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# (6,6′-Dimethoxybiphenyl-2,2′-diyl)bis(diphenylphosphane) *P*,*P*′-dioxide dihydrate

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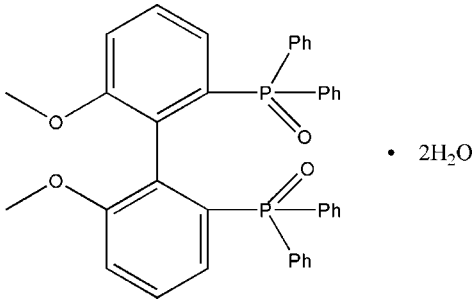
Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.044; $wR$ factor = 0.130; data-to-parameter ratio = 14.9.

In the title compound, $C_{38}H_{32}O_4P_2\cdot 2H_2O$, the dihedral angle between the methoxyphenol rings is 84.11 (7)°. O—H···O hydrogen bonds connect the water molecules of crystallization with the main molecule.

## Related literature

For the synthesis of the title compound and its unsolvated crystal structure, see: Doherty *et al.* (2009). For similar structures, see: Meijboom (2011); Wang *et al.* (2011); Warsink *et al.* (2011).




• $2H_2O$

## Experimental

### Crystal data

$C_{38}H_{32}O_4P_2\cdot 2H_2O$
$M_r$ = 650.61
Orthorhombic, *Pbca*
$a$ = 13.108 (3) Å
$b$ = 15.650 (3) Å
$c$ = 33.967 (7) Å
$V$ = 6968 (3) Å$^3$
$Z$ = 8
Cu $K\alpha$ radiation
$\mu$ = 1.50 mm$^{-1}$
$T$ = 296 K
0.23 × 0.10 × 0.10 mm

### Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min}$ = 0.725, $T_{max}$ = 0.865
31453 measured reflections
6197 independent reflections
4670 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.042

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.130
$S$ = 1.03
6197 reflections
415 parameters
3 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.29 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1′—H1′$A$···O4 | 0.85 | 2.31 | 2.854 (4) | 123 |
| O2′—H2′$A$···O3 | 0.85 | 1.96 | 2.799 (3) | 169 |

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6568).

## References

Bruker (2009). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Doherty, S., Smyth, C. H., Harrington, R. W. & Clegg, W. (2009). *Organometallics*, **28**, 5273–5276.
Meijboom, R. (2011). *Acta Cryst.* E**67**, m1663.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Wang, H., Zhang, X.-M., Li, P. & Chen, H.-Y. (2011). *Acta Cryst.* E**67**, o3149.
Warsink, S., Koen, R. & Roodt, A. (2011). *Acta Cryst.* E**67**, m1666.

# supporting information

*Acta Cryst.* (2012). E**68**, o804 [doi:10.1107/S1600536812005314]

# (6,6′-Dimethoxybiphenyl-2,2′-diyl)bis(diphenylphosphane) *P*,*P*′-dioxide dihydrate

**Dongmei Dai, Lin Tang and Yanqing Gong**

## S1. Comment

The title compound, (I) (Fig. 1), (6,6′-Dimethoxybiphenyl-2,2′-diyl)- bis(diphenylphosphine)dioxide dihydrate can be synthesized according to the procedure of Doherty *et al.*, (2009).

Compared with the structure of $C_{38}H_{32}O_4P_2$ (CCDC 756817), Doherty *et al.*, (2009), in which weak intermolecular C—H···O hydrogen bonds pull adjacent molecules closer, the interesting difference is the two solvent waters in the asymmetric unit, which form strong O—H···O hydrogen bonds with O atom of P═O,(Fig. 2). The waters take place of the adjacent bulky molecules to stabilize the crystal packing. As a result, the molecules pack in a different, much looser form in the crystal. And the calculated density of the crystal also confirms this point, 1.240 and 1.419 g $cm^{-3}$ for the title compoud and the previous structure, respectively.

The whole structure exhibits as a dimer of triarylphosphorus oxide through C1—C7 covalent bond. The bond lengths [1.803 (2)–1.812 (2) Å] and angles[104.05 (10)–107.27 (11)°] of $C_{aryl}$—P do not show large deviations from those observed in related structures (Meijboom, 2011; Wang, *et al.*, 2011; Warsink, *et al.*, 2011). Two methoxyphenyl rings locate almost perpendicular to each other, with a dihedral angle of 84.11(0.07)°.

## S2. Experimental

The title compound was prepared according to the procedure of Doherty *et al.*, (2009) through double cycloaddition-elimination by using 1,4-bis-(diphenylphosphinoyl)buta-1,3-diyne and 1-methoxy-1,3-cycolhexadiene, heated with microwave in toluene solution. Colourless blocks were obtained from acetone/water(1:1) solution after about a week at room temperature.

## S3. Refinement

The water H atoms were located in difference Fourier map and were then subsequently treated as riding atoms with O—H distances of 0.85 Å and $U_{iso}$(H) = 1.5$U_{eq}$(O). The locations of the water H atoms should be regarded as less certain than those of the other atoms. All non-solvent H atoms were placed in geometically idealized positions and constrained to ride on their parent atoms with C—H distances of 0.93 Å(0.96 for methyl group) and $U_{iso}$(H) = 1.2(1.5 for CH3)$U_{eq}$(C) for CH.

