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6,6′-[(3,3′-Di-*tert*-butyl-5,5′-dimethoxy-1,1′-biphenyl-2,2′-diyl)bis(oxy)]bis(dibenzo[*d*,*f*][1,3,2]-dioxaphosphepine) benzene monosolvate

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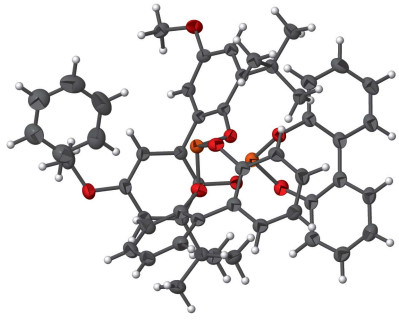
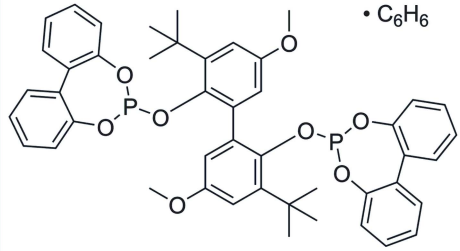
Structural data: full structural data are available from iucrdata.iucr.org

The crystal structure of the benzene monosolvate of the well known organic diphosphite ligand BIPHEPHOS, $C_{46}H_{44}O_8P_2\cdot C_6H_6$, is reported for the first time. Single crystals of BIPHEPHOS were obtained from a benzene solution after layering with *n*-heptane at room temperature. One specific property of this type of diphosphite structure is the twisting of the biphenyl units. In the crystal, C—H$\cdots\pi$ contacts and π–π stacking interactions [centroid-to-centroid distance = 3.8941 (15) Å] are observed.

3D view



Chemical scheme



Structure description

BIPHEPHOS is a very prominent organic diphosphite ligand applied as co-catalyst in the field of highly *n*-regioselective rhodium-catalysed alkene hydroformylation (Börner & Franke, 2016). Atropisomerism of the dibenzo[*d*,*f*][1,3,2]dioxaphosphepine moiety in related diphosphites has been studied in solution and by DFT methods (Whiteker *et al.*, 1995, Briggs & Whiteker, 2001, Franke *et al.*, 2003). Interestingly, until now no crystal structure of BIPHEPHOS has been reported. Crystal structures have been published for similar diphosphites containing the same dibenzo[*d*,*f*][1,3,2]dioxaphosphepine unit but with the unsubstituted 2,2′-dihydroxybiphenyl and the 2,2′-dihydroxy-3,3′,5,5′-tetra(*t*-butyl)biphenyl backbone (Baker *et al.*, 1991; Meyer *et al.*, 1993; Hao *et al.*, 2012; Liu *et al.*, 2014). The common feature of these types of ligands is the twisting about the biphenyl linkages. For the title compound (Fig. 1), the dihedral angle about the biphenyl axis of the backbone between the C1–C6 and C7–C12 planes is 61.79 (10)°. Within the seven-

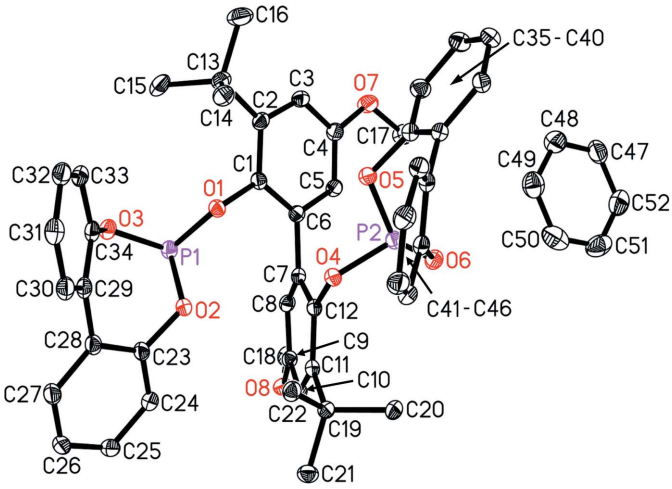


Figure 1
The molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level. Hydrogen atoms are omitted for clarity.

membered rings, the dihedral angles about the biphenyl units are 43.83 (11)° for C2–C28/C29–C34 and 41.78 (11)° for C35–C40/C41–C46.

Crystals of the title compound contain co-crystallized solvent (one benzene per one molecule diphosphite), which interacts with the ligand *via* C—H···π interactions (Table 1). In the crystal, centrosymmetrically related molecules are linked by a π–π stacking interaction involving the C23–C28 rings with a centroid-to-centroid distance of 3.8941 (15) Å.

Synthesis and crystallization

The diphosphite was provided by Evonik Performance Materials GmbH (OxoPhos® 17). A solution of $C_{46}H_{44}O_8P_2$ (0.08 mmol, 0.063 g) in benzene (2 ml) was prepared under argon using standard Schlenk techniques. The solution was brought to 50°C for dissolving the solid material. At room temperature the solution was then layered with *ca* 0.4 ml of *n*-heptane. Crystallization as fine needles took place at room temperature after several days.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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

*Cg*1 is the centroid of the C47–C52 benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C39—H39···*Cg*1^{i}	0.95	2.75	3.678 (3)	165

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{46}H_{44}O_8P_2{\cdot}C_6H_6$
M_r	864.86
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	150
a, b, c (Å)	25.1072 (5), 8.8634 (2), 21.3401 (4)
β (°)	112.4441 (12)
V (Å^3)	4389.20 (16)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	1.36
Crystal size (mm)	0.34 × 0.04 × 0.04
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2014)
T_{min}, T_{max}	0.80, 0.95
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	41898, 7745, 5805
R_{int}	0.067
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.040, 0.107, 1.01
No. of reflections	7745
No. of parameters	567
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.25, −0.34

Computer programs: *APEX2* (Bruker, 2014), *SAINT* (Bruker, 2013), *XP* in *SHELXTL* and *SHELXS97* (Sheldrick, 2008), *SHELXL2014/7* (Sheldrick, 2015) and *publCIF* (Westrip, 2010).

