Lees & Platt, 2005). The P═O distances in these complexes are slightly longer due to coordination of phosphoryl groups to Ln atoms [from 1.499 (3) to 1.510 (3) Å]. Apparent P═ O distances in partially oxygenated $C_2H_5C(CH_2PPh_2)_3$ (Chekhlov, 2000) are significantly shorter, due to a statistical character of the structure.

In (I), the hydrogen bonds in which water molecules act as proton donors (Table 2, Fig. 2) link organic molecules into chains along the [100] direction.

Experimental

The compound was prepared according to the procedure described by Pisareva *et al.* (2005) and recrystallized from dichloroethane.

Refinement

The H atoms of the organic molecule were refined in idealized geometrical positions with displacement parameters being equal to 1.2 (CH_2 groups and phenyl rings) or 1.5 (CH_3 groups) times U_{eq} of the attached C atoms. The H atoms of the water molecule were located in a difference map and refined with individual isotropic displacement parameters and free coordinates.

Figures

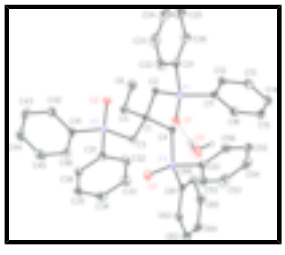


Fig. 1. A view of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by circles of arbitrary size. The H atoms of the organic molecule are omitted for clarity.

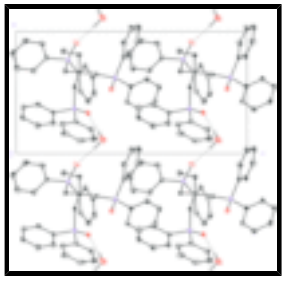


Fig. 2. The packing of (I), viewed down the [010] axis, showing the chains formed due to hydrogen bonding. H atoms not involved in hydrogen bonding are omitted.

1,1,1-Tris[(diphenylphosphoryl)methyl]propane monohydrate

Crystal data

$C_{42}H_{41}O_3P_3·H_2O$	F_{000} = 744
M_r = 704.67	D_x = 1.305 Mg m^{-3}
Monoclinic, *Pc*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: P -2yc	Cell parameters from 5344 reflections
a = 9.5303 (2) Å	θ = 2.1–30.0º
b = 10.4028 (2) Å	μ = 0.21 mm^{-1}
c = 18.0951 (4) Å	*T* = 100 (2) K
β = 90.1433 (14)º	Fragment, colourless
V = 1793.98 (6) Å^3	0.18 × 0.16 × 0.10 mm
Z = 2	

Data collection

Bruker Kappa APEX II area-detector diffractometer	8909 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.059
Monochromator: graphite	θ_{max} = 30.0º
T = 100(2) K	θ_{min} = 2.1º
ω and φ scans	*h* = −13→13
Absorption correction: none	*k* = −14→14
41040 measured reflections	*l* = −25→25
10441 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: mixed
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.035	$w = 1/[\sigma^2(F_o^2) + (0.0298P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.068	$(\Delta/\sigma)_{max}$ = 0.001
S = 0.95	$\Delta\rho_{max}$ = 0.29 e Å^{-3}
10441 reflections	$\Delta\rho_{min}$ = −0.25 e Å^{-3}

450 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Extinction correction: none
Absolute structure: Flack (1983), 5207 Friedel pairs
Flack parameter: −0.04 (4)

