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# *tert*-Butyl *N*-(phosphinoyloxy)carbamate

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The title compound, $C_{17}H_{20}NO_4P$, contains pyramidal N atoms and adopts similar conformations in its three independent molecules (*A*, *B* and *C*). Molecules *A* and *B* form a dimer in the crystal structure by way of a pair of N—H···O hydrogen bonds, as does *C* with its inversion-generated partner.

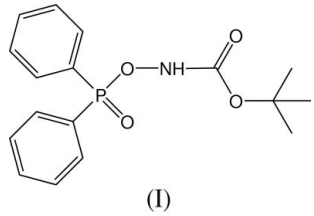
**Key indicators**

Single-crystal X-ray study
*T* = 120 K
Mean σ(C–C) = 0.003 Å
*R* factor = 0.043
*wR* factor = 0.125
Data-to-parameter ratio = 18.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

## Comment

*N*-Alkyl-*O*-(diphenylphosphoryl)hydroxylamines have been reported to be potentially useful electrophilic aminating agents, for example in Schmidt reactions (Harger, 1981; Boche & Schrott, 1982*a*,*b*; Boche *et al.*, 1988). It was not unreasonable to infer that such systems would act as nitrogen peracid equivalents and could be widely applicable reagents in organic synthesis (Masse & Sturtz, 1988*a*,*b*). In the course of exploring this possibility, we prepared the title compound, (I).




(I)

The asymmetric unit of (I) comprises three independent molecules (Fig. 1, Table 1). Molecules *A* and *B* are related by an approximate local inversion centre and are linked into a dimer by a pair of N—H···O hydrogen bonds (Table 2). The third independent molecule (*C*) forms a similar hydrogen-bonded dimer with its symmetry-equivalent partner, generated by a crystallographic inversion centre. The geometries and conformations of all three molecules are essentially the same. The P atoms have tetrahedral geometry. The P—O single bond in (I) is substantially longer [mean 1.616 (2) Å] than in diphenylphosphinic acid $Ph_2P(=O)OH$ [1.550 (1) Å; Lyssenko *et al.*, 2002] or in $Ph_2P(=O)OBu$-*t* [1.569 (3) Å; Grice *et al.*, 2004], but comparable to the values in $Ph_2P(=O)ONEt_2$ [1.599 (1) Å; Spek & Veldman, 1999] or $Ph_2P(=O)ON=C(Cl)Pr$-*i* [1.613 (3) Å; Martynov *et al.*, 1988]. It is noteworthy that the difference between the P=O and the P—O*R* bond lengths is increased from 0.048 (1) Å in $Ph_2P(=O)OH$ and 0.093 (4) Å in $Ph_2P(=O)OBu$-*t* to 0.136 (2) Å in (I), 0.128 (1) Å in $Ph_2P(=O)ONEt_2$ and 0.151 (4) Å in $Ph_2P(=O)ON=C(Cl)Pr$-*i*. Thus. we may conclude that bonding to an N atom reduces the additional π-component of the (formally) single P—O bond. The P—C(Ph) distances in (I) [mean 1.786 (2) Å] are as expected.

The N atoms are substantially pyramidal ($sp^3$) in all three independent molecules in (I): atoms H1*N*, H2*N* and H3*N*