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full crystallographic data

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Crystal data

$C_{46}H_{44}O_8P_2·C_6H_6$
M_r = 864.86
Monoclinic, $P2_1/c$
a = 25.1072 (5) Å
b = 8.8634 (2) Å
c = 21.3401 (4) Å
β = 112.4441 (12)°
V = 4389.20 (16) Å^3
Z = 4
$F(000)$ = 1824
D_x = 1.309 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 4431 reflections
θ = 3.8–66.5°
μ = 1.36 mm^{-1}
T = 150 K
Needle, colourless
0.34 × 0.04 × 0.04 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: microfocus
Multilayer monochromator
Detector resolution: 8.3333 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2014)
T_{min} = 0.80, T_{max} = 0.95
41898 measured reflections
7745 independent reflections
5805 reflections with $I > 2\sigma(I)$
R_{int} = 0.067
θ_{max} = 66.7°, θ_{min} = 1.9°
h = −29→29
k = −10→9
l = −25→25

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.107
S = 1.01
7745 reflections
567 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0532P)^2 + 0.4402P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Special details

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

Refinement. All H atoms were placed geometrically and refined using a riding atom approximation, with C–H = 0.95–0.98 Å, and with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C) for methyl H atoms. A rotating model was used for the methyl groups.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.21470 (8)	0.4417 (2)	0.51667 (10)	0.0260 (4)
C2	0.15970 (9)	0.3898 (3)	0.47514 (10)	0.0296 (4)
C3	0.11333 (9)	0.4728 (3)	0.47748 (10)	0.0318 (4)
H3	0.0754	0.4417	0.4499	0.038*
C4	0.12035 (9)	0.5992 (3)	0.51846 (10)	0.0318 (4)
C5	0.17481 (9)	0.6423 (2)	0.56151 (10)	0.0290 (4)
H5	0.1798	0.7261	0.5909	0.035*
C6	0.22288 (8)	0.5612 (2)	0.56154 (9)	0.0260 (4)
C7	0.28090 (8)	0.6109 (2)	0.61018 (9)	0.0257 (4)
C8	0.29895 (9)	0.7562 (2)	0.60285 (9)	0.0275 (4)
H8	0.2752	0.8192	0.5671	0.033*
C9	0.35139 (9)	0.8083 (2)	0.64762 (10)	0.0271 (4)
C10	0.38784 (8)	0.7113 (2)	0.69685 (10)	0.0274 (4)
H10	0.4253	0.7457	0.7246	0.033*
C11	0.37152 (8)	0.5665 (2)	0.70686 (9)	0.0255 (4)
C12	0.31566 (8)	0.5203 (2)	0.66410 (9)	0.0248 (4)
C13	0.14907 (9)	0.2501 (3)	0.42951 (11)	0.0357 (5)
C14	0.18159 (10)	0.1141 (3)	0.47133 (12)	0.0382 (5)
H14A	0.2231	0.1340	0.4890	0.057*
H14B	0.1731	0.0241	0.4425	0.057*
H14C	0.1693	0.0974	0.5092	0.057*
C15	0.16772 (11)	0.2851 (3)	0.37019 (11)	0.0430 (6)
H15A	0.1452	0.3702	0.3440	0.065*
H15B	0.1611	0.1962	0.3408	0.065*
H15C	0.2088	0.3110	0.3880	0.065*
C16	0.08521 (11)	0.2061 (3)	0.39926 (15)	0.0552 (7)
H16A	0.0713	0.1908	0.4359	0.083*
H16B	0.0806	0.1125	0.3732	0.083*
H16C	0.0630	0.2868	0.3693	0.083*
C17	0.07469 (11)	0.7922 (3)	0.55768 (15)	0.0536 (7)
H17A	0.0915	0.7573	0.6048	0.080*
H17B	0.0364	0.8344	0.5484	0.080*
H17C	0.0995	0.8700	0.5504	0.080*
C18	0.33003 (10)	1.0591 (2)	0.60870 (11)	0.0333 (5)
H18A	0.3148	1.0291	0.5609	0.050*
H18B	0.3483	1.1585	0.6135	0.050*
H18C	0.2985	1.0639	0.6250	0.050*
C19	0.41356 (8)	0.4679 (2)	0.76405 (10)	0.0278 (4)
C20	0.39320 (9)	0.4608 (3)	0.82366 (10)	0.0317 (4)
H20A	0.3545	0.4168	0.8080	0.048*
H20B	0.3923	0.5629	0.8409	0.048*
H20C	0.4199	0.3982	0.8599	0.048*