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

	x	*y*	*z*	U_{iso}*/U_{eq}
P1	0.36674 (4)	0.40193 (4)	0.75195 (2)	0.01293 (9)
P2	0.62248 (4)	0.41286 (4)	0.92906 (2)	0.01280 (8)
P3	0.76944 (4)	0.11940 (4)	0.72480 (2)	0.01326 (8)
O1	0.33318 (11)	0.30643 (11)	0.81087 (6)	0.0176 (2)
O2	0.53020 (11)	0.52545 (11)	0.91384 (6)	0.0173 (2)
O3	0.90964 (12)	0.12698 (12)	0.76149 (6)	0.0211 (3)
O4	0.11458 (18)	0.14636 (18)	0.87526 (9)	0.0428 (4)
H4C	0.172 (3)	0.187 (2)	0.8590 (13)	0.040 (7)*
H4D	0.049 (3)	0.140 (2)	0.8426 (15)	0.059 (8)*
C1	0.66847 (15)	0.37270 (15)	0.77288 (8)	0.0128 (3)
C2	0.54549 (15)	0.46501 (15)	0.75384 (8)	0.0127 (3)
H2A	0.5649	0.5027	0.7047	0.015*
H2B	0.5478	0.5364	0.7900	0.015*
C3	0.64933 (16)	0.30879 (15)	0.84973 (8)	0.0127 (3)
H3A	0.7333	0.2555	0.8597	0.015*
H3B	0.5681	0.2497	0.8465	0.015*
C4	0.67527 (16)	0.26866 (15)	0.71198 (8)	0.0138 (3)
H4A	0.7151	0.3103	0.6675	0.017*
H4B	0.5772	0.2456	0.6996	0.017*
C5	0.80796 (16)	0.44960 (16)	0.77433 (9)	0.0152 (3)
H5A	0.8074	0.5055	0.8186	0.018*
H5B	0.8860	0.3877	0.7804	0.018*
C6	0.83946 (18)	0.53319 (17)	0.70727 (9)	0.0214 (4)
H6A	0.9297	0.5768	0.7144	0.032*
H6B	0.7651	0.5974	0.7013	0.032*
H6C	0.8439	0.4792	0.6630	0.032*
C11	0.33661 (16)	0.33075 (16)	0.66157 (8)	0.0137 (3)
C12	0.40089 (16)	0.37265 (16)	0.59636 (9)	0.0169 (3)
H12	0.4595	0.4465	0.5971	0.020*
C13	0.37941 (17)	0.30679 (17)	0.53082 (9)	0.0195 (3)
H13	0.4229	0.3359	0.4867	0.023*
C14	0.29503 (17)	0.19891 (17)	0.52929 (9)	0.0202 (4)
H14	0.2823	0.1528	0.4845	0.024*
C15	0.22902 (18)	0.15812 (17)	0.59328 (9)	0.0216 (4)
H15	0.1695	0.0849	0.5919	0.026*
C16	0.24919 (17)	0.22344 (17)	0.65929 (9)	0.0179 (3)
H16	0.2034	0.1950	0.7029	0.021*
C21	0.25877 (16)	0.54402 (16)	0.75900 (9)	0.0169 (3)
C22	0.19743 (17)	0.57038 (19)	0.82698 (10)	0.0232 (4)
H22	0.2074	0.5112	0.8666	0.028*

C23	0.12141 (18)	0.6833 (2)	0.83705 (11)	0.0307 (5)
H23	0.0801	0.7014	0.8836	0.037*
C24	0.10612 (18)	0.76866 (19)	0.77967 (12)	0.0297 (4)
H24	0.0555	0.8463	0.7868	0.036*
C25	0.16454 (18)	0.74143 (19)	0.71131 (11)	0.0270 (4)
H25	0.1523	0.7999	0.6715	0.032*
C26	0.24067 (17)	0.62945 (17)	0.70078 (10)	0.0211 (4)
H26	0.2804	0.6112	0.6538	0.025*
C31	0.55012 (16)	0.30556 (16)	0.99754 (8)	0.0154 (3)
C32	0.42571 (17)	0.24121 (17)	0.98061 (9)	0.0192 (3)
H32	0.3829	0.2532	0.9336	0.023*
C33	0.36416 (18)	0.16000 (17)	1.03175 (9)	0.0218 (4)
H33	0.2799	0.1158	1.0197	0.026*
C34	0.42611 (18)	0.14341 (17)	1.10081 (9)	0.0206 (4)
H34	0.3849	0.0866	1.1356	0.025*
C35	0.54691 (17)	0.20900 (17)	1.11887 (9)	0.0190 (3)
H35	0.5871	0.1993	1.1667	0.023*
C36	0.61033 (17)	0.28930 (17)	1.06753 (9)	0.0179 (3)
H36	0.6946	0.3332	1.0799	0.022*
C41	0.79016 (16)	0.46439 (16)	0.96587 (8)	0.0159 (3)
C42	0.79740 (17)	0.58912 (17)	0.99403 (9)	0.0189 (3)
H42	0.7180	0.6441	0.9909	0.023*
C43	0.92054 (19)	0.63294 (18)	1.02658 (10)	0.0241 (4)
H43	0.9257	0.7178	1.0458	0.029*
C44	1.03596 (19)	0.55195 (19)	1.03080 (10)	0.0258 (4)
H44	1.1198	0.5814	1.0538	0.031*
C45	1.03050 (18)	0.42938 (19)	1.00206 (10)	0.0253 (4)
H45	1.1105	0.3750	1.0048	0.030*
C46	0.90713 (17)	0.38523 (17)	0.96887 (9)	0.0210 (4)
H46	0.9033	0.3012	0.9484	0.025*
C51	0.78361 (16)	0.05900 (15)	0.63142 (9)	0.0146 (3)
C52	0.90304 (17)	−0.01057 (17)	0.61250 (10)	0.0215 (4)
H52	0.9718	−0.0288	0.6491	0.026*
C53	0.92244 (19)	−0.05350 (19)	0.54055 (11)	0.0275 (4)
H53	1.0043	−0.1009	0.5281	0.033*
C54	0.82304 (19)	−0.02736 (18)	0.48714 (10)	0.0249 (4)
H54	0.8379	−0.0545	0.4376	0.030*
C55	0.70208 (19)	0.03810 (17)	0.50538 (10)	0.0225 (4)
H55	0.6325	0.0538	0.4688	0.027*
C56	0.68198 (17)	0.08104 (16)	0.57717 (9)	0.0188 (3)
H56	0.5984	0.1258	0.5895	0.023*
C61	0.65514 (16)	0.00712 (15)	0.77204 (8)	0.0137 (3)
C62	0.71799 (17)	−0.10455 (16)	0.79916 (9)	0.0181 (3)
H62	0.8166	−0.1155	0.7952	0.022*
C63	0.63681 (19)	−0.19994 (17)	0.83196 (9)	0.0213 (4)
H63	0.6800	−0.2764	0.8496	0.026*
C64	0.4936 (2)	−0.18373 (15)	0.83895 (10)	0.0213 (3)
H64	0.4383	−0.2491	0.8612	0.026*
C65	0.43039 (18)	−0.07184 (17)	0.81350 (9)	0.0212 (4)

