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# Methyl 2-diphenylphosphoryloxy-2-azabicyclo[2.2.1]hept-5-ene-3-*exo*-carboxylate

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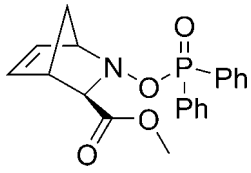
Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.035; $wR$ factor = 0.091; data-to-parameter ratio = 15.8.

In the title compound, $C_{20}H_{20}NO_4P$, the dihedral angle between the phenyl rings is 68.52 (7)°. In the crystal structure, the molecules are linked by a weak C—H···$\pi$(arene) interaction along [010] involving the phenyl CH group and the phenyl rings. There are no further significant intermolecular interactions.

## Related literature

For the preparation of the precursor of the title compound, see: Sousa *et al.* (2008). For related literature about this type of bicyclic compound and their relevance see: Vale *et al.* (2006), Alves *et al.* (2006), Yoda *et al.* (1995).




## Experimental

### Crystal data

$C_{20}H_{20}NO_4P$
$M_r$ = 369.34
Monoclinic, $P2_1/c$
$a$ = 18.4223 (6) Å
$b$ = 8.5522 (3) Å
$c$ = 11.6022 (4) Å
$\beta$ = 97.1810 (10)°
$V$ = 1813.60 (11) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.18 mm$^{-1}$
$T$ = 100 (2) K
0.37 × 0.34 × 0.34 mm

### Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2006)
$T_{min}$ = 0.871, $T_{max}$ = 0.940
14828 measured reflections
3717 independent reflections
3172 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.033

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.091
$S$ = 1.05
3717 reflections
236 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.29 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.41 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C12—H12···$Cg1^{i}$ | 0.95 | 2.77 | 3.566 (2) | 142 |

Symmetry code: (i) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$. $Cg1$ is the centroid of the C15–C20 ring.

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT* (Bruker, 2006); data reduction: *SAINT*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2188).

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# supporting information

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## Methyl 2-diphenylphosphoryloxy-2-azabicyclo[2.2.1]hept-5-ene-3-*exo*-carboxylate

**Carlos A. D. Sousa, M. Luísa C. Vale, José E. Rodríguez-Borges and Xerardo Garcia-Mera**

### S1. Comment

The stucture of the title compound, (I), is shown in Fig. 1. It can be seen the existence of three chiral centers at C2 (*R*), C5 (S) and C6 (*R*). In the crystalline structure, the molecules are linked by a weak C—H···$\pi$ interaction, Fig. 2 [H12-$\pi^{i}$ 2.77 Å, C12-H12-$\pi$ 142°, C12-$\pi$ 3.566 (2) Å, symmetry code: (i) 1-x,1/2+y, 1/2-z] along [010] directions. There are no further significant intermolecular interactions.

### S2. Experimental

The title compound was synthesized from the previously prepared (3*exo*)-2-hydroxy-2-azabicyclo[2.2.1]hept-5-ene-3-carboxylate (Sousa *et al.* 2008). Equimolar amounts of (3*exo*)-2-hydroxy-2-azabicyclo[2.2.1]hept-5-ene-3-carboxylate (0.56 g, 3.3 mmol) and diphenylpfosphinic chloride (0.63 ml, 3.3 mmol), in the presence of 1 eq. of anidrous triethylamine and and a catalytic quantity of DMAP, were let to react overnigth in dichloromethane, at room temperature under argon atmosphere. Water was added and the product was extracted with dichloromethane (3 × 15 ml). The organic layers were dried over sodium sulfate and the solvent was evaporated. The obtained product was purified by flash chromatography (eluent: dichloromethane/diethyl ether 1:1), leading to a light clear yellow oil in 80% yield. Crystals of (I) were made from a slow evaporation of a dichloromethane/hexane solution.

### S3. Refinement

All H atoms were found in a difference Fourier map and placed in geometrically idealized and constrained to ride on their parent atoms [C—H = 0.95–1.00 Å and $U_{iso}$(H) = 1.2 or 1.5$U_{eq}$(C)].