C21	0.47487 (9)	0.5340 (3)	0.79171 (11)	0.0360 (5)
H21A	0.5011	0.4640	0.8247	0.054*
H21B	0.4748	0.6310	0.8138	0.054*
H21C	0.4878	0.5492	0.7542	0.054*
C22	0.41810 (9)	0.3085 (3)	0.73876 (10)	0.0317 (4)
H22A	0.4303	0.3145	0.7003	0.048*
H22B	0.3805	0.2587	0.7244	0.048*
H22C	0.4465	0.2503	0.7754	0.048*
C23	0.42077 (9)	0.4076 (3)	0.56208 (10)	0.0300 (4)
C24	0.45812 (9)	0.5236 (3)	0.59366 (10)	0.0343 (5)
H24	0.4453	0.6070	0.6121	0.041*
C25	0.51494 (10)	0.5166 (3)	0.59814 (11)	0.0397 (5)
H25	0.5411	0.5958	0.6194	0.048*
C26	0.53311 (9)	0.3941 (3)	0.57156 (11)	0.0408 (6)
H26	0.5718	0.3895	0.5746	0.049*
C27	0.49539 (9)	0.2779 (3)	0.54057 (11)	0.0368 (5)
H27	0.5086	0.1939	0.5229	0.044*
C28	0.43789 (9)	0.2825 (3)	0.53483 (10)	0.0310 (4)
C29	0.39772 (9)	0.1581 (3)	0.50225 (10)	0.0309 (4)
C30	0.41526 (10)	0.0076 (3)	0.51498 (11)	0.0352 (5)
H30	0.4534	−0.0142	0.5456	0.042*
C31	0.37855 (10)	−0.1101 (3)	0.48411 (11)	0.0385 (5)
H31	0.3912	−0.2114	0.4946	0.046*
C32	0.32335 (10)	−0.0804 (3)	0.43777 (11)	0.0375 (5)
H32	0.2984	−0.1613	0.4158	0.045*
C33	0.30467 (9)	0.0682 (3)	0.42360 (10)	0.0332 (5)
H33	0.2672	0.0895	0.3912	0.040*
C34	0.34123 (9)	0.1848 (3)	0.45714 (10)	0.0293 (4)
C35	0.15006 (9)	0.2095 (3)	0.64810 (10)	0.0298 (4)
C36	0.09582 (10)	0.2675 (3)	0.61247 (11)	0.0363 (5)
H36	0.0911	0.3526	0.5838	0.044*
C37	0.04799 (10)	0.2002 (3)	0.61888 (12)	0.0401 (5)
H37	0.0105	0.2392	0.5947	0.048*
C38	0.05564 (9)	0.0756 (3)	0.66094 (12)	0.0375 (5)
H38	0.0231	0.0287	0.6651	0.045*
C39	0.11007 (9)	0.0195 (3)	0.69661 (11)	0.0330 (5)
H39	0.1147	−0.0653	0.7254	0.040*
C40	0.15884 (9)	0.0861 (2)	0.69093 (10)	0.0288 (4)
C41	0.21719 (9)	0.0226 (2)	0.72726 (10)	0.0279 (4)
C42	0.22625 (9)	−0.1325 (3)	0.73141 (10)	0.0320 (4)
H42	0.1944	−0.1984	0.7113	0.038*
C43	0.28060 (10)	−0.1929 (3)	0.76411 (11)	0.0369 (5)
H43	0.2859	−0.2992	0.7664	0.044*
C44	0.32739 (10)	−0.0979 (3)	0.79350 (11)	0.0372 (5)
H44	0.3649	−0.1391	0.8151	0.045*
C45	0.31965 (9)	0.0563 (3)	0.79145 (10)	0.0334 (5)
H45	0.3516	0.1213	0.8126	0.040*
C46	0.26507 (9)	0.1158 (2)	0.75833 (9)	0.0284 (4)