H65	0.3322	−0.0598	0.8193	0.025*
C66	0.51071 (16)	0.02237 (16)	0.77960 (9)	0.0168 (3)
H66	0.4668	0.0980	0.7614	0.020*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.01193 (19)	0.0149 (2)	0.01195 (19)	−0.00107 (16)	−0.00066 (14)	0.00143 (16)
P2	0.01457 (19)	0.0123 (2)	0.01157 (19)	0.00009 (16)	−0.00123 (14)	−0.00020 (16)
P3	0.01216 (18)	0.0129 (2)	0.01469 (19)	−0.00004 (15)	−0.00148 (14)	−0.00014 (16)
O1	0.0176 (6)	0.0211 (7)	0.0140 (5)	−0.0026 (5)	−0.0015 (4)	0.0043 (5)
O2	0.0202 (6)	0.0154 (6)	0.0164 (6)	0.0038 (5)	−0.0008 (4)	0.0006 (4)
O3	0.0153 (5)	0.0230 (7)	0.0251 (6)	−0.0006 (5)	−0.0064 (5)	−0.0005 (5)
O4	0.0309 (8)	0.0641 (12)	0.0334 (9)	−0.0231 (8)	−0.0093 (7)	0.0192 (8)
C1	0.0133 (7)	0.0118 (8)	0.0133 (7)	−0.0010 (6)	−0.0012 (5)	0.0013 (6)
C2	0.0129 (7)	0.0130 (8)	0.0121 (7)	−0.0026 (6)	−0.0013 (5)	0.0011 (6)
C3	0.0136 (7)	0.0121 (8)	0.0125 (7)	−0.0006 (6)	−0.0020 (5)	0.0000 (6)
C4	0.0139 (7)	0.0141 (8)	0.0135 (7)	−0.0015 (6)	−0.0002 (6)	0.0003 (6)
C5	0.0142 (7)	0.0143 (8)	0.0170 (8)	−0.0022 (6)	−0.0004 (6)	0.0005 (6)
C6	0.0200 (8)	0.0240 (9)	0.0203 (8)	−0.0091 (7)	0.0018 (6)	0.0010 (7)
C11	0.0121 (7)	0.0160 (8)	0.0130 (7)	0.0023 (6)	−0.0015 (5)	0.0007 (6)
C12	0.0147 (7)	0.0167 (9)	0.0194 (8)	−0.0008 (6)	−0.0026 (6)	0.0032 (7)
C13	0.0189 (8)	0.0239 (9)	0.0155 (8)	0.0004 (7)	0.0016 (6)	0.0006 (7)
C14	0.0213 (8)	0.0234 (9)	0.0159 (8)	0.0010 (7)	−0.0034 (6)	−0.0029 (7)
C15	0.0227 (9)	0.0188 (9)	0.0232 (9)	−0.0069 (7)	−0.0022 (7)	0.0002 (7)
C16	0.0172 (8)	0.0189 (9)	0.0176 (8)	−0.0031 (7)	−0.0008 (6)	0.0032 (6)
C21	0.0118 (7)	0.0166 (8)	0.0224 (8)	−0.0008 (6)	−0.0026 (6)	−0.0008 (7)
C22	0.0184 (8)	0.0286 (10)	0.0227 (9)	0.0035 (7)	0.0008 (7)	−0.0013 (7)
C23	0.0200 (9)	0.0376 (12)	0.0344 (11)	0.0041 (8)	0.0027 (8)	−0.0157 (9)
C24	0.0133 (8)	0.0234 (10)	0.0523 (13)	0.0019 (7)	0.0007 (8)	−0.0078 (9)
C25	0.0173 (8)	0.0216 (10)	0.0420 (11)	0.0020 (7)	0.0023 (8)	0.0046 (8)
C26	0.0151 (8)	0.0224 (9)	0.0258 (9)	0.0008 (7)	0.0022 (6)	0.0030 (7)
C31	0.0174 (8)	0.0151 (8)	0.0138 (8)	0.0022 (6)	0.0002 (6)	0.0005 (6)
C32	0.0180 (8)	0.0259 (10)	0.0137 (8)	0.0004 (7)	−0.0014 (6)	0.0025 (7)
C33	0.0184 (8)	0.0250 (10)	0.0221 (9)	−0.0032 (7)	0.0020 (7)	0.0042 (7)
C34	0.0232 (9)	0.0207 (9)	0.0180 (8)	0.0023 (7)	0.0054 (7)	0.0047 (7)
C35	0.0274 (9)	0.0179 (9)	0.0119 (7)	0.0045 (7)	−0.0013 (6)	0.0016 (6)
C36	0.0226 (8)	0.0165 (9)	0.0147 (8)	0.0014 (7)	−0.0024 (6)	−0.0022 (6)
C41	0.0180 (8)	0.0183 (9)	0.0115 (7)	−0.0042 (6)	−0.0018 (6)	0.0017 (6)
C42	0.0212 (8)	0.0194 (9)	0.0161 (8)	−0.0035 (7)	−0.0012 (6)	−0.0010 (7)
C43	0.0265 (9)	0.0232 (10)	0.0227 (9)	−0.0104 (7)	−0.0010 (7)	−0.0047 (7)
C44	0.0230 (9)	0.0327 (11)	0.0216 (9)	−0.0121 (8)	−0.0053 (7)	0.0020 (8)
C45	0.0188 (8)	0.0296 (11)	0.0275 (10)	0.0002 (7)	−0.0038 (7)	0.0047 (8)
C46	0.0228 (8)	0.0185 (9)	0.0217 (9)	−0.0016 (7)	−0.0044 (7)	0.0005 (7)
C51	0.0157 (7)	0.0120 (8)	0.0162 (8)	−0.0014 (6)	0.0025 (6)	0.0000 (6)
C52	0.0145 (8)	0.0217 (9)	0.0282 (10)	−0.0007 (7)	−0.0013 (7)	−0.0047 (7)
C53	0.0207 (9)	0.0272 (11)	0.0345 (10)	0.0012 (8)	0.0074 (7)	−0.0107 (8)
C54	0.0314 (10)	0.0235 (10)	0.0198 (9)	−0.0066 (8)	0.0079 (7)	−0.0066 (7)