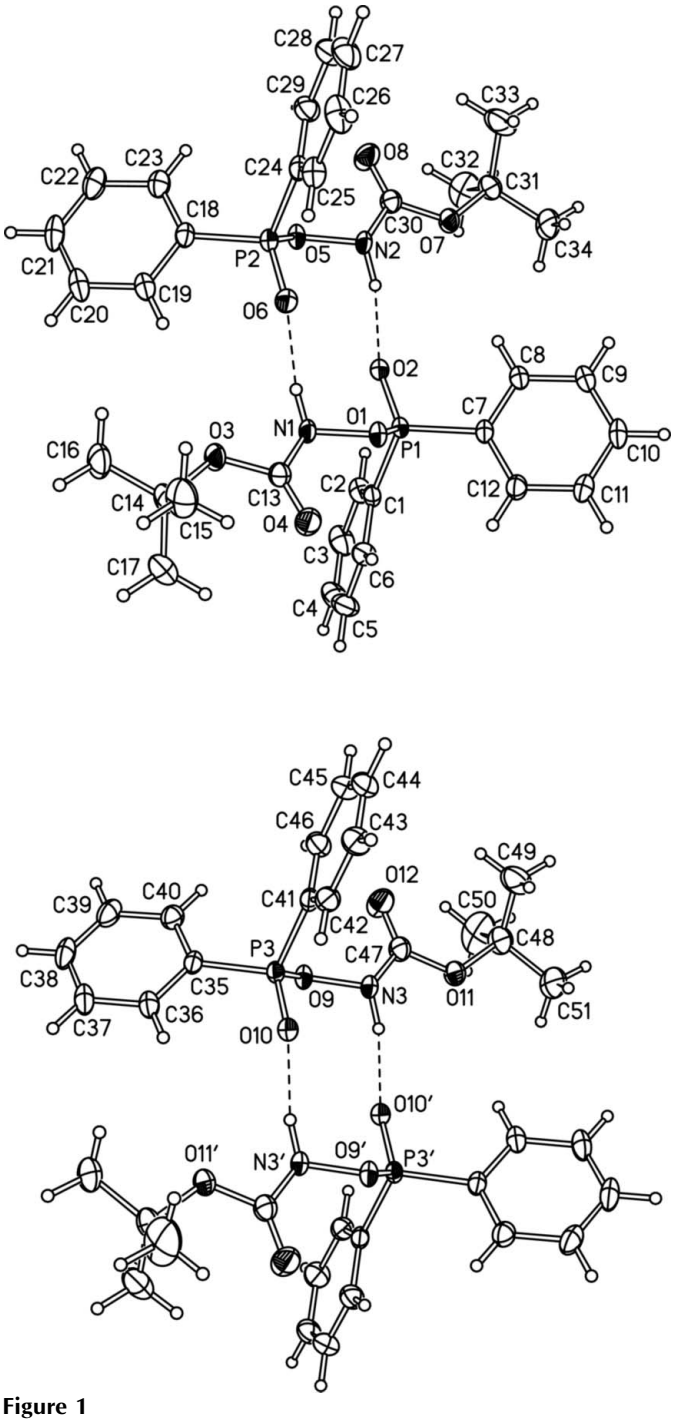



**Figure 1**
The structures of the constituent molecules of (I) showing 50% displacement ellipsoids (arbitrary spheres for the H atoms) and hydrogen bonding (dashed lines) interactions. Top: molecules *A* and *B*. Bottom: molecule *C* and its symmetry equivalent by inversion (symmetry code for primed atom labels: $1-x, 2-y, 1-z$).

deviate from the corresponding O—N—C planes by 0.68 (2), 0.70 (2) and 0.63 (2) Å, respectively (see also Table 1). Similar geometry has been observed in TosOONHC(═O)OBu-*t* (Tos = *p*-toluenesulfonyl; Boche *et al.*, 1994; Blatch *et al.*, 2006) and in $(O_2N)Cl_2C_6H_2$—ONHC(═O)OBu-*t* (Boyles *et al.*, 2002), whereas in $Me_3ONHC(=O)OBu$-*t* the N atom has a planar configuration (Mortl *et al.*, 1995).

## Experimental

A solution of *t*-BuOC(O)NHOH (6.39 g, 0.048 mol) in an anhydrous mixture of $CH_2Cl_2$ (160 ml) and toluene (320 ml) was cooled to 273 K under argon, treated with 1.2 equivalents of triethylamine (6.021 g, 0.0595 mol) and stirred for 30 min. Diphenylphosphinyl chloride (9.16 ml, 0.048 mol) was added; the solution was warmed to room temperature, stirred for another 72 h and evaporated. The solid residue was recrystallized from $CH_2Cl_2$ to give colourless crystals of (I) (12.14 g, 76%). Mass-spectrum, *m*/*z* ES (+): 233.3 ($Ph_2PO_2NH_2$, 82%) and 356.33 (45%). The melting point was not measured because of the thermal instability and explosive nature of (I).