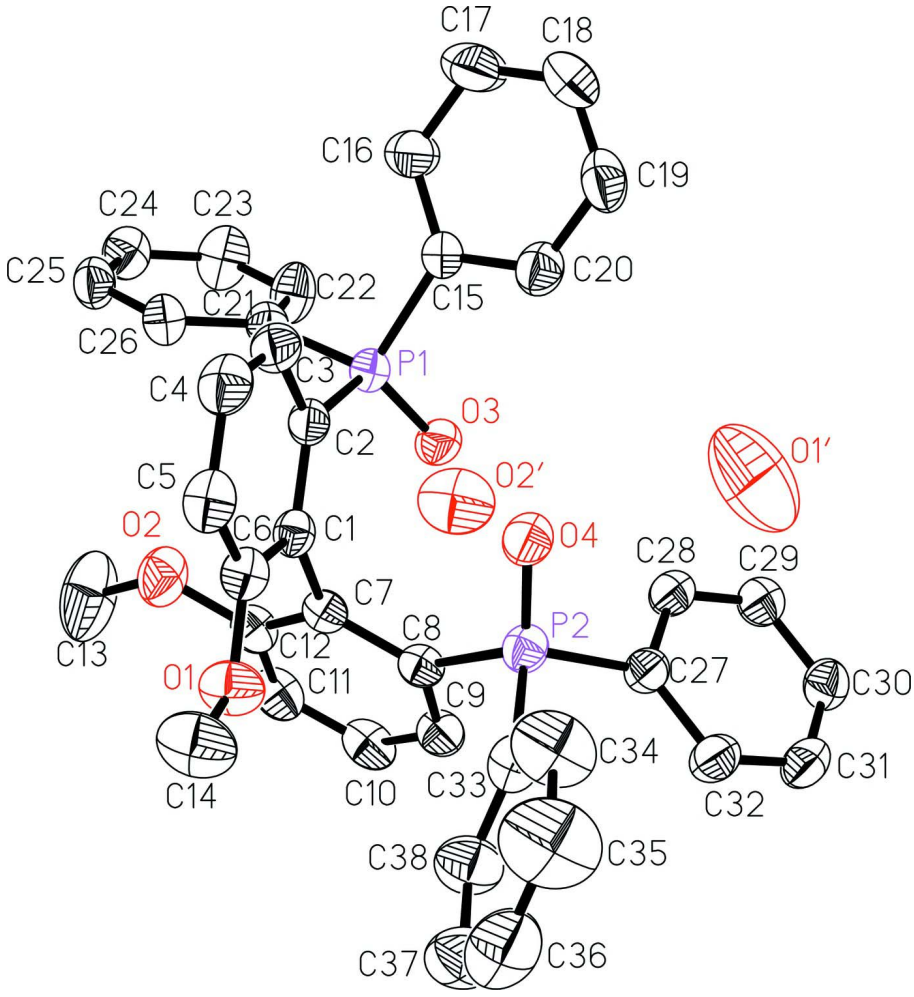



**Figure 1**

View of the molecule of (I) showing displacement ellipsoids drawn at the 30% probability level. Hydrogen atoms were omitted for clarity.

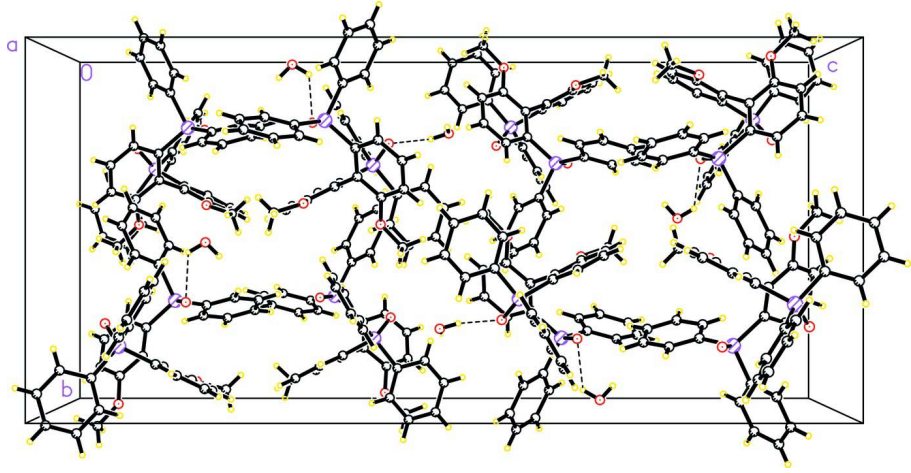


**Figure 2**

The packing of the title compound, viewed down the *a* axis. Dashed lines indicate the O—H···O hydrogen bonds.

**(6,6′-Dimethoxybiphenyl-2,2′-diyl)bis(diphenylphosphane) *P*,*P*′-dioxide dihydrate**

*Crystal data*

$C_{38}H_{32}O_4P_2·2H_2O$
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Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
*a* = 13.108 (3) Å
*b* = 15.650 (3) Å
*c* = 33.967 (7) Å
*V* = 6968 (3) Å$^3$
*Z* = 8
*F*(000) = 2736
$D_x$ = 1.240 Mg m$^{-3}$
Cu *Kα* radiation, *λ* = 1.54178 Å
Cell parameters from 4670 reflections
*θ* = 2.6–67.7°
*μ* = 1.50 mm$^{-1}$
*T* = 296 K
Block, colourless
0.23 × 0.10 × 0.10 mm