C55	0.0307 (10)	0.0175 (9)	0.0193 (9)	−0.0016 (7)	−0.0027 (7)	0.0019 (7)
C56	0.0211 (8)	0.0177 (9)	0.0178 (8)	0.0036 (7)	0.0000 (6)	0.0005 (7)
C61	0.0194 (8)	0.0117 (8)	0.0098 (7)	−0.0026 (6)	−0.0019 (6)	−0.0024 (6)
C62	0.0208 (8)	0.0180 (9)	0.0156 (8)	0.0021 (7)	−0.0023 (6)	−0.0010 (6)
C63	0.0358 (10)	0.0137 (8)	0.0142 (8)	0.0023 (7)	−0.0027 (7)	0.0003 (6)
C64	0.0332 (9)	0.0159 (8)	0.0148 (7)	−0.0083 (8)	0.0016 (6)	0.0006 (8)
C65	0.0198 (8)	0.0222 (9)	0.0216 (9)	−0.0063 (7)	0.0013 (6)	−0.0011 (7)
C66	0.0178 (8)	0.0143 (8)	0.0183 (8)	0.0000 (6)	−0.0033 (6)	0.0000 (6)

Geometric parameters (Å, °)

P1—O1	1.4927 (12)	C24—H24	0.9500
P1—C21	1.8057 (17)	C25—C26	1.386 (2)
P1—C11	1.8175 (16)	C25—H25	0.9500
P1—C2	1.8257 (15)	C26—H26	0.9500
P2—O2	1.4899 (12)	C31—C32	1.395 (2)
P2—C31	1.8060 (16)	C31—C36	1.399 (2)
P2—C41	1.8109 (16)	C32—C33	1.385 (2)
P2—C3	1.8165 (16)	C32—H32	0.9500
P3—O3	1.4926 (12)	C33—C34	1.392 (2)
P3—C4	1.8081 (16)	C33—H33	0.9500
P3—C51	1.8081 (16)	C34—C35	1.377 (2)
P3—C61	1.8128 (16)	C34—H34	0.9500
O4—H4C	0.75 (3)	C35—C36	1.389 (2)
O4—H4D	0.86 (3)	C35—H35	0.9500
C1—C2	1.553 (2)	C36—H36	0.9500
C1—C3	1.553 (2)	C41—C46	1.387 (2)
C1—C4	1.546 (2)	C41—C42	1.396 (2)
C1—C5	1.552 (2)	C42—C43	1.389 (2)
C2—H2A	0.9900	C42—H42	0.9500
C2—H2B	0.9900	C43—C44	1.388 (3)
C3—H3A	0.9900	C43—H43	0.9500
C3—H3B	0.9900	C44—C45	1.378 (3)
C4—H4A	0.9900	C44—H44	0.9500
C4—H4B	0.9900	C45—C46	1.397 (2)
C5—C6	1.523 (2)	C45—H45	0.9500
C5—H5A	0.9900	C46—H46	0.9500
C5—H5B	0.9900	C51—C52	1.392 (2)
C6—H6A	0.9800	C51—C56	1.396 (2)
C6—H6B	0.9800	C52—C53	1.389 (2)
C6—H6C	0.9800	C52—H52	0.9500
C11—C16	1.393 (2)	C53—C54	1.379 (3)
C11—C12	1.400 (2)	C53—H53	0.9500
C12—C13	1.384 (2)	C54—C55	1.380 (3)
C12—H12	0.9500	C54—H54	0.9500
C13—C14	1.381 (2)	C55—C56	1.387 (2)
C13—H13	0.9500	C55—H55	0.9500
C14—C15	1.386 (2)	C56—H56	0.9500
C14—H14	0.9500	C61—C66	1.393 (2)

C15—C16	1.387 (2)	C61—C62	1.396 (2)