C47	0.04143 (12)	0.7196 (4)	0.77065 (14)	0.0537 (7)
H47	0.0019	0.7458	0.7563	0.064*
C48	0.05877 (12)	0.6181 (4)	0.73342 (14)	0.0540 (7)
H48	0.0311	0.5740	0.6937	0.065*
C49	0.11611 (13)	0.5804 (3)	0.75358 (15)	0.0550 (7)
H49	0.1281	0.5103	0.7280	0.066*
C50	0.15594 (13)	0.6450 (4)	0.81117 (17)	0.0627 (9)
H50	0.1956	0.6203	0.8252	0.075*
C51	0.13798 (15)	0.7463 (4)	0.84858 (15)	0.0629 (8)
H51	0.1654	0.7903	0.8885	0.076*
C52	0.08077 (14)	0.7832 (4)	0.82824 (14)	0.0590 (7)
H52	0.0685	0.8524	0.8540	0.071*
O1	0.26355 (6)	0.36536 (16)	0.51544 (7)	0.0273 (3)
O2	0.36542 (6)	0.40936 (17)	0.56232 (7)	0.0294 (3)
O3	0.32105 (6)	0.33231 (17)	0.44072 (7)	0.0309 (3)
O4	0.29329 (6)	0.38073 (16)	0.67427 (7)	0.0265 (3)
O5	0.19750 (6)	0.27103 (17)	0.63783 (7)	0.0309 (3)
O6	0.25769 (6)	0.27110 (17)	0.76141 (7)	0.0310 (3)
O7	0.06992 (7)	0.6688 (2)	0.51339 (8)	0.0427 (4)
O8	0.37168 (7)	0.95099 (17)	0.64772 (7)	0.0337 (3)
P1	0.31057 (2)	0.45307 (6)	0.49248 (2)	0.02799 (13)
P2	0.23811 (2)	0.38617 (6)	0.69709 (3)	0.02787 (13)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0238 (9)	0.0298 (11)	0.0245 (9)	0.0053 (8)	0.0094 (8)	0.0046 (8)
C2	0.0302 (10)	0.0345 (12)	0.0224 (9)	0.0016 (8)	0.0081 (8)	−0.0001 (8)
C3	0.0246 (10)	0.0404 (13)	0.0268 (10)	0.0017 (8)	0.0057 (8)	−0.0021 (8)
C4	0.0266 (10)	0.0397 (13)	0.0273 (10)	0.0085 (9)	0.0083 (8)	0.0012 (9)
C5	0.0301 (10)	0.0300 (11)	0.0259 (9)	0.0030 (8)	0.0096 (8)	−0.0008 (8)
C6	0.0268 (10)	0.0276 (11)	0.0231 (9)	0.0009 (8)	0.0090 (8)	0.0032 (7)
C7	0.0267 (10)	0.0297 (11)	0.0229 (9)	0.0006 (8)	0.0119 (8)	−0.0013 (7)
C8	0.0307 (10)	0.0286 (11)	0.0228 (9)	0.0024 (8)	0.0100 (8)	0.0026 (8)
C9	0.0310 (10)	0.0268 (11)	0.0258 (9)	−0.0033 (8)	0.0132 (8)	−0.0003 (8)
C10	0.0258 (10)	0.0332 (12)	0.0237 (9)	−0.0031 (8)	0.0100 (8)	−0.0029 (8)
C11	0.0263 (10)	0.0294 (11)	0.0241 (9)	0.0004 (8)	0.0133 (8)	−0.0007 (7)
C12	0.0269 (10)	0.0255 (11)	0.0248 (9)	−0.0008 (8)	0.0131 (8)	−0.0010 (7)
C13	0.0310 (11)	0.0398 (13)	0.0325 (10)	0.0000 (9)	0.0079 (9)	−0.0093 (9)
C14	0.0420 (12)	0.0331 (12)	0.0401 (12)	−0.0030 (9)	0.0162 (10)	−0.0069 (9)
C15	0.0471 (14)	0.0492 (15)	0.0258 (10)	0.0085 (11)	0.0061 (10)	−0.0075 (9)
C16	0.0358 (13)	0.0578 (18)	0.0619 (17)	−0.0049 (11)	0.0075 (12)	−0.0290 (14)
C17	0.0395 (14)	0.0598 (18)	0.0567 (15)	0.0138 (12)	0.0130 (12)	−0.0185 (13)
C18	0.0458 (13)	0.0247 (11)	0.0308 (10)	0.0010 (9)	0.0162 (9)	0.0020 (8)
C19	0.0261 (10)	0.0337 (12)	0.0229 (9)	0.0010 (8)	0.0086 (8)	0.0018 (8)
C20	0.0336 (11)	0.0388 (12)	0.0225 (9)	0.0013 (9)	0.0105 (8)	0.0014 (8)
C21	0.0276 (11)	0.0433 (13)	0.0328 (10)	−0.0002 (9)	0.0069 (9)	0.0054 (9)
C22	0.0328 (11)	0.0356 (12)	0.0266 (10)	0.0062 (9)	0.0112 (8)	0.0029 (8)