*Data collection*

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
*φ* and *ω* scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min}$ = 0.725, $T_{max}$ = 0.865
31453 measured reflections
6197 independent reflections
4670 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.042
$\theta_{max}$ = 67.7°, $\theta_{min}$ = 2.6°
*h* = −15→12
*k* = −18→18
*l* = −35→40

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.130
*S* = 1.03
6197 reflections
415 parameters
3 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
$w = 1/[\sigma^2(F_o^2) + (0.066P)^2 + 1.3879P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.20 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.29 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 | 0.80685 (4) | 0.18583 (3) | 0.593688 (18) | 0.05612 (16) |
| P2 | 1.07260 (4) | 0.30232 (4) | 0.651596 (17) | 0.05821 (17) |

| | | | | |
|---|---|---|---|---|
| O1 | 1.10418 (14) | 0.07362 (13) | 0.67804 (5) | 0.0805 (5) |
| O2 | 1.00948 (16) | 0.01615 (12) | 0.58155 (6) | 0.0875 (6) |
| O3 | 0.87621 (12) | 0.24506 (10) | 0.57242 (5) | 0.0680 (4) |
| O4 | 0.96975 (12) | 0.30072 (10) | 0.66970 (5) | 0.0710 (4) |
| C1 | 0.96504 (16) | 0.12224 (12) | 0.64144 (6) | 0.0516 (5) |
| C2 | 0.86030 (16) | 0.13410 (12) | 0.63672 (6) | 0.0539 (5) |
| C3 | 0.79402 (19) | 0.10720 (15) | 0.66641 (7) | 0.0682 (6) |
| H3A | 0.7241 | 0.1153 | 0.6635 | 0.082* |
| C4 | 0.8310 (2) | 0.06900 (16) | 0.69975 (8) | 0.0751 (7) |
| H4A | 0.7858 | 0.0507 | 0.7191 | 0.090* |
| C5 | 0.9343 (2) | 0.05729 (16) | 0.70501 (7) | 0.0699 (6) |
| H5A | 0.9589 | 0.0320 | 0.7279 | 0.084* |
| C6 | 1.00078 (18) | 0.08347 (14) | 0.67601 (6) | 0.0597 (5) |
| C7 | 1.04118 (15) | 0.14815 (13) | 0.61094 (6) | 0.0515 (5) |
| C8 | 1.09387 (15) | 0.22572 (14) | 0.61251 (6) | 0.0532 (5) |
| C9 | 1.16457 (18) | 0.24536 (15) | 0.58293 (7) | 0.0641 (6) |
| H9A | 1.2000 | 0.2968 | 0.5838 | 0.077* |
| C10 | 1.1816 (2) | 0.18884 (17) | 0.55269 (7) | 0.0727 (7) |
| H10A | 1.2285 | 0.2025 | 0.5331 | 0.087* |
| C11 | 1.1304 (2) | 0.11233 (18) | 0.55099 (7) | 0.0728 (7) |
| H11A | 1.1422 | 0.0747 | 0.5303 | 0.087* |
| C12 | 1.06147 (18) | 0.09148 (15) | 0.58007 (7) | 0.0622 (6) |
| C13 | 1.0309 (4) | −0.0473 (2) | 0.55322 (12) | 0.1502 (19) |
| H13A | 0.9883 | −0.0962 | 0.5579 | 0.225* |
| H13B | 1.1013 | −0.0637 | 0.5552 | 0.225* |
| H13C | 1.0176 | −0.0251 | 0.5274 | 0.225* |
| C14 | 1.1479 (3) | 0.0481 (2) | 0.71429 (9) | 0.1078 (11) |
| H14A | 1.2206 | 0.0438 | 0.7114 | 0.162* |
| H14B | 1.1207 | −0.0065 | 0.7219 | 0.162* |
| H14C | 1.1321 | 0.0896 | 0.7342 | 0.162* |
| C15 | 0.69724 (17) | 0.24298 (13) | 0.61195 (7) | 0.0627 (6) |
| C16 | 0.5982 (2) | 0.2192 (2) | 0.60574 (14) | 0.1231 (14) |
| H16A | 0.5844 | 0.1698 | 0.5915 | 0.148* |
| C17 | 0.5185 (3) | 0.2673 (3) | 0.62022 (16) | 0.152 (2) |
| H17A | 0.4517 | 0.2492 | 0.6163 | 0.183* |
| C18 | 0.5367 (3) | 0.3408 (2) | 0.64014 (11) | 0.1039 (10) |
| H18A | 0.4828 | 0.3735 | 0.6497 | 0.125* |
| C19 | 0.6331 (3) | 0.36566 (17) | 0.64588 (9) | 0.0855 (8) |
| H19A | 0.6459 | 0.4163 | 0.6593 | 0.103* |
| C20 | 0.7139 (2) | 0.31772 (15) | 0.63230 (8) | 0.0727 (6) |
| H20A | 0.7804 | 0.3360 | 0.6369 | 0.087* |
| C21 | 0.75769 (17) | 0.10246 (13) | 0.56231 (6) | 0.0573 (5) |
| C22 | 0.7201 (2) | 0.12554 (17) | 0.52578 (8) | 0.0844 (8) |
| H22A | 0.7222 | 0.1825 | 0.5181 | 0.101* |
| C23 | 0.6794 (3) | 0.0651 (2) | 0.50058 (8) | 0.0949 (9) |
| H23A | 0.6537 | 0.0815 | 0.4762 | 0.114* |
| C24 | 0.6771 (2) | −0.01876 (18) | 0.51153 (8) | 0.0778 (7) |
| H24A | 0.6497 | −0.0594 | 0.4946 | 0.093* |