*Crystal data*

$C_{17}H_{20}NO_4P$
$M_r = 333.31$
Triclinic, $P\overline{1}$
$a = 12.181$ (1) Å
$b = 13.734$ (1) Å
$c = 16.532$ (1) Å
$\alpha = 101.10$ (1)°
$\beta = 96.77$ (1)°
$\gamma = 92.93$ (1)°
$V = 2687.3$ (3) Å$^3$
$Z = 6$
$D_x = 1.236$ Mg m$^{-3}$
Mo $K\alpha$ radiation
$\mu = 0.17$ mm$^{-1}$
$T = 120$ (2) K
Block, colourless
0.23 × 0.21 × 0.19 mm

*Data collection*

Bruker SMART 6K CCD diffractometer
$\omega$ scans
Absorption correction: none
11843 measured reflections
11796 independent reflections
9155 reflections with $I > 2\sigma(I)$
$R_{int} = 0.040$
$\theta_{max} = 30.0°$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.125$
$S = 1.00$
11796 reflections
652 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0824P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.55$ e Å$^{-3}$
$\Delta\rho_{min} = -0.53$ e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| P1—O2 | 1.4808 (9) | P2—O5 | 1.6162 (11) |
| P1—O1 | 1.6144 (11) | P3—O10 | 1.4813 (10) |
| P2—O6 | 1.4783 (10) | P3—O9 | 1.6187 (11) |
| | | | |
| C13—N1—O1 | 111.09 (10) | O5—N2—H2N | 109.6 (11) |
| C13—N1—H1N | 114.4 (11) | C47—N3—O9 | 111.97 (11) |
| O1—N1—H1N | 107.8 (11) | C47—N3—H3N | 117.6 (12) |
| C30—N2—O5 | 111.24 (10) | O9—N3—H3N | 110.6 (11) |
| C30—N2—H2N | 115.3 (12) | | |

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

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1N···O6 | 0.886 (17) | 1.836 (17) | 2.7138 (16) | 170.0 (15) |
| N2—H2N···O2 | 0.95 (2) | 1.83 (2) | 2.7688 (16) | 168.4 (17) |
| N3—H3N···O10$^{i}$ | 0.923 (18) | 1.855 (19) | 2.7666 (16) | 169.0 (16) |

Symmetry code: (i) $-x+1, -y+2, -z+1$.

The amino H atoms were located in difference maps and their positions and $U_{iso}$ values were freely refined. The phenyl H atoms were treated as riding on their C atoms with C—H = 0.95 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. The methyl groups were refined as rigid bodies (C—H = 0.98 Å) allowed to rotate around their linking C—C bonds, with a common refined $U_{iso}$ for the three H atoms.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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# supporting information

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*Crystal data*

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Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 12.181$ (1) Å
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$\alpha = 101.10$ (1)°
$\beta = 96.77$ (1)°
$\gamma = 92.93$ (1)°
$V = 2687.3$ (3) Å$^3$
$Z = 6$
$F(000) = 1056$
$D_x = 1.236$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5811 reflections
$\theta = 2.2$–$34.4$°
$\mu = 0.17$ mm$^{-1}$
$T = 120$ K
Block, colourless
0.23 × 0.21 × 0.19 mm

*Data collection*

Bruker SMART CCD 6K diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8 pixels mm$^{-1}$
$\omega$ scans
11843 measured reflections
11796 independent reflections
9155 reflections with $I > 2\sigma(I)$
$R_{int} = 0.040$
$\theta_{max} = 30.0°$, $\theta_{min} = 1.5°$
$h = -16 \rightarrow 13$
$k = -16 \rightarrow 19$
$l = -23 \rightarrow 16$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.125$
$S = 1.00$
11796 reflections
652 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0824P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.55$ e Å$^{-3}$
$\Delta\rho_{min} = -0.53$ e Å$^{-3}$

*Special details*

**Experimental**. The data collection nominally covered a hemisphere of reciprocal Space, by 1 run of $\omega$ scans each set at different $\varphi$ and/or $2\theta$ angles and each scan (10 s exposure) covering 0.3° in $\omega$. Crystal to detector distance 4.85 cm. 1H NMR (200 MHz, CDCl3): $\delta$ 1.39 (s, 9 H, CMe3), 7.44–7.48 (m, 4 H, ArH) 7.53–7.57 (m, 2 H, ArH), 7.94–7.99 (m, 4 H, ArH) and 8.63 (broad s, 1 H, NH). 13 C NMR (100 MHz, CDCl3): $\delta$ 27.9 (CH3), 83.1 (CMe3), 128.4 (ArH), 128.5 (ArH), 129.5 [*ARP*(O)], 132.4 (ArH), 132.8 (ArH) and 156.2 (ArH). 31P NMR (81 MHz, CDCl3): $\delta$ 40.96. Mass-spectrum, m/*z* ES (+): 233.3 (Ph2PO2NH2, 82%) and 356.33 (45%).