C15—H15	0.9500	C62—C63	1.392 (2)
C16—H16	0.9500	C62—H62	0.9500
C21—C26	1.389 (2)	C63—C64	1.382 (3)
C21—C22	1.391 (2)	C63—H63	0.9500
C22—C23	1.393 (3)	C64—C65	1.389 (2)
C22—H22	0.9500	C64—H64	0.9500
C23—C24	1.373 (3)	C65—C66	1.387 (2)
C23—H23	0.9500	C65—H65	0.9500
C24—C25	1.387 (3)	C66—H66	0.9500
O1—P1—C2	115.31 (7)	C23—C24—C25	120.02 (18)
O1—P1—C11	109.75 (7)	C23—C24—H24	120.0
O1—P1—C21	111.79 (7)	C25—C24—H24	120.0
C2—P1—C11	107.97 (7)	C26—C25—C24	120.38 (18)
C2—P1—C21	103.67 (7)	C26—C25—H25	119.8
C11—P1—C21	107.96 (7)	C24—C25—H25	119.8
O2—P2—C3	114.00 (7)	C25—C26—C21	119.85 (16)
O2—P2—C31	112.73 (7)	C25—C26—H26	120.1
O2—P2—C41	110.81 (7)	C21—C26—H26	120.1
C3—P2—C31	103.21 (7)	C32—C31—C36	119.16 (15)
C3—P2—C41	109.94 (7)	C32—C31—P2	118.14 (12)
C31—P2—C41	105.59 (7)	C36—C31—P2	122.64 (13)
O3—P3—C4	117.08 (7)	C33—C32—C31	120.48 (15)
O3—P3—C51	111.42 (7)	C33—C32—H32	119.8
O3—P3—C61	111.26 (7)	C31—C32—H32	119.8
C4—P3—C51	102.51 (7)	C32—C33—C34	119.76 (16)
C4—P3—C61	108.37 (7)	C32—C33—H33	120.1
C51—P3—C61	105.26 (7)	C34—C33—H33	120.1
H4C—O4—H4D	107 (3)	C35—C34—C33	120.29 (15)
C2—C1—C3	111.91 (12)	C35—C34—H34	119.9
C2—C1—C4	107.92 (12)	C33—C34—H34	119.9
C2—C1—C5	109.33 (12)	C34—C35—C36	120.28 (15)
C3—C1—C4	110.11 (12)	C34—C35—H35	119.9
C3—C1—C5	107.95 (12)	C36—C35—H35	119.9
C4—C1—C5	109.60 (12)	C35—C36—C31	119.99 (15)
C1—C2—P1	119.05 (11)	C35—C36—H36	120.0
C1—C2—H2A	107.6	C31—C36—H36	120.0
P1—C2—H2A	107.6	C46—C41—C42	119.91 (15)
C1—C2—H2B	107.6	C46—C41—P2	123.17 (13)
P1—C2—H2B	107.6	C42—C41—P2	116.89 (13)
H2A—C2—H2B	107.0	C43—C42—C41	120.06 (16)
C1—C3—P2	118.01 (11)	C43—C42—H42	120.0
C1—C3—H3A	107.8	C41—C42—H42	120.0
P2—C3—H3A	107.8	C44—C43—C42	119.54 (17)
C1—C3—H3B	107.8	C44—C43—H43	120.2
P2—C3—H3B	107.8	C42—C43—H43	120.2
H3A—C3—H3B	107.1	C45—C44—C43	120.79 (17)
C1—C4—P3	122.10 (11)	C45—C44—H44	119.6
C1—C4—H4A	106.8	C43—C44—H44	119.6