| | | | | |
|---|---|---|---|---|
| C25 | 0.7145 (2) | −0.04304 (15) | 0.54702 (8) | 0.0721 (7) |
| H25A | 0.7129 | −0.1003 | 0.5543 | 0.087* |
| C26 | 0.7550 (2) | 0.01716 (14) | 0.57237 (7) | 0.0670 (6) |
| H26A | 0.7809 | −0.0002 | 0.5966 | 0.080* |
| C27 | 1.09683 (17) | 0.40435 (14) | 0.62868 (7) | 0.0587 (5) |
| C28 | 1.02525 (18) | 0.43283 (15) | 0.60170 (7) | 0.0668 (6) |
| H28A | 0.9701 | 0.3982 | 0.5951 | 0.080* |
| C29 | 1.0353 (2) | 0.51243 (16) | 0.58446 (8) | 0.0740 (7) |
| H29A | 0.9868 | 0.5313 | 0.5664 | 0.089* |
| C30 | 1.1163 (2) | 0.56346 (16) | 0.59384 (8) | 0.0738 (7) |
| H30A | 1.1224 | 0.6172 | 0.5824 | 0.089* |
| C31 | 1.1881 (2) | 0.53592 (16) | 0.61996 (9) | 0.0782 (7) |
| H31A | 1.2435 | 0.5707 | 0.6259 | 0.094* |
| C32 | 1.17926 (19) | 0.45643 (16) | 0.63770 (8) | 0.0726 (7) |
| H32A | 1.2284 | 0.4381 | 0.6556 | 0.087* |
| C33 | 1.1719 (2) | 0.28665 (16) | 0.68767 (7) | 0.0703 (6) |
| C34 | 1.1509 (3) | 0.3085 (3) | 0.72569 (10) | 0.1249 (13) |
| H34A | 1.0867 | 0.3293 | 0.7323 | 0.150* |
| C35 | 1.2251 (5) | 0.2998 (4) | 0.75440 (12) | 0.168 (2) |
| H35A | 1.2104 | 0.3166 | 0.7800 | 0.202* |
| C36 | 1.3176 (4) | 0.2678 (3) | 0.74624 (13) | 0.1293 (16) |
| H36A | 1.3661 | 0.2615 | 0.7660 | 0.155* |
| C37 | 1.3394 (3) | 0.2448 (3) | 0.70872 (12) | 0.1192 (13) |
| H37A | 1.4035 | 0.2230 | 0.7026 | 0.143* |
| C38 | 1.2661 (3) | 0.2537 (2) | 0.67941 (9) | 0.0958 (9) |
| H38A | 1.2814 | 0.2370 | 0.6538 | 0.115* |
| O2′ | 0.9356 (2) | 0.2718 (2) | 0.49425 (8) | 0.1491 (11) |
| H2′A | 0.9192 | 0.2565 | 0.5174 | 0.224* |
| H2′B | 0.9978 | 0.2869 | 0.4960 | 0.224* |
| O1′ | 0.9143 (4) | 0.4666 (3) | 0.69732 (12) | 0.228 (2) |
| H1′A | 0.9114 | 0.4391 | 0.6758 | 0.342* |
| H1′B | 0.9306 | 0.4285 | 0.7140 | 0.342* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| P1 | 0.0547 (3) | 0.0467 (3) | 0.0669 (4) | −0.0015 (2) | 0.0027 (2) | 0.0036 (2) |
| P2 | 0.0619 (3) | 0.0572 (3) | 0.0556 (3) | −0.0052 (2) | 0.0050 (2) | −0.0018 (3) |
| O1 | 0.0752 (11) | 0.1074 (14) | 0.0588 (10) | 0.0052 (10) | −0.0087 (8) | 0.0174 (9) |
| O2 | 0.1042 (15) | 0.0723 (11) | 0.0862 (13) | −0.0095 (10) | 0.0138 (10) | −0.0268 (10) |
| O3 | 0.0634 (9) | 0.0622 (9) | 0.0785 (11) | −0.0065 (7) | 0.0018 (8) | 0.0160 (8) |
| O4 | 0.0722 (10) | 0.0677 (10) | 0.0731 (10) | −0.0086 (8) | 0.0224 (8) | −0.0119 (8) |
| C1 | 0.0609 (12) | 0.0465 (10) | 0.0474 (11) | −0.0029 (9) | 0.0048 (9) | −0.0025 (9) |
| C2 | 0.0591 (12) | 0.0454 (10) | 0.0571 (12) | −0.0022 (9) | 0.0062 (9) | −0.0018 (9) |
| C3 | 0.0646 (14) | 0.0648 (14) | 0.0753 (16) | −0.0039 (11) | 0.0159 (11) | 0.0025 (12) |
| C4 | 0.0916 (19) | 0.0719 (15) | 0.0618 (15) | −0.0090 (13) | 0.0237 (13) | 0.0038 (12) |
| C5 | 0.0956 (19) | 0.0663 (14) | 0.0479 (13) | −0.0063 (13) | 0.0060 (12) | 0.0021 (11) |
| C6 | 0.0712 (14) | 0.0584 (13) | 0.0497 (12) | −0.0036 (10) | 0.0000 (10) | −0.0021 (10) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C7 | 0.0524 (11) | 0.0572 (11) | 0.0448 (11) | 0.0051 (9) | −0.0008 (8) | 0.0027 (9) |