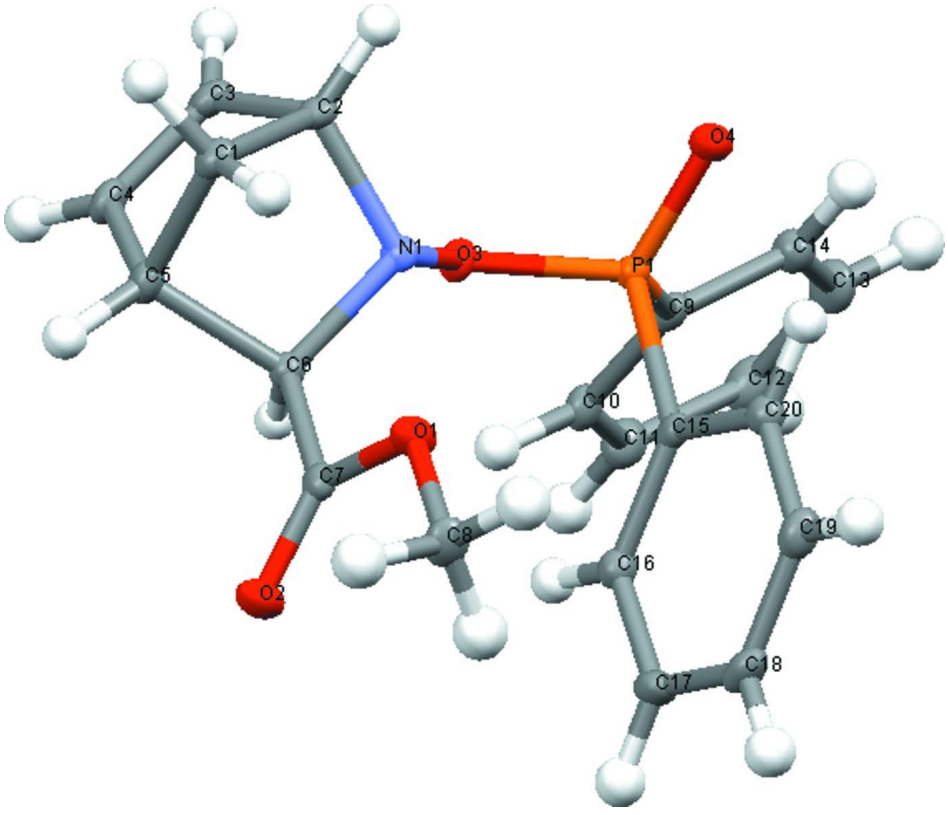


**Figure 1**

A view of (I), showing the three chiral carbons C2, C5 and C6 and the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as spheres of arbitary radius.