P3—C4—H4A	106.8	C44—C45—C46	119.84 (17)
C1—C4—H4B	106.8	C44—C45—H45	120.1
P3—C4—H4B	106.8	C46—C45—H45	120.1
H4A—C4—H4B	106.7	C41—C46—C45	119.82 (17)
C6—C5—C1	116.83 (13)	C41—C46—H46	120.1
C6—C5—H5A	108.1	C45—C46—H46	120.1
C1—C5—H5A	108.1	C52—C51—C56	118.58 (15)
C6—C5—H5B	108.1	C52—C51—P3	118.26 (12)
C1—C5—H5B	108.1	C56—C51—P3	123.14 (12)
H5A—C5—H5B	107.3	C53—C52—C51	120.56 (16)
C5—C6—H6A	109.5	C53—C52—H52	119.7
C5—C6—H6B	109.5	C51—C52—H52	119.7
H6A—C6—H6B	109.5	C54—C53—C52	120.05 (16)
C5—C6—H6C	109.5	C54—C53—H53	120.0
H6A—C6—H6C	109.5	C52—C53—H53	120.0
H6B—C6—H6C	109.5	C53—C54—C55	120.18 (17)
C16—C11—C12	119.16 (15)	C53—C54—H54	119.9
C16—C11—P1	116.50 (12)	C55—C54—H54	119.9
C12—C11—P1	124.26 (12)	C54—C55—C56	120.02 (17)
C13—C12—C11	120.24 (15)	C54—C55—H55	120.0
C13—C12—H12	119.9	C56—C55—H55	120.0
C11—C12—H12	119.9	C55—C56—C51	120.55 (16)
C14—C13—C12	120.28 (15)	C55—C56—H56	119.7
C14—C13—H13	119.9	C51—C56—H56	119.7
C12—C13—H13	119.9	C66—C61—C62	118.93 (15)
C13—C14—C15	119.85 (16)	C66—C61—P3	124.63 (12)
C13—C14—H14	120.1	C62—C61—P3	116.37 (12)
C15—C14—H14	120.1	C63—C62—C61	120.32 (15)
C14—C15—C16	120.48 (16)	C63—C62—H62	119.8
C14—C15—H15	119.8	C61—C62—H62	119.8
C16—C15—H15	119.8	C64—C63—C62	120.15 (16)
C15—C16—C11	119.97 (15)	C64—C63—H63	119.9
C15—C16—H16	120.0	C62—C63—H63	119.9
C11—C16—H16	120.0	C63—C64—C65	119.98 (16)
C26—C21—C22	119.55 (16)	C63—C64—H64	120.0
C26—C21—P1	122.68 (13)	C65—C64—H64	120.0
C22—C21—P1	117.70 (13)	C66—C65—C64	119.96 (16)
C21—C22—C23	120.14 (18)	C66—C65—H65	120.0
C21—C22—H22	119.9	C64—C65—H65	120.0
C23—C22—H22	119.9	C65—C66—C61	120.63 (15)
C24—C23—C22	120.03 (17)	C65—C66—H66	119.7
C24—C23—H23	120.0	C61—C66—H66	119.7
C22—C23—H23	120.0		
C4—C1—C2—P1	63.32 (15)	C41—P2—C31—C36	8.84 (16)
C5—C1—C2—P1	−177.53 (10)	C3—P2—C31—C36	124.26 (14)
C3—C1—C2—P1	−57.98 (15)	C36—C31—C32—C33	−1.4 (2)
O1—P1—C2—C1	38.96 (14)	P2—C31—C32—C33	−178.59 (13)
C21—P1—C2—C1	161.48 (11)	C31—C32—C33—C34	0.6 (3)
C11—P1—C2—C1	−84.16 (12)	C32—C33—C34—C35	1.1 (3)