| C8 | 0.0519 (12) | 0.0597 (12) | 0.0482 (11) | 0.0046 (9) | 0.0035 (8) | 0.0033 (9) |
| C9 | 0.0623 (13) | 0.0659 (13) | 0.0642 (14) | 0.0042 (11) | 0.0123 (10) | 0.0116 (11) |
| C10 | 0.0767 (16) | 0.0872 (18) | 0.0543 (14) | 0.0201 (14) | 0.0200 (11) | 0.0167 (12) |
| C11 | 0.0863 (18) | 0.0801 (17) | 0.0522 (13) | 0.0184 (14) | 0.0109 (11) | −0.0026 (12) |
| C12 | 0.0686 (14) | 0.0652 (13) | 0.0528 (12) | 0.0090 (11) | 0.0026 (10) | −0.0033 (10) |
| C13 | 0.245 (6) | 0.096 (3) | 0.110 (3) | −0.033 (3) | 0.049 (3) | −0.046 (2) |
| C14 | 0.105 (2) | 0.150 (3) | 0.0676 (18) | 0.018 (2) | −0.0213 (16) | 0.0263 (19) |
| C15 | 0.0613 (13) | 0.0479 (11) | 0.0788 (15) | −0.0004 (10) | 0.0012 (11) | −0.0032 (11) |
| C16 | 0.0627 (18) | 0.093 (2) | 0.213 (4) | −0.0049 (15) | 0.009 (2) | −0.073 (3) |
| C17 | 0.065 (2) | 0.135 (3) | 0.257 (6) | 0.007 (2) | 0.014 (3) | −0.096 (4) |
| C18 | 0.089 (2) | 0.088 (2) | 0.134 (3) | 0.0257 (18) | 0.0201 (19) | −0.020 (2) |
| C19 | 0.112 (2) | 0.0569 (14) | 0.088 (2) | 0.0085 (15) | 0.0185 (16) | −0.0100 (13) |
| C20 | 0.0800 (17) | 0.0644 (14) | 0.0738 (16) | −0.0074 (12) | 0.0047 (12) | −0.0072 (12) |
| C21 | 0.0608 (13) | 0.0540 (12) | 0.0570 (12) | 0.0011 (9) | 0.0060 (10) | 0.0006 (10) |
| C22 | 0.120 (2) | 0.0645 (15) | 0.0687 (16) | −0.0097 (15) | −0.0074 (15) | 0.0094 (13) |
| C23 | 0.137 (3) | 0.091 (2) | 0.0572 (15) | −0.0121 (19) | −0.0136 (16) | 0.0031 (15) |
| C24 | 0.0936 (19) | 0.0745 (17) | 0.0652 (16) | −0.0039 (14) | 0.0093 (13) | −0.0166 (13) |
| C25 | 0.0838 (17) | 0.0520 (13) | 0.0804 (17) | 0.0009 (11) | 0.0039 (13) | −0.0065 (12) |
| C26 | 0.0778 (16) | 0.0547 (12) | 0.0686 (15) | 0.0027 (11) | −0.0018 (12) | 0.0009 (11) |
| C27 | 0.0562 (12) | 0.0574 (12) | 0.0626 (13) | −0.0039 (9) | 0.0022 (10) | −0.0029 (10) |
| C28 | 0.0606 (14) | 0.0619 (13) | 0.0779 (16) | −0.0073 (11) | −0.0072 (11) | 0.0004 (12) |
| C29 | 0.0722 (16) | 0.0658 (15) | 0.0841 (18) | 0.0024 (12) | −0.0062 (12) | 0.0043 (13) |
| C30 | 0.0782 (17) | 0.0580 (13) | 0.0851 (18) | −0.0042 (12) | 0.0047 (13) | 0.0057 (13) |
| C31 | 0.0718 (16) | 0.0683 (15) | 0.094 (2) | −0.0198 (12) | −0.0023 (14) | 0.0010 (14) |
| C32 | 0.0649 (15) | 0.0703 (15) | 0.0827 (17) | −0.0114 (11) | −0.0117 (12) | 0.0052 (13) |
| C33 | 0.0855 (18) | 0.0676 (14) | 0.0579 (14) | −0.0066 (12) | −0.0076 (12) | 0.0045 (11) |
| C34 | 0.139 (3) | 0.172 (4) | 0.0640 (19) | 0.026 (3) | −0.0141 (19) | −0.015 (2) |
| C35 | 0.192 (5) | 0.242 (6) | 0.071 (2) | 0.030 (5) | −0.041 (3) | −0.024 (3) |
| C36 | 0.163 (4) | 0.127 (3) | 0.098 (3) | −0.025 (3) | −0.065 (3) | 0.027 (2) |
| C37 | 0.103 (3) | 0.139 (3) | 0.116 (3) | 0.000 (2) | −0.041 (2) | 0.022 (3) |
| C38 | 0.091 (2) | 0.120 (2) | 0.0771 (18) | 0.0091 (18) | −0.0196 (15) | 0.0020 (17) |
| O2′ | 0.147 (2) | 0.198 (3) | 0.1023 (18) | 0.006 (2) | 0.0169 (16) | 0.027 (2) |
| O1′ | 0.287 (5) | 0.217 (4) | 0.179 (3) | 0.099 (4) | 0.046 (3) | −0.055 (3) |