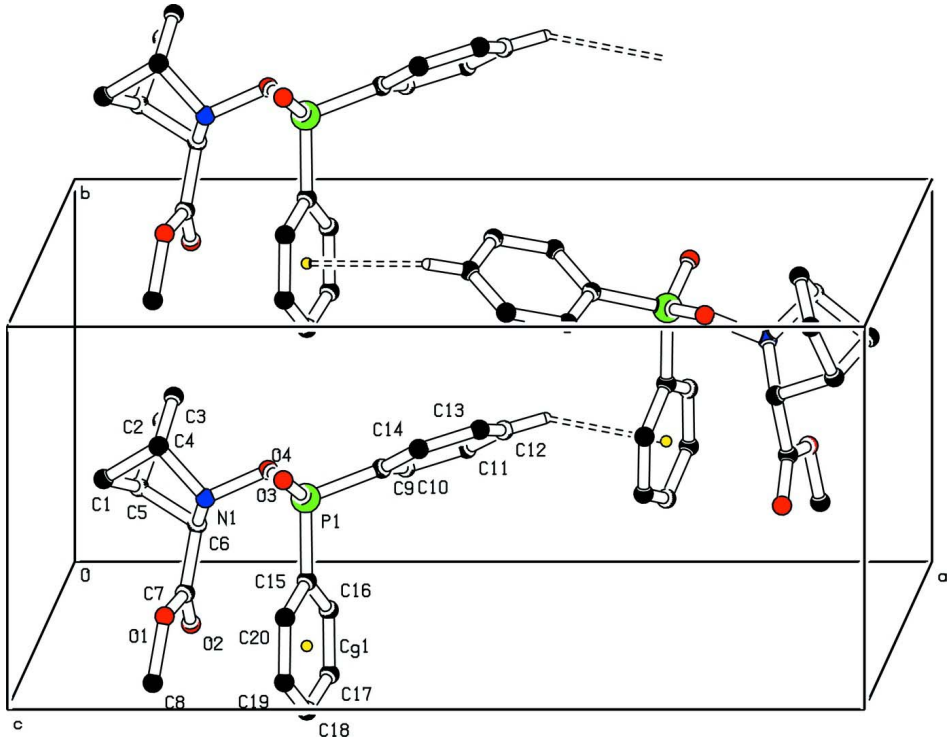


**Figure 2**

Part of the crystal structute of (I) viewed along the c axis. Dashed lines show C—H···$\pi$ (arene) interactions. Only H atoms participating in hydrogen bonding are shown. $\pi$ is the centroid of the ring defined by atoms C15-C20.

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*Crystal data*

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$a$ = 18.4223 (6) Å
$b$ = 8.5522 (3) Å
$c$ = 11.6022 (4) Å
$\beta$ = 97.181 (1)°
$V$ = 1813.60 (11) Å$^3$
$Z$ = 4
$F(000)$ = 776
$D_x$ = 1.353 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 1953 reflections
$\theta$ = 3.1–25.9°
$\mu$ = 0.18 mm$^{-1}$
$T$ = 100 K
Prism, colourless
0.37 × 0.34 × 0.34 mm

*Data collection*

Bruker ApexII CCD area-detector diffractometer
Radiation source: sealed tube
Graphite monochromator
phi and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2006)
$T_{min}$ = 0.871, $T_{max}$ = 0.940
14828 measured reflections
3717 independent reflections
3172 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.033
$\theta_{max}$ = 26.4°, $\theta_{min}$ = 2.2°
$h$ = −23→22
$k$ = 0→10
$l$ = 0→14

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.091$
$S = 1.05$
3717 reflections
236 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
$w = 1/[\sigma^2(F_o^2) + (0.0429P)^2 + 0.8843P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.29$ e Å$^{-3}$
$\Delta\rho_{min} = -0.41$ 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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.04005 (8) | 0.24766 (17) | 0.08174 (13) | 0.0156 (3) |
| H1A | −0.005 | 0.3099 | 0.0614 | 0.019* |
| H1B | 0.0318 | 0.1646 | 0.1381 | 0.019* |
| C2 | 0.10739 (8) | 0.34831 (17) | 0.12086 (13) | 0.0140 (3) |
| H2 | 0.1038 | 0.4159 | 0.1902 | 0.017* |
| C3 | 0.11615 (8) | 0.43459 (18) | 0.00937 (13) | 0.0157 (3) |
| H3 | 0.1328 | 0.539 | 0.003 | 0.019* |
| C4 | 0.09614 (8) | 0.33679 (18) | −0.07788 (13) | 0.0170 (3) |
| H4 | 0.0965 | 0.358 | −0.1582 | 0.02* |
| C5 | 0.07271 (8) | 0.18529 (18) | −0.02579 (13) | 0.0152 (3) |
| H5 | 0.0408 | 0.1144 | −0.0788 | 0.018* |
| C6 | 0.14566 (8) | 0.11298 (17) | 0.03659 (12) | 0.0124 (3) |
| H6 | 0.1849 | 0.1189 | −0.0155 | 0.015* |
| C7 | 0.13407 (7) | −0.05517 (17) | 0.07081 (12) | 0.0126 (3) |
| C8 | 0.10791 (9) | −0.23359 (18) | 0.21479 (14) | 0.0188 (3) |
| H8A | 0.0638 | −0.2759 | 0.1694 | 0.028* |
| H8B | 0.1021 | −0.2364 | 0.2976 | 0.028* |
| H8C | 0.1503 | −0.2968 | 0.201 | 0.028* |
| C9 | 0.37374 (8) | 0.32042 (17) | 0.19955 (13) | 0.0135 (3) |
| C10 | 0.39313 (8) | 0.28107 (18) | 0.09030 (13) | 0.0156 (3) |
| H10 | 0.3592 | 0.2281 | 0.0353 | 0.019* |
| C11 | 0.46227 (8) | 0.31986 (19) | 0.06275 (14) | 0.0196 (3) |
| H11 | 0.4758 | 0.2923 | −0.011 | 0.024* |
| C12 | 0.51155 (8) | 0.3985 (2) | 0.14240 (15) | 0.0220 (4) |
| H12 | 0.559 | 0.4232 | 0.1236 | 0.026* |