C4—C1—C3—P2	−174.49 (10)	C33—C34—C35—C36	−2.0 (3)
C5—C1—C3—P2	65.90 (15)	C34—C35—C36—C31	1.1 (2)
C2—C1—C3—P2	−54.46 (16)	C32—C31—C36—C35	0.6 (2)
O2—P2—C3—C1	39.48 (14)	P2—C31—C36—C35	177.59 (12)
C31—P2—C3—C1	162.10 (11)	O2—P2—C41—C46	−164.41 (13)
C41—P2—C3—C1	−85.64 (13)	C31—P2—C41—C46	73.23 (15)
C5—C1—C4—P3	78.24 (15)	C3—P2—C41—C46	−37.48 (16)
C3—C1—C4—P3	−40.37 (17)	O2—P2—C41—C42	17.44 (15)
C2—C1—C4—P3	−162.79 (11)	C31—P2—C41—C42	−104.92 (13)
O3—P3—C4—C1	−42.42 (14)	C3—P2—C41—C42	144.37 (12)
C51—P3—C4—C1	−164.67 (12)	C46—C41—C42—C43	−1.5 (2)
C61—P3—C4—C1	84.37 (13)	P2—C41—C42—C43	176.71 (13)
C4—C1—C5—C6	67.01 (17)	C41—C42—C43—C44	0.1 (3)
C3—C1—C5—C6	−173.05 (13)	C42—C43—C44—C45	1.0 (3)
C2—C1—C5—C6	−51.09 (17)	C43—C44—C45—C46	−0.6 (3)
O1—P1—C11—C16	19.90 (15)	C42—C41—C46—C45	1.9 (2)
C21—P1—C11—C16	−102.17 (13)	P2—C41—C46—C45	−176.20 (13)
C2—P1—C11—C16	146.34 (12)	C44—C45—C46—C41	−0.8 (3)
O1—P1—C11—C12	−156.57 (13)	O3—P3—C51—C52	20.61 (15)
C21—P1—C11—C12	81.36 (15)	C4—P3—C51—C52	146.62 (13)
C2—P1—C11—C12	−30.13 (16)	C61—P3—C51—C52	−100.10 (14)
C16—C11—C12—C13	−1.0 (2)	O3—P3—C51—C56	−158.05 (13)
P1—C11—C12—C13	175.35 (12)	C4—P3—C51—C56	−32.04 (15)
C11—C12—C13—C14	−0.3 (2)	C61—P3—C51—C56	81.23 (15)
C12—C13—C14—C15	1.5 (3)	C56—C51—C52—C53	2.0 (3)
C13—C14—C15—C16	−1.3 (3)	P3—C51—C52—C53	−176.72 (14)
C14—C15—C16—C11	−0.1 (3)	C51—C52—C53—C54	0.0 (3)
C12—C11—C16—C15	1.3 (2)	C52—C53—C54—C55	−2.0 (3)
P1—C11—C16—C15	−175.39 (13)	C53—C54—C55—C56	1.8 (3)
O1—P1—C21—C26	−161.46 (13)	C54—C55—C56—C51	0.2 (3)
C11—P1—C21—C26	−40.65 (16)	C52—C51—C56—C55	−2.1 (2)
C2—P1—C21—C26	73.72 (15)	P3—C51—C56—C55	176.55 (13)
O1—P1—C21—C22	21.58 (15)	O3—P3—C61—C66	144.99 (13)
C11—P1—C21—C22	142.39 (13)	C4—P3—C61—C66	14.91 (15)
C2—P1—C21—C22	−103.24 (14)	C51—P3—C61—C66	−94.19 (14)
C26—C21—C22—C23	−1.6 (3)	O3—P3—C61—C62	−38.16 (14)
P1—C21—C22—C23	175.42 (13)	C4—P3—C61—C62	−168.25 (12)
C21—C22—C23—C24	0.4 (3)	C51—P3—C61—C62	82.65 (13)
C22—C23—C24—C25	1.0 (3)	C66—C61—C62—C63	1.2 (2)
C23—C24—C25—C26	−1.2 (3)	P3—C61—C62—C63	−175.86 (12)
C24—C25—C26—C21	−0.1 (3)	C61—C62—C63—C64	−1.0 (2)
C22—C21—C26—C25	1.5 (2)	C62—C63—C64—C65	−0.3 (2)
P1—C21—C26—C25	−175.43 (13)	C63—C64—C65—C66	1.4 (3)
O2—P2—C31—C32	64.77 (15)	C64—C65—C66—C61	−1.3 (2)
C41—P2—C31—C32	−174.11 (13)	C62—C61—C66—C65	0.0 (2)
C3—P2—C31—C32	−58.69 (14)	P3—C61—C66—C65	176.74 (13)
O2—P2—C31—C36	−112.27 (14)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4C···O1	0.75 (3)	2.16 (3)	2.912 (2)	179 (3)
O4—H4D···O3^{i}	0.86 (3)	1.98 (3)	2.8413 (19)	176 (3)