C23	0.0276 (10)	0.0404 (13)	0.0245 (9)	0.0022 (8)	0.0128 (8)	0.0025 (8)
C24	0.0343 (11)	0.0431 (13)	0.0269 (10)	−0.0024 (9)	0.0133 (9)	−0.0038 (9)
C25	0.0297 (11)	0.0571 (16)	0.0311 (11)	−0.0083 (10)	0.0104 (9)	−0.0037 (10)
C26	0.0265 (11)	0.0679 (17)	0.0294 (10)	−0.0005 (10)	0.0122 (9)	−0.0010 (10)
C27	0.0316 (11)	0.0525 (15)	0.0287 (10)	0.0071 (10)	0.0144 (9)	0.0026 (9)
C28	0.0307 (10)	0.0397 (13)	0.0250 (9)	0.0054 (9)	0.0134 (8)	0.0037 (8)
C29	0.0340 (11)	0.0380 (12)	0.0255 (9)	0.0045 (9)	0.0169 (9)	0.0002 (8)
C30	0.0366 (11)	0.0419 (13)	0.0299 (10)	0.0095 (9)	0.0158 (9)	0.0003 (9)
C31	0.0487 (13)	0.0351 (13)	0.0383 (11)	0.0065 (10)	0.0238 (11)	0.0002 (9)
C32	0.0425 (13)	0.0394 (13)	0.0359 (11)	−0.0028 (10)	0.0207 (10)	−0.0079 (9)
C33	0.0347 (11)	0.0418 (13)	0.0253 (9)	0.0018 (9)	0.0140 (9)	−0.0031 (8)
C34	0.0346 (11)	0.0345 (12)	0.0232 (9)	0.0051 (9)	0.0161 (8)	0.0013 (8)
C35	0.0290 (10)	0.0349 (12)	0.0268 (9)	−0.0040 (8)	0.0121 (8)	−0.0025 (8)
C36	0.0324 (11)	0.0399 (13)	0.0340 (11)	−0.0007 (9)	0.0099 (9)	0.0035 (9)
C37	0.0282 (11)	0.0482 (15)	0.0418 (12)	0.0007 (9)	0.0111 (10)	−0.0012 (10)
C38	0.0291 (11)	0.0461 (14)	0.0403 (11)	−0.0059 (9)	0.0167 (9)	−0.0050 (10)
C39	0.0352 (11)	0.0360 (13)	0.0323 (10)	−0.0048 (9)	0.0179 (9)	−0.0027 (9)
C40	0.0282 (10)	0.0344 (12)	0.0255 (9)	−0.0032 (8)	0.0121 (8)	−0.0046 (8)
C41	0.0311 (10)	0.0327 (12)	0.0239 (9)	−0.0021 (8)	0.0149 (8)	−0.0015 (8)
C42	0.0342 (11)	0.0349 (12)	0.0287 (10)	−0.0028 (9)	0.0139 (9)	−0.0030 (8)
C43	0.0430 (12)	0.0338 (13)	0.0338 (11)	0.0029 (9)	0.0147 (10)	−0.0019 (9)
C44	0.0332 (11)	0.0451 (14)	0.0310 (10)	0.0078 (10)	0.0099 (9)	0.0001 (9)
C45	0.0317 (11)	0.0438 (13)	0.0238 (9)	−0.0033 (9)	0.0095 (8)	−0.0029 (8)
C46	0.0324 (10)	0.0332 (11)	0.0221 (9)	−0.0015 (8)	0.0131 (8)	0.0000 (8)
C47	0.0491 (15)	0.0670 (19)	0.0527 (15)	−0.0009 (13)	0.0281 (13)	0.0081 (13)
C48	0.0528 (16)	0.0603 (18)	0.0471 (14)	−0.0054 (13)	0.0170 (12)	0.0015 (12)
C49	0.0605 (17)	0.0547 (18)	0.0553 (16)	0.0096 (13)	0.0284 (14)	0.0104 (13)
C50	0.0435 (15)	0.068 (2)	0.072 (2)	0.0043 (13)	0.0163 (14)	0.0379 (17)
C51	0.071 (2)	0.061 (2)	0.0418 (14)	−0.0161 (15)	0.0046 (14)	0.0121 (13)
C52	0.074 (2)	0.0626 (19)	0.0441 (14)	−0.0003 (15)	0.0271 (14)	0.0040 (13)
O1	0.0252 (7)	0.0294 (8)	0.0279 (7)	0.0021 (5)	0.0109 (6)	0.0002 (5)
O2	0.0253 (7)	0.0385 (9)	0.0265 (7)	0.0002 (6)	0.0122 (6)	−0.0024 (6)
O3	0.0357 (8)	0.0337 (8)	0.0245 (7)	0.0034 (6)	0.0129 (6)	0.0021 (6)
O4	0.0268 (7)	0.0266 (7)	0.0274 (7)	−0.0005 (5)	0.0120 (6)	0.0007 (5)
O5	0.0300 (7)	0.0366 (8)	0.0263 (7)	−0.0065 (6)	0.0111 (6)	0.0007 (6)
O6	0.0363 (8)	0.0314 (8)	0.0262 (7)	−0.0050 (6)	0.0131 (6)	−0.0023 (6)
O7	0.0274 (8)	0.0520 (11)	0.0421 (9)	0.0098 (7)	0.0058 (7)	−0.0125 (7)
O8	0.0359 (8)	0.0289 (8)	0.0325 (7)	−0.0056 (6)	0.0089 (6)	0.0040 (6)
P1	0.0297 (3)	0.0298 (3)	0.0263 (2)	0.0013 (2)	0.0127 (2)	0.00127 (19)
P2	0.0283 (3)	0.0292 (3)	0.0284 (2)	−0.0014 (2)	0.0133 (2)	−0.0002 (2)

Geometric parameters (Å, º)

C1—C6	1.390 (3)	C25—C26	1.381 (4)
C1—C2	1.403 (3)	C25—H25	0.9500
C1—O1	1.409 (2)	C26—C27	1.384 (4)
C2—C3	1.394 (3)	C26—H26	0.9500
C2—C13	1.534 (3)	C27—C28	1.402 (3)

C3—C4	1.390 (3)	C27—H27	0.9500
C3—H3	0.9500	C28—C29	1.476 (3)
C4—O7	1.375 (3)	C29—C34	1.397 (3)
C4—C5	1.379 (3)	C29—C30	1.398 (3)
C5—C6	1.404 (3)	C30—C31	1.381 (4)
C5—H5	0.9500	C30—H30	0.9500
C6—C7	1.496 (3)	C31—C32	1.386 (3)
C7—C8	1.394 (3)	C31—H31	0.9500
C7—C12	1.401 (3)	C32—C33	1.393 (3)
C8—C9	1.378 (3)	C32—H32	0.9500
C8—H8	0.9500	C33—C34	1.386 (3)
C9—O8	1.364 (3)	C33—H33	0.9500
C9—C10	1.395 (3)	C34—O3	1.397 (3)
C10—C11	1.389 (3)	C35—C36	1.380 (3)
C10—H10	0.9500	C35—C40	1.388 (3)
C11—C12	1.411 (3)	C35—O5	1.401 (2)
C11—C19	1.544 (3)	C36—C37	1.394 (3)
C12—O4	1.409 (2)	C36—H36	0.9500
C13—C16	1.533 (3)	C37—C38	1.390 (4)
C13—C14	1.536 (3)	C37—H37	0.9500
C13—C15	1.540 (3)	C38—C39	1.379 (3)
C14—H14A	0.9800	C38—H38	0.9500
C14—H14B	0.9800	C39—C40	1.406 (3)
C14—H14C	0.9800	C39—H39	0.9500
C15—H15A	0.9800	C40—C41	1.482 (3)
C15—H15B	0.9800	C41—C42	1.391 (3)
C15—H15C	0.9800	C41—C46	1.399 (3)
C16—H16A	0.9800	C42—C43	1.381 (3)
C16—H16B	0.9800	C42—H42	0.9500
C16—H16C	0.9800	C43—C44	1.386 (3)
C17—O7	1.420 (3)	C43—H43	0.9500
C17—H17A	0.9800	C44—C45	1.379 (3)
C17—H17B	0.9800	C44—H44	0.9500
C17—H17C	0.9800	C45—C46	1.384 (3)
C18—O8	1.428 (3)	C45—H45	0.9500
C18—H18A	0.9800	C46—O6	1.394 (3)
C18—H18B	0.9800	C47—C52	1.370 (4)
C18—H18C	0.9800	C47—C48	1.376 (4)
C19—C22	1.532 (3)	C47—H47	0.9500
C19—C21	1.539 (3)	C48—C49	1.377 (4)
C19—C20	1.543 (3)	C48—H48	0.9500
C20—H20A	0.9800	C49—C50	1.379 (5)
C20—H20B	0.9800	C49—H49	0.9500
C20—H20C	0.9800	C50—C51	1.386 (5)
C21—H21A	0.9800	C50—H50	0.9500
C21—H21B	0.9800	C51—C52	1.372 (5)
C21—H21C	0.9800	C51—H51	0.9500
C22—H22A	0.9800	C52—H52	0.9500