| | | | | |
|---|---|---|---|---|
| C13 | 0.49180 (9) | 0.4412 (2) | 0.24924 (15) | 0.0228 (4) |
| H13 | 0.5253 | 0.4974 | 0.3029 | 0.027* |
| C14 | 0.42326 (8) | 0.40209 (18) | 0.27826 (14) | 0.0180 (3) |
| H14 | 0.41 | 0.431 | 0.3519 | 0.022* |
| C15 | 0.29184 (7) | 0.05353 (17) | 0.27096 (13) | 0.0125 (3) |
| C16 | 0.31090 (8) | −0.05303 (18) | 0.18853 (13) | 0.0156 (3) |
| H16 | 0.3218 | −0.017 | 0.1151 | 0.019* |
| C17 | 0.31393 (9) | −0.21192 (18) | 0.21417 (14) | 0.0190 (3) |
| H17 | 0.3284 | −0.2841 | 0.1591 | 0.023* |
| C18 | 0.29590 (8) | −0.26536 (18) | 0.31971 (15) | 0.0200 (3) |
| H18 | 0.2972 | −0.3742 | 0.3364 | 0.024* |
| C19 | 0.27594 (8) | −0.16006 (19) | 0.40103 (14) | 0.0185 (3) |
| H19 | 0.2629 | −0.197 | 0.4729 | 0.022* |
| C20 | 0.27503 (8) | −0.00078 (18) | 0.37771 (13) | 0.0149 (3) |
| H20 | 0.2629 | 0.0713 | 0.4346 | 0.018* |
| N1 | 0.16332 (6) | 0.21932 (14) | 0.13812 (11) | 0.0116 (3) |
| O1 | 0.11889 (6) | −0.07336 (12) | 0.17981 (9) | 0.0166 (2) |
| O2 | 0.13582 (6) | −0.16137 (12) | 0.00278 (9) | 0.0190 (2) |
| O3 | 0.23563 (5) | 0.28855 (12) | 0.12671 (9) | 0.0131 (2) |
| O4 | 0.27071 (6) | 0.34776 (12) | 0.34900 (9) | 0.0158 (2) |
| P1 | 0.288697 (19) | 0.26111 (4) | 0.24650 (3) | 0.01112 (11) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0105 (7) | 0.0152 (8) | 0.0210 (8) | 0.0011 (6) | 0.0016 (6) | 0.0022 (6) |
| C2 | 0.0121 (7) | 0.0126 (7) | 0.0175 (7) | 0.0026 (6) | 0.0025 (6) | −0.0004 (6) |
| C3 | 0.0118 (7) | 0.0132 (7) | 0.0218 (8) | 0.0026 (6) | 0.0016 (6) | 0.0041 (6) |
| C4 | 0.0154 (7) | 0.0179 (8) | 0.0172 (7) | 0.0029 (6) | 0.0000 (6) | 0.0060 (6) |
| C5 | 0.0130 (7) | 0.0149 (8) | 0.0167 (7) | −0.0011 (6) | −0.0020 (6) | 0.0021 (6) |
| C6 | 0.0112 (7) | 0.0123 (7) | 0.0135 (7) | −0.0005 (6) | 0.0008 (5) | 0.0000 (6) |
| C7 | 0.0081 (6) | 0.0149 (7) | 0.0145 (7) | 0.0003 (6) | −0.0001 (5) | −0.0004 (6) |
| C8 | 0.0206 (8) | 0.0145 (8) | 0.0209 (8) | −0.0045 (6) | 0.0010 (6) | 0.0058 (6) |
| C9 | 0.0106 (7) | 0.0116 (7) | 0.0180 (7) | 0.0001 (6) | 0.0005 (6) | 0.0035 (6) |
| C10 | 0.0133 (7) | 0.0161 (8) | 0.0169 (7) | −0.0018 (6) | −0.0003 (6) | 0.0041 (6) |
| C11 | 0.0170 (8) | 0.0232 (8) | 0.0192 (8) | 0.0001 (6) | 0.0047 (6) | 0.0046 (6) |
| C12 | 0.0131 (7) | 0.0242 (9) | 0.0289 (9) | −0.0026 (6) | 0.0031 (6) | 0.0075 (7) |
| C13 | 0.0152 (8) | 0.0235 (9) | 0.0283 (9) | −0.0056 (7) | −0.0031 (7) | −0.0009 (7) |
| C14 | 0.0157 (7) | 0.0174 (8) | 0.0205 (8) | −0.0009 (6) | 0.0002 (6) | −0.0009 (6) |
| C15 | 0.0086 (7) | 0.0114 (7) | 0.0165 (7) | −0.0001 (5) | −0.0021 (5) | 0.0011 (6) |
| C16 | 0.0139 (7) | 0.0163 (8) | 0.0162 (7) | 0.0004 (6) | 0.0008 (6) | 0.0004 (6) |
| C17 | 0.0188 (8) | 0.0145 (8) | 0.0226 (8) | 0.0031 (6) | −0.0022 (6) | −0.0060 (6) |
| C18 | 0.0166 (8) | 0.0120 (8) | 0.0296 (9) | −0.0001 (6) | −0.0043 (7) | 0.0042 (7) |
| C19 | 0.0151 (7) | 0.0198 (8) | 0.0202 (8) | −0.0022 (6) | 0.0002 (6) | 0.0068 (6) |
| C20 | 0.0121 (7) | 0.0154 (8) | 0.0169 (7) | −0.0008 (6) | 0.0008 (6) | −0.0007 (6) |
| N1 | 0.0071 (6) | 0.0117 (6) | 0.0161 (6) | −0.0022 (5) | 0.0017 (5) | −0.0004 (5) |
| O1 | 0.0224 (6) | 0.0119 (5) | 0.0163 (5) | −0.0032 (4) | 0.0049 (4) | 0.0011 (4) |
| O2 | 0.0245 (6) | 0.0139 (6) | 0.0189 (6) | −0.0012 (4) | 0.0038 (5) | −0.0031 (5) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O3 | 0.0079 (5) | 0.0142 (5) | 0.0168 (5) | −0.0033 (4) | 0.0003 (4) | 0.0029 (4) |
| O4 | 0.0165 (5) | 0.0135 (5) | 0.0177 (5) | −0.0003 (4) | 0.0030 (4) | −0.0014 (4) |
| P1 | 0.00981 (19) | 0.01024 (19) | 0.0131 (2) | −0.00066 (14) | 0.00083 (14) | 0.00053 (14) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—C2 | 1.531 (2) | C10—C11 | 1.392 (2) |
| C1—C5 | 1.546 (2) | C10—H10 | 0.95 |
| C1—H1A | 0.99 | C11—C12 | 1.386 (2) |
| C1—H1B | 0.99 | C11—H11 | 0.95 |
| C2—N1 | 1.5057 (18) | C12—C13 | 1.384 (2) |
| C2—C3 | 1.515 (2) | C12—H12 | 0.95 |
| C2—H2 | 1 | C13—C14 | 1.388 (2) |
| C3—C4 | 1.329 (2) | C13—H13 | 0.95 |
| C3—H3 | 0.95 | C14—H14 | 0.95 |
| C4—C5 | 1.515 (2) | C15—C20 | 1.393 (2) |
| C4—H4 | 0.95 | C15—C16 | 1.397 (2) |
| C5—C6 | 1.571 (2) | C15—P1 | 1.7976 (15) |
| C5—H5 | 1 | C16—C17 | 1.391 (2) |
| C6—N1 | 1.4915 (18) | C16—H16 | 0.95 |
| C6—C7 | 1.514 (2) | C17—C18 | 1.386 (2) |
| C6—H6 | 1 | C17—H17 | 0.95 |
| C7—O2 | 1.2064 (18) | C18—C19 | 1.387 (2) |
| C7—O1 | 1.3379 (17) | C18—H18 | 0.95 |
| C8—O1 | 1.4505 (18) | C19—C20 | 1.389 (2) |
| C8—H8A | 0.98 | C19—H19 | 0.95 |
| C8—H8B | 0.98 | C20—H20 | 0.95 |
| C8—H8C | 0.98 | N1—O3 | 1.4786 (15) |
| C9—C14 | 1.395 (2) | O3—P1 | 1.6133 (10) |
| C9—C10 | 1.400 (2) | O4—P1 | 1.4737 (11) |
| C9—P1 | 1.7950 (15) | | |
| | | | |
| C2—C1—C5 | 92.89 (11) | C11—C10—H10 | 120.2 |
| C2—C1—H1A | 113.1 | C9—C10—H10 | 120.2 |
| C5—C1—H1A | 113.1 | C12—C11—C10 | 120.30 (15) |
| C2—C1—H1B | 113.1 | C12—C11—H11 | 119.8 |
| C5—C1—H1B | 113.1 | C10—C11—H11 | 119.8 |
| H1A—C1—H1B | 110.5 | C13—C12—C11 | 120.11 (14) |
| N1—C2—C3 | 109.04 (11) | C13—C12—H12 | 119.9 |
| N1—C2—C1 | 98.23 (11) | C11—C12—H12 | 119.9 |
| C3—C2—C1 | 100.92 (12) | C12—C13—C14 | 120.20 (15) |
| N1—C2—H2 | 115.5 | C12—C13—H13 | 119.9 |
| C3—C2—H2 | 115.5 | C14—C13—H13 | 119.9 |
| C1—C2—H2 | 115.5 | C13—C14—C9 | 120.14 (15) |
| C4—C3—C2 | 107.14 (13) | C13—C14—H14 | 119.9 |
| C4—C3—H3 | 126.4 | C9—C14—H14 | 119.9 |
| C2—C3—H3 | 126.4 | C20—C15—C16 | 119.59 (14) |
| C3—C4—C5 | 107.46 (13) | C20—C15—P1 | 117.59 (11) |