Symmetry codes: (i) *x*−1, *y*, *z*.

Fig. 1

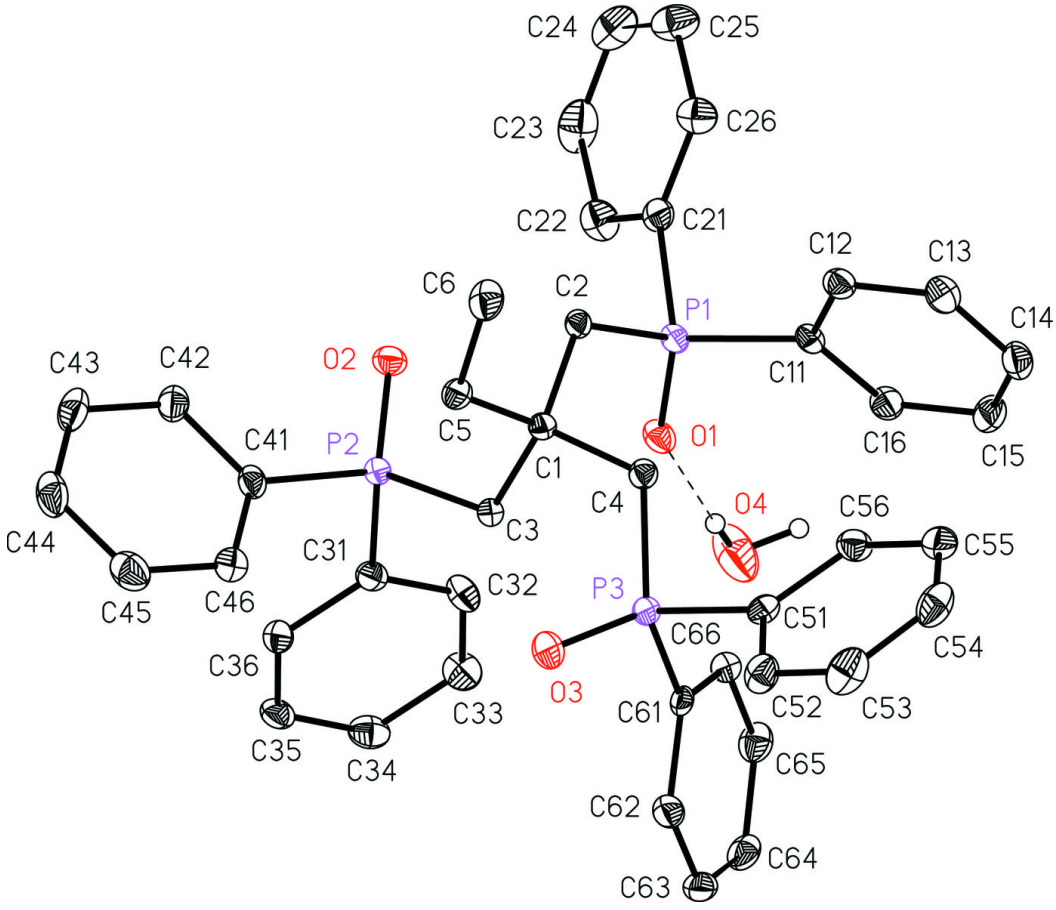

C24
C25
C23
C26
C22
C21
C12
C13
C6
C2
P1
C14
C43
C42
O2
C11
C5
O1
C41
P2
C16
C15
C1
C4
O4
C56
C44
C3
C55
C31
C32
P3
C45
C46
C51
C66
C54
C36
O3
C52
C53
C33
C61
C35
C34
C65
C62
C64
C63

Fig. 2