C22—H22B	0.9800	O1—P1	1.6372 (14)
C22—H22C	0.9800	O2—P1	1.6433 (14)
C23—C24	1.381 (3)	O3—P1	1.6300 (15)
C23—O2	1.392 (2)	O4—P2	1.6352 (13)
C23—C28	1.394 (3)	O5—P2	1.6419 (15)
C24—C25	1.394 (3)	O6—P2	1.6282 (15)
C24—H24	0.9500		

C6—C1—C2	122.39 (18)	C23—C24—H24	120.4
C6—C1—O1	118.55 (17)	C25—C24—H24	120.4
C2—C1—O1	118.98 (18)	C26—C25—C24	119.8 (2)
C3—C2—C1	116.0 (2)	C26—C25—H25	120.1
C3—C2—C13	120.23 (19)	C24—C25—H25	120.1
C1—C2—C13	123.77 (19)	C25—C26—C27	120.5 (2)
C4—C3—C2	122.7 (2)	C25—C26—H26	119.7
C4—C3—H3	118.6	C27—C26—H26	119.7
C2—C3—H3	118.6	C26—C27—C28	121.0 (2)
O7—C4—C5	125.3 (2)	C26—C27—H27	119.5
O7—C4—C3	114.84 (19)	C28—C27—H27	119.5
C5—C4—C3	119.86 (19)	C23—C28—C27	117.2 (2)
C4—C5—C6	119.48 (19)	C23—C28—C29	121.77 (19)
C4—C5—H5	120.3	C27—C28—C29	121.0 (2)
C6—C5—H5	120.3	C34—C29—C30	117.2 (2)
C1—C6—C5	119.26 (18)	C34—C29—C28	121.9 (2)
C1—C6—C7	123.42 (18)	C30—C29—C28	120.9 (2)
C5—C6—C7	117.31 (18)	C31—C30—C29	121.6 (2)
C8—C7—C12	119.64 (18)	C31—C30—H30	119.2
C8—C7—C6	117.78 (17)	C29—C30—H30	119.2
C12—C7—C6	122.49 (18)	C30—C31—C32	120.0 (2)
C9—C8—C7	119.79 (18)	C30—C31—H31	120.0
C9—C8—H8	120.1	C32—C31—H31	120.0
C7—C8—H8	120.1	C31—C32—C33	119.8 (2)
O8—C9—C8	124.69 (19)	C31—C32—H32	120.1
O8—C9—C10	115.65 (18)	C33—C32—H32	120.1
C8—C9—C10	119.66 (19)	C34—C33—C32	119.4 (2)
C11—C10—C9	122.54 (19)	C34—C33—H33	120.3
C11—C10—H10	118.7	C32—C33—H33	120.3
C9—C10—H10	118.7	C33—C34—C29	121.8 (2)
C10—C11—C12	116.61 (18)	C33—C34—O3	117.58 (19)
C10—C11—C19	119.47 (18)	C29—C34—O3	120.5 (2)
C12—C11—C19	123.90 (18)	C36—C35—C40	122.08 (19)
C7—C12—O4	118.00 (17)	C36—C35—O5	119.21 (19)
C7—C12—C11	121.21 (19)	C40—C35—O5	118.60 (18)
O4—C12—C11	120.78 (17)	C35—C36—C37	119.5 (2)
C16—C13—C2	111.91 (19)	C35—C36—H36	120.2
C16—C13—C14	106.5 (2)	C37—C36—H36	120.2
C2—C13—C14	109.85 (18)	C38—C37—C36	119.5 (2)
C16—C13—C15	107.6 (2)	C38—C37—H37	120.3