| | | | |
|---|---|---|---|
| C3—C4—H4 | 126.3 | C16—C15—P1 | 122.82 (11) |
| C5—C4—H4 | 126.3 | C17—C16—C15 | 119.85 (14) |
| C4—C5—C1 | 100.71 (12) | C17—C16—H16 | 120.1 |
| C4—C5—C6 | 104.50 (12) | C15—C16—H16 | 120.1 |
| C1—C5—C6 | 99.25 (11) | C18—C17—C16 | 120.24 (15) |
| C4—C5—H5 | 116.6 | C18—C17—H17 | 119.9 |
| C1—C5—H5 | 116.6 | C16—C17—H17 | 119.9 |
| C6—C5—H5 | 116.6 | C17—C18—C19 | 120.02 (14) |
| N1—C6—C7 | 113.33 (11) | C17—C18—H18 | 120 |
| N1—C6—C5 | 102.32 (11) | C19—C18—H18 | 120 |
| C7—C6—C5 | 110.74 (12) | C18—C19—C20 | 120.14 (15) |
| N1—C6—H6 | 110.1 | C18—C19—H19 | 119.9 |
| C7—C6—H6 | 110.1 | C20—C19—H19 | 119.9 |
| C5—C6—H6 | 110.1 | C19—C20—C15 | 120.11 (14) |
| O2—C7—O1 | 123.83 (14) | C19—C20—H20 | 119.9 |
| O2—C7—C6 | 121.82 (13) | C15—C20—H20 | 119.9 |
| O1—C7—C6 | 114.29 (12) | O3—N1—C6 | 106.47 (10) |
| O1—C8—H8A | 109.5 | O3—N1—C2 | 107.69 (10) |
| O1—C8—H8B | 109.5 | C6—N1—C2 | 105.28 (11) |
| H8A—C8—H8B | 109.5 | C7—O1—C8 | 115.30 (12) |
| O1—C8—H8C | 109.5 | N1—O3—P1 | 108.68 (8) |
| H8A—C8—H8C | 109.5 | O4—P1—O3 | 116.67 (6) |
| H8B—C8—H8C | 109.5 | O4—P1—C9 | 113.30 (7) |
| C14—C9—C10 | 119.54 (13) | O3—P1—C9 | 98.94 (6) |
| C14—C9—P1 | 117.84 (12) | O4—P1—C15 | 112.04 (7) |
| C10—C9—P1 | 122.54 (11) | O3—P1—C15 | 106.49 (6) |
| C11—C10—C9 | 119.67 (14) | C9—P1—C15 | 108.35 (7) |