**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.

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| P1 | 0.10055 (3) | 0.26988 (3) | 0.27389 (2) | 0.01775 (10) |
| O1 | 0.18559 (8) | 0.36743 (7) | 0.30630 (6) | 0.0210 (2) |
| O2 | 0.08405 (8) | 0.23290 (7) | 0.18273 (6) | 0.0207 (2) |
| O4 | 0.18353 (11) | 0.55106 (8) | 0.39437 (6) | 0.0342 (3) |
| O3 | 0.14394 (10) | 0.60494 (7) | 0.27240 (6) | 0.0274 (3) |
| N1 | 0.14437 (11) | 0.44524 (9) | 0.26621 (7) | 0.0209 (3) |
| H1N | 0.1747 (14) | 0.4421 (12) | 0.2197 (10) | 0.022 (4)* |
| C1 | −0.02620 (12) | 0.30120 (11) | 0.31342 (8) | 0.0210 (3) |
| C2 | −0.12504 (14) | 0.25301 (12) | 0.26970 (10) | 0.0290 (4) |
| H2 | −0.1243 | 0.2066 | 0.2191 | 0.035* |
| C3 | −0.22496 (15) | 0.27315 (14) | 0.30047 (12) | 0.0372 (4) |
| H3 | −0.2926 | 0.2408 | 0.2706 | 0.045* |
| C4 | −0.22558 (16) | 0.33991 (14) | 0.37421 (13) | 0.0411 (5) |
| H4 | −0.2938 | 0.3523 | 0.3954 | 0.049* |
| C5 | −0.12866 (16) | 0.38862 (13) | 0.41741 (11) | 0.0376 (4) |
| H5 | −0.1302 | 0.4349 | 0.4679 | 0.045* |
| C6 | −0.02786 (14) | 0.37012 (12) | 0.38722 (9) | 0.0289 (4) |
| H6 | 0.0392 | 0.4043 | 0.4167 | 0.035* |
| C7 | 0.16770 (12) | 0.18519 (11) | 0.32898 (8) | 0.0196 (3) |
| C8 | 0.20979 (12) | 0.10244 (11) | 0.28261 (9) | 0.0211 (3) |
| H8 | 0.2023 | 0.0926 | 0.2237 | 0.025* |
| C9 | 0.26281 (12) | 0.03471 (11) | 0.32361 (9) | 0.0239 (3) |
| H9 | 0.2920 | −0.0213 | 0.2925 | 0.029* |
| C10 | 0.27309 (13) | 0.04875 (11) | 0.40974 (9) | 0.0261 (4) |
| H10 | 0.3091 | 0.0022 | 0.4373 | 0.031* |
| C11 | 0.23058 (13) | 0.13071 (12) | 0.45561 (9) | 0.0270 (4) |
| H11 | 0.2374 | 0.1400 | 0.5145 | 0.032* |
| C12 | 0.17830 (13) | 0.19906 (11) | 0.41571 (9) | 0.0248 (3) |
| H12 | 0.1497 | 0.2552 | 0.4472 | 0.030* |
| C13 | 0.16195 (13) | 0.53720 (11) | 0.32002 (9) | 0.0230 (3) |
| C14 | 0.14467 (16) | 0.71156 (11) | 0.31001 (10) | 0.0310 (4) |

| | | | | |
|---|---|---|---|---|
| C15 | 0.25948 (18) | 0.74960 (13) | 0.35381 (12) | 0.0452 (5) |
| H151 | 0.3140 | 0.7340 | 0.3150 | 0.055 (4)* |
| H152 | 0.2769 | 0.7176 | 0.4014 | 0.055 (4)* |
| H153 | 0.2619 | 0.8218 | 0.3734 | 0.055 (4)* |
| C16 | 0.11550 (18) | 0.75684 (13) | 0.23410 (11) | 0.0417 (5) |
| H161 | 0.1721 | 0.7435 | 0.1967 | 0.052 (4)* |
| H162 | 0.1124 | 0.8289 | 0.2516 | 0.052 (4)* |
| H163 | 0.0432 | 0.7274 | 0.2050 | 0.052 (4)* |
| C17 | 0.05589 (19) | 0.72724 (14) | 0.36741 (12) | 0.0479 (5) |
| H