C2—C13—C15	109.0 (2)	C36—C37—H37	120.3
C14—C13—C15	112.01 (19)	C39—C38—C37	120.5 (2)
C13—C14—H14A	109.5	C39—C38—H38	119.8
C13—C14—H14B	109.5	C37—C38—H38	119.8
H14A—C14—H14B	109.5	C38—C39—C40	120.9 (2)
C13—C14—H14C	109.5	C38—C39—H39	119.5
H14A—C14—H14C	109.5	C40—C39—H39	119.5
H14B—C14—H14C	109.5	C35—C40—C39	117.6 (2)
C13—C15—H15A	109.5	C35—C40—C41	121.36 (18)
C13—C15—H15B	109.5	C39—C40—C41	121.0 (2)
H15A—C15—H15B	109.5	C42—C41—C46	117.6 (2)
C13—C15—H15C	109.5	C42—C41—C40	120.90 (19)
H15A—C15—H15C	109.5	C46—C41—C40	121.5 (2)
H15B—C15—H15C	109.5	C43—C42—C41	121.4 (2)
C13—C16—H16A	109.5	C43—C42—H42	119.3
C13—C16—H16B	109.5	C41—C42—H42	119.3
H16A—C16—H16B	109.5	C42—C43—C44	119.8 (2)
C13—C16—H16C	109.5	C42—C43—H43	120.1
H16A—C16—H16C	109.5	C44—C43—H43	120.1
H16B—C16—H16C	109.5	C45—C44—C43	120.2 (2)
O7—C17—H17A	109.5	C45—C44—H44	119.9
O7—C17—H17B	109.5	C43—C44—H44	119.9
H17A—C17—H17B	109.5	C44—C45—C46	119.6 (2)
O7—C17—H17C	109.5	C44—C45—H45	120.2
H17A—C17—H17C	109.5	C46—C45—H45	120.2
H17B—C17—H17C	109.5	C45—C46—O6	118.40 (19)
O8—C18—H18A	109.5	C45—C46—C41	121.4 (2)
O8—C18—H18B	109.5	O6—C46—C41	119.96 (18)
H18A—C18—H18B	109.5	C52—C47—C48	120.5 (3)
O8—C18—H18C	109.5	C52—C47—H47	119.7
H18A—C18—H18C	109.5	C48—C47—H47	119.7
H18B—C18—H18C	109.5	C47—C48—C49	120.1 (3)
C22—C19—C21	106.76 (17)	C47—C48—H48	119.9
C22—C19—C20	110.18 (17)	C49—C48—H48	119.9
C21—C19—C20	107.29 (17)	C48—C49—C50	119.6 (3)
C22—C19—C11	111.34 (16)	C48—C49—H49	120.2
C21—C19—C11	111.70 (17)	C50—C49—H49	120.2
C20—C19—C11	109.46 (16)	C49—C50—C51	119.9 (3)
C19—C20—H20A	109.5	C49—C50—H50	120.1
C19—C20—H20B	109.5	C51—C50—H50	120.1
H20A—C20—H20B	109.5	C52—C51—C50	120.2 (3)
C19—C20—H20C	109.5	C52—C51—H51	119.9
H20A—C20—H20C	109.5	C50—C51—H51	119.9
H20B—C20—H20C	109.5	C47—C52—C51	119.7 (3)
C19—C21—H21A	109.5	C47—C52—H52	120.2
C19—C21—H21B	109.5	C51—C52—H52	120.2
H21A—C21—H21B	109.5	C1—O1—P1	120.65 (13)
C19—C21—H21C	109.5	C23—O2—P1	119.44 (12)

H21A—C21—H21C	109.5	C34—O3—P1	125.54 (12)
H21B—C21—H21C	109.5	C12—O4—P2	116.93 (12)
C19—C22—H22A	109.5	C35—O5—P2	115.87 (12)
C19—C22—H22B	109.5	C46—O6—P2	125.57 (12)
H22A—C22—H22B	109.5	C4—O7—C17	116.87 (18)
C19—C22—H22C	109.5	C9—O8—C18	115.57 (17)
H22A—C22—H22C	109.5	O3—P1—O1	102.27 (8)
H22B—C22—H22C	109.5	O3—P1—O2	99.42 (8)
C24—C23—O2	118.85 (19)	O1—P1—O2	93.84 (7)
C24—C23—C28	122.4 (2)	O6—P2—O4	102.43 (8)
O2—C23—C28	118.5 (2)	O6—P2—O5	99.14 (8)
C23—C24—C25	119.1 (2)	O4—P2—O5	95.73 (7)