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| C5—C1—C2—N1 | −60.85 (12) | C18—C19—C20—C15 | 2.1 (2) |
| C5—C1—C2—C3 | 50.47 (12) | C16—C15—C20—C19 | −1.2 (2) |
| N1—C2—C3—C4 | 68.17 (15) | P1—C15—C20—C19 | 179.43 (11) |
| C1—C2—C3—C4 | −34.58 (15) | C7—C6—N1—O3 | 120.70 (12) |
| C2—C3—C4—C5 | 0.88 (16) | C5—C6—N1—O3 | −120.03 (11) |
| C3—C4—C5—C1 | 32.73 (15) | C7—C6—N1—C2 | −125.14 (12) |
| C3—C4—C5—C6 | −69.86 (15) | C5—C6—N1—C2 | −5.87 (13) |
| C2—C1—C5—C4 | −49.87 (12) | C3—C2—N1—O3 | 51.25 (14) |
| C2—C1—C5—C6 | 56.93 (12) | C1—C2—N1—O3 | 155.87 (10) |
| C4—C5—C6—N1 | 71.20 (13) | C3—C2—N1—C6 | −62.06 (14) |
| C1—C5—C6—N1 | −32.48 (13) | C1—C2—N1—C6 | 42.56 (13) |
| C4—C5—C6—C7 | −167.72 (12) | O2—C7—O1—C8 | 3.0 (2) |
| C1—C5—C6—C7 | 88.59 (13) | C6—C7—O1—C8 | −179.72 (12) |
| N1—C6—C7—O2 | −162.95 (13) | C6—N1—O3—P1 | −127.24 (9) |
| C5—C6—C7—O2 | 82.74 (17) | C2—N1—O3—P1 | 120.25 (10) |
| N1—C6—C7—O1 | 19.76 (17) | N1—O3—P1—O4 | −68.47 (10) |
| C5—C6—C7—O1 | −94.56 (14) | N1—O3—P1—C9 | 169.71 (9) |
| C14—C9—C10—C11 | 1.8 (2) | N1—O3—P1—C15 | 57.44 (9) |
| P1—C9—C10—C11 | −174.85 (12) | C14—C9—P1—O4 | 18.47 (14) |
| C9—C10—C11—C12 | −0.7 (2) | C10—C9—P1—O4 | −164.79 (12) |