*Geometric parameters (Å, º)*

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| P1—O3 | 1.4859 (16) | C18—C19 | 1.337 (4) |
| P1—C15 | 1.802 (2) | C18—H18A | 0.9300 |
| P1—C21 | 1.804 (2) | C19—C20 | 1.377 (4) |
| P1—C2 | 1.812 (2) | C19—H19A | 0.9300 |
| P2—O4 | 1.4819 (16) | C20—H20A | 0.9300 |
| P2—C33 | 1.804 (3) | C21—C26 | 1.378 (3) |
| P2—C27 | 1.804 (2) | C21—C22 | 1.383 (3) |
| P2—C8 | 1.810 (2) | C22—C23 | 1.383 (4) |
| O1—C6 | 1.366 (3) | C22—H22A | 0.9300 |
| O1—C14 | 1.416 (3) | C23—C24 | 1.364 (4) |
| O2—C12 | 1.363 (3) | C23—H23A | 0.9300 |

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| O2—C13 | 1.411 (4) | C24—C25 | 1.356 (4) |
| C1—C2 | 1.395 (3) | C24—H24A | 0.9300 |
| C1—C6 | 1.402 (3) | C25—C26 | 1.383 (3) |
| C1—C7 | 1.495 (3) | C25—H25A | 0.9300 |
| C2—C3 | 1.396 (3) | C26—H26A | 0.9300 |
| C3—C4 | 1.369 (4) | C27—C28 | 1.385 (3) |
| C3—H3A | 0.9300 | C27—C32 | 1.388 (3) |
| C4—C5 | 1.379 (4) | C28—C29 | 1.383 (3) |
| C4—H4A | 0.9300 | C28—H28A | 0.9300 |
| C5—C6 | 1.377 (3) | C29—C30 | 1.366 (4) |
| C5—H5A | 0.9300 | C29—H29A | 0.9300 |
| C7—C8 | 1.398 (3) | C30—C31 | 1.364 (4) |
| C7—C12 | 1.399 (3) | C30—H30A | 0.9300 |
| C8—C9 | 1.401 (3) | C31—C32 | 1.387 (3) |
| C9—C10 | 1.374 (3) | C31—H31A | 0.9300 |
| C9—H9A | 0.9300 | C32—H32A | 0.9300 |
| C10—C11 | 1.374 (4) | C33—C34 | 1.364 (4) |
| C10—H10A | 0.9300 | C33—C38 | 1.368 (4) |
| C11—C12 | 1.378 (3) | C34—C35 | 1.384 (6) |
| C11—H11A | 0.9300 | C34—H34A | 0.9300 |
| C13—H13A | 0.9600 | C35—C36 | 1.341 (6) |
| C13—H13B | 0.9600 | C35—H35A | 0.9300 |
| C13—H13C | 0.9600 | C36—C37 | 1.355 (6) |
| C14—H14A | 0.9600 | C36—H36A | 0.9300 |
| C14—H14B | 0.9600 | C37—C38 | 1.391 (4) |
| C14—H14C | 0.9600 | C37—H37A | 0.9300 |
| C15—C16 | 1.367 (4) | C38—H38A | 0.9300 |
| C15—C20 | 1.376 (3) | O2′—H2′A | 0.8497 |
| C16—C17 | 1.378 (4) | O2′—H2′B | 0.8501 |
| C16—H16A | 0.9300 | O1′—H1′A | 0.8498 |
| C17—C18 | 1.356 (5) | O1′—H1′B | 0.8500 |
| C17—H17A | 0.9300 | | |

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| O3—P1—C15 | 110.21 (10) | C18—C17—C16 | 120.6 (3) |
| O3—P1—C21 | 112.49 (10) | C18—C17—H17A | 119.7 |
| C15—P1—C21 | 106.11 (11) | C16—C17—H17A | 119.7 |
| O3—P1—C2 | 115.74 (10) | C19—C18—C17 | 119.0 (3) |
| C15—P1—C2 | 104.62 (11) | C19—C18—H18A | 120.5 |
| C21—P1—C2 | 106.96 (10) | C17—C18—H18A | 120.5 |
| O4—P2—C33 | 111.83 (12) | C18—C19—C20 | 121.3 (3) |
| O4—P2—C27 | 110.74 (10) | C18—C19—H19A | 119.3 |
| C33—P2—C27 | 106.64 (11) | C20—C19—H19A | 119.3 |
| O4—P2—C8 | 115.67 (10) | C15—C20—C19 | 120.6 (3) |
| C33—P2—C8 | 107.27 (11) | C15—C20—H20A | 119.7 |
| C27—P2—C8 | 104.04 (10) | C19—C20—H20A | 119.7 |
| C6—O1—C14 | 118.5 (2) | C26—C21—C22 | 117.8 (2) |
| C12—O2—C13 | 119.0 (2) | C26—C21—P1 | 124.26 (18) |
| C2—C1—C6 | 118.86 (19) | C22—C21—P1 | 117.94 (18) |

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| C2—C1—C7 | 122.79 (18) | C23—C22—C21 | 120.9 (2) |
| C6—C1—C7 | 118.35 (19) | C23—C22—H22A | 119.5 |
| C1—C2—C3 | 119.3 (2) | C21—C22—H22A | 119.5 |
| C1—C2—P1 | 122.21 (15) | C24—C23—C22 | 119.9 (3) |
| C3—C2—P1 | 118.48 (18) | C24—C23—H23A | 120.1 |
| C4—C3—C2 | 120.6 (2) | C22—C23—H23A | 120.1 |
| C4—C3—H3A | 119.7 | C25—C24—C23 | 120.2 (3) |
| C2—C3—H3A | 119.7 | C25—C24—H24A | 119.9 |
| C3—C4—C5 | 120.8 (2) | C23—C24—H24A | 119.9 |
| C3—C4—H4A | 119.6 | C24—C25—C26 | 120.1 (2) |
| C5—C4—H4A | 119.6 | C24—C25—H25A | 119.9 |
| C6—C5—C4 | 119.3 (2) | C26—C25—H25A | 119.9 |
| C6—C5—H5A | 120.4 | C21—C26—C25 | 121.0 (2) |
| C4—C5—H5A | 120.4 | C21—C26—H26A | 119.5 |
| O1—C6—C5 | 123.9 (2) | C25—C26—H26A | 119.5 |
| O1—C6—C1 | 114.99 (19) | C28—C27—C32 | 119.0 (2) |
| C5—C6—C1 | 121.1 (2) | C28—C27—P2 | 116.79 (17) |
| C8—C7—C12 | 119.0 (2) | C32—C27—P2 | 124.16 (19) |
| C8—C7—C1 | 122.63 (18) | C29—C28—C27 | 120.4 (2) |
| C12—C7—C1 | 118.33 (19) | C29—C28—H28A | 119.8 |
| C7—C8—C9 | 119.4 (2) | C27—C28—H28A | 119.8 |
| C7—C8—P2 | 121.80 (15) | C30—C29—C28 | 120.1 (2) |
| C9—C8—P2 | 118.85 (18) | C30—C29—H29A | 119.9 |
| C10—C9—C8 | 120.2 (2) | C28—C29—H29A | 119.9 |
| C10—C9—H9A | 119.9 | C31—C30—C29 | 120.2 (2) |
| C8—C9—H9A | 119.9 | C31—C30—H30A | 119.9 |
| C9—C10—C11 | 120.9 (2) | C29—C30—H30A | 119.9 |
| C9—C10—H10A | 119.6 | C30—C31—C32 | 120.6 (2) |
| C11—C10—H10A | 119.6 | C30—C31—H31A | 119.7 |
| C10—C11—C12 | 119.8 (2) | C32—C31—H31A | 119.7 |
| C10—C11—H11A | 120.1 | C31—C32—C27 | 119.7 (2) |
| C12—C11—H11A | 120.1 | C31—C32—H32A | 120.1 |
| O2—C12—C11 | 124.0 (2) | C27—C32—H32A | 120.1 |
| O2—C12—C7 | 115.2 (2) | C34—C33—C38 | 118.1 (3) |
| C11—C12—C7 | 120.8 (2) | C34—C33—P2 | 117.6 (3) |
| O2—C13—H13A | 109.5 | C38—C33—P2 | 124.3 (2) |
| O2—C13—H13B | 109.5 | C33—C34—C35 | 120.1 (4) |
| H13A—C13—H13B | 109.5 | C33—C34—H34A | 120.0 |
| O2—C13—H13C | 109.5 | C35—C34—H34A | 120.0 |
| H13A—C13—H13C | 109.5 | C36—C35—C34 | 121.8 (4) |
| H13B—C13—H13C | 109.5 | C36—C35—H35A | 119.1 |
| O1—C14—H14A | 109.5 | C34—C35—H35A | 119.1 |
| O1—C14—H14B | 109.5 | C35—C36—C37 | 119.0 (4) |
| H14A—C14—H14B | 109.5 | C35—C36—H36A | 120.5 |
| O1—C14—H14C | 109.5 | C37—C36—H36A | 120.5 |
| H14A—C14—H14C | 109.5 | C36—C37—C38 | 120.1 (4) |
| H14B—C14—H14C | 109.5 | C36—C37—H37A | 120.0 |
| C16—C15—C20 | 117.4 (2) | C38—C37—H37A | 120.0 |

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| C16—C15—P1 | 124.7 (2) | C33—C38—C37 | 121.0 (3) |
| C20—C15—P1 | 117.90 (19) | C33—C38—H38A | 119.5 |
| C15—C16—C17 | 121.1 (3) | C37—C38—H38A | 119.5 |
| C15—C16—H16A | 119.5 | H2′A—O2′—H2′B | 104.8 |
| C17—C16—H16A | 119.5 | H1′A—O1′—H1′B | 103.5 |
| | | | |
| C6—C1—C2—C3 | 0.0 (3) | C21—P1—C15—C20 | −170.98 (19) |
| C7—C1—C2—C3 | 179.25 (19) | C2—P1—C15—C20 | 76.1 (2) |
| C6—C1—C2—P1 | 178.48 (16) | C20—C15—C16—C17 | −1.6 (6) |
| C7—C1—C2—P1 | −2.3 (3) | P1—C15—C16—C17 | −178.9 (4) |
| O3—P1—C2—C1 | −24.2 (2) | C15—C16—C17—C18 | 1.8 (8) |
| C15—P1—C2—C1 | −145.72 (17) | C16—C17—C18—C19 | −0.6 (7) |
| C21—P1—C2—C1 | 101.98 (18) | C17—C18—C19—C20 | −0.6 (6) |
| O3—P1—C2—C3 | 154.22 (17) | C16—C15—C20—C19 | 0.4 (4) |
| C15—P1—C2—C3 | 32.7 (2) | P1—C15—C20—C19 | 177.9 (2) |
| C21—P1—C2—C3 | −79.55 (19) | C18—C19—C20—C15 | 0.7 (5) |
| C1—C2—C3—C4 | −0.5 (3) | O3—P1—C21—C26 | 133.4 (2) |
| P1—C2—C3—C4 | −178.99 (19) | C15—P1—C21—C26 | −106.0 (2) |
| C2—C3—C4—C5 | 0.9 (4) | C2—P1—C21—C26 | 5.3 (2) |
| C3—C4—C5—C6 | −0.9 (4) | O3—P1—C21—C22 | −46.7 (2) |
| C14—O1—C6—C5 | −10.7 (4) | C15—P1—C21—C22 | 73.9 (2) |
| C14—O1—C6—C1 | 169.9 (2) | C2—P1—C21—C22 | −174.8 (2) |
| C4—C5—C6—O1 | −178.9 (2) | C26—C21—C22—C23 | 1.2 (4) |
| C4—C5—C6—C1 | 0.4 (4) | P1—C21—C22—C23 | −178.7 (3) |
| C2—C1—C6—O1 | 179.40 (19) | C21—C22—C23—C24 | −0.7 (5) |
| C7—C1—C6—O1 | 0.1 (3) | C22—C23—C24—C25 | 0.0 (5) |
| C2—C1—C6—C5 | 0.0 (3) | C23—C24—C25—C26 | 0.2 (4) |
| C7—C1—C6—C5 | −179.3 (2) | C22—C21—C26—C25 | −1.1 (4) |
| C2—C1—C7—C8 | 96.8 (3) | P1—C21—C26—C25 | 178.8 (2) |
| C6—C1—C7—C8 | −84.0 (3) | C24—C25—C26—C21 | 0.4 (4) |
| C2—C1—C7—C12 | −84.4 (3) | O4—P2—C27—C28 | −53.6 (2) |
| C6—C1—C7—C12 | 94.8 (2) | C33—P2—C27—C28 | −175.45 (19) |
| C12—C7—C8—C9 | 0.9 (3) | C8—P2—C27—C28 | 71.3 (2) |
| C1—C7—C8—C9 | 179.64 (19) | O4—P2—C27—C32 | 123.6 (2) |
| C12—C7—C8—P2 | −179.68 (16) | C33—P2—C27—C32 | 1.7 (2) |
| C1—C7—C8—P2 | −0.9 (3) | C8—P2—C27—C32 | −111.5 (2) |
| O4—P2—C8—C7 | −27.9 (2) | C32—C27—C28—C29 | −0.8 (4) |
| C33—P2—C8—C7 | 97.62 (19) | P2—C27—C28—C29 | 176.4 (2) |
| C27—P2—C8—C7 | −149.62 (17) | C27—C28—C29—C30 | 0.3 (4) |
| O4—P2—C8—C9 | 151.54 (17) | C28—C29—C30—C31 | 0.6 (4) |
| C33—P2—C8—C9 | −82.90 (19) | C29—C30—C31—C32 | −0.9 (4) |
| C27—P2—C8—C9 | 29.9 (2) | C30—C31—C32—C27 | 0.2 (4) |
| C7—C8—C9—C10 | 0.1 (3) | C28—C27—C32—C31 | 0.6 (4) |
| P2—C8—C9—C10 | −179.40 (18) | P2—C27—C32—C31 | −176.5 (2) |
| C8—C9—C10—C11 | −0.3 (4) | O4—P2—C33—C34 | −25.2 (3) |
| C9—C10—C11—C12 | −0.5 (4) | C27—P2—C33—C34 | 96.0 (3) |
| C13—O2—C12—C11 | 4.0 (4) | C8—P2—C33—C34 | −153.0 (3) |
| C13—O2—C12—C7 | −175.4 (3) | O4—P2—C33—C38 | 154.3 (2) |

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| C10—C11—C12—O2 | −177.9 (2) | C27—P2—C33—C38 | −84.5 (3) |
| C10—C11—C12—C7 | 1.4 (4) | C8—P2—C33—C38 | 26.4 (3) |
| C8—C7—C12—O2 | 177.8 (2) | C38—C33—C34—C35 | 2.1 (6) |
| C1—C7—C12—O2 | −1.1 (3) | P2—C33—C34—C35 | −178.4 (4) |
| C8—C7—C12—C11 | −1.6 (3) | C33—C34—C35—C36 | −1.9 (8) |
| C1—C7—C12—C11 | 179.5 (2) | C34—C35—C36—C37 | 1.1 (8) |
| O3—P1—C15—C16 | 128.4 (3) | C35—C36—C37—C38 | −0.6 (7) |
| C21—P1—C15—C16 | 6.4 (3) | C34—C33—C38—C37 | −1.6 (5) |
| C2—P1—C15—C16 | −106.5 (3) | P2—C33—C38—C37 | 178.9 (3) |
| O3—P1—C15—C20 | −48.9 (2) | C36—C37—C38—C33 | 0.9 (6) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1′—H1′*A*···O4 | 0.85 | 2.31 | 2.854 (4) | 123 |
| O2′—H2′*A*···O3 | 0.85 | 1.96 | 2.799 (3) | 169 |