C6—C1—C2—C3	4.9 (3)	C31—C32—C33—C34	−1.3 (3)
O1—C1—C2—C3	−178.35 (17)	C32—C33—C34—C29	3.6 (3)
C6—C1—C2—C13	−174.64 (19)	C32—C33—C34—O3	179.15 (17)
O1—C1—C2—C13	2.1 (3)	C30—C29—C34—C33	−3.1 (3)
C1—C2—C3—C4	−0.3 (3)	C28—C29—C34—C33	176.17 (18)
C13—C2—C3—C4	179.2 (2)	C30—C29—C34—O3	−178.52 (16)
C2—C3—C4—O7	178.8 (2)	C28—C29—C34—O3	0.8 (3)
C2—C3—C4—C5	−3.3 (3)	C40—C35—C36—C37	−0.5 (3)
O7—C4—C5—C6	−179.9 (2)	O5—C35—C36—C37	175.6 (2)
C3—C4—C5—C6	2.5 (3)	C35—C36—C37—C38	−0.2 (4)
C2—C1—C6—C5	−5.7 (3)	C36—C37—C38—C39	0.6 (4)
O1—C1—C6—C5	177.48 (17)	C37—C38—C39—C40	−0.5 (3)
C2—C1—C6—C7	175.04 (18)	C36—C35—C40—C39	0.6 (3)
O1—C1—C6—C7	−1.7 (3)	O5—C35—C40—C39	−175.47 (17)
C4—C5—C6—C1	1.9 (3)	C36—C35—C40—C41	178.2 (2)
C4—C5—C6—C7	−178.85 (18)	O5—C35—C40—C41	2.1 (3)
C1—C6—C7—C8	117.3 (2)	C38—C39—C40—C35	−0.1 (3)
C5—C6—C7—C8	−61.9 (2)	C38—C39—C40—C41	−177.74 (19)
C1—C6—C7—C12	−66.1 (3)	C35—C40—C41—C42	−136.9 (2)
C5—C6—C7—C12	114.7 (2)	C39—C40—C41—C42	40.6 (3)
C12—C7—C8—C9	1.6 (3)	C35—C40—C41—C46	42.9 (3)
C6—C7—C8—C9	178.23 (17)	C39—C40—C41—C46	−139.6 (2)
C7—C8—C9—O8	−175.82 (17)	C46—C41—C42—C43	−0.9 (3)
C7—C8—C9—C10	4.7 (3)	C40—C41—C42—C43	178.84 (18)
O8—C9—C10—C11	174.68 (17)	C41—C42—C43—C44	−0.1 (3)
C8—C9—C10—C11	−5.8 (3)	C42—C43—C44—C45	1.4 (3)
C9—C10—C11—C12	0.4 (3)	C43—C44—C45—C46	−1.7 (3)
C9—C10—C11—C19	−177.90 (17)	C44—C45—C46—O6	175.18 (17)
C8—C7—C12—O4	173.19 (16)	C44—C45—C46—C41	0.6 (3)
C6—C7—C12—O4	−3.3 (3)	C42—C41—C46—C45	0.6 (3)
C8—C7—C12—C11	−7.1 (3)	C40—C41—C46—C45	−179.11 (17)
C6—C7—C12—C11	176.38 (17)	C42—C41—C46—O6	−173.83 (16)
C10—C11—C12—C7	6.0 (3)	C40—C41—C46—O6	6.4 (3)
C19—C11—C12—C7	−175.73 (17)	C52—C47—C48—C49	−0.5 (4)
C10—C11—C12—O4	−174.29 (16)	C47—C48—C49—C50	−0.1 (4)

C19—C11—C12—O4	3.9 (3)	C48—C49—C50—C51	0.5 (4)
C3—C2—C13—C16	−8.9 (3)	C49—C50—C51—C52	−0.5 (4)
C1—C2—C13—C16	170.6 (2)	C48—C47—C52—C51	0.6 (4)
C3—C2—C13—C14	−126.9 (2)	C50—C51—C52—C47	−0.1 (4)
C1—C2—C13—C14	52.6 (3)	C6—C1—O1—P1	−63.9 (2)
C3—C2—C13—C15	110.0 (2)	C2—C1—O1—P1	119.26 (17)
C1—C2—C13—C15	−70.5 (3)	C24—C23—O2—P1	105.80 (19)
C10—C11—C19—C22	−133.04 (19)	C28—C23—O2—P1	−79.4 (2)
C12—C11—C19—C22	48.8 (2)	C33—C34—O3—P1	116.70 (17)
C10—C11—C19—C21	−13.8 (2)	C29—C34—O3—P1	−67.7 (2)
C12—C11—C19—C21	168.04 (18)	C7—C12—O4—P2	−66.19 (19)
C10—C11—C19—C20	104.9 (2)	C11—C12—O4—P2	114.13 (17)
C12—C11—C19—C20	−73.3 (2)	C36—C35—O5—P2	102.7 (2)
O2—C23—C24—C25	175.12 (19)	C40—C35—O5—P2	−81.1 (2)
C28—C23—C24—C25	0.5 (3)	C45—C46—O6—P2	116.01 (18)
C23—C24—C25—C26	−0.5 (3)	C41—C46—O6—P2	−69.4 (2)
C24—C25—C26—C27	−0.1 (4)	C5—C4—O7—C17	−1.9 (3)
C25—C26—C27—C28	0.6 (3)	C3—C4—O7—C17	175.8 (2)
C24—C23—C28—C27	0.0 (3)	C8—C9—O8—C18	14.4 (3)
O2—C23—C28—C27	−174.62 (17)	C10—C9—O8—C18	−166.10 (17)
C24—C23—C28—C29	178.94 (19)	C34—O3—P1—O1	−59.05 (17)
O2—C23—C28—C29	4.3 (3)	C34—O3—P1—O2	37.00 (17)
C26—C27—C28—C23	−0.6 (3)	C1—O1—P1—O3	−134.20 (14)
C26—C27—C28—C29	−179.5 (2)	C1—O1—P1—O2	125.29 (14)
C23—C28—C29—C34	43.9 (3)	C23—O2—P1—O3	53.57 (17)
C27—C28—C29—C34	−137.1 (2)	C23—O2—P1—O1	156.69 (16)
C23—C28—C29—C30	−136.8 (2)	C46—O6—P2—O4	−66.92 (16)
C27—C28—C29—C30	42.1 (3)	C46—O6—P2—O5	31.07 (17)
C34—C29—C30—C31	0.4 (3)	C12—O4—P2—O6	−131.10 (13)
C28—C29—C30—C31	−178.94 (19)	C12—O4—P2—O5	128.21 (13)
C29—C30—C31—C32	1.9 (3)	C35—O5—P2—O6	60.03 (15)
C30—C31—C32—C33	−1.4 (3)	C35—O5—P2—O4	163.63 (14)

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the C47–C52 benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C39—H39···*Cg*1^{i}	0.95	2.75	3.678 (3)	165

Symmetry code: (i) *x*, *y*−1, *z*.