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| C10—C11—C12—C13 | −1.0 (2) | C14—C9—P1—O3 | 142.70 (12) |
| C11—C12—C13—C14 | 1.6 (3) | C10—C9—P1—O3 | −40.56 (14) |
| C12—C13—C14—C9 | −0.4 (2) | C14—C9—P1—C15 | −106.51 (13) |
| C10—C9—C14—C13 | −1.3 (2) | C10—C9—P1—C15 | 70.23 (14) |
| P1—C9—C14—C13 | 175.54 (12) | C20—C15—P1—O4 | 2.24 (13) |
| C20—C15—C16—C17 | −0.9 (2) | C16—C15—P1—O4 | −177.14 (11) |
| P1—C15—C16—C17 | 178.48 (11) | C20—C15—P1—O3 | −126.43 (11) |
| C15—C16—C17—C18 | 2.0 (2) | C16—C15—P1—O3 | 54.19 (13) |
| C16—C17—C18—C19 | −1.1 (2) | C20—C15—P1—C9 | 127.97 (11) |
| C17—C18—C19—C20 | −1.0 (2) | C16—C15—P1—C9 | −51.41 (14) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C12—H12···*Cg*1$^{i}$ | 0.95 | 2.77 | 3.566 (2) | 142 |

Symmetry code: (i) −*x*+1, *y*+1/2, −*z*+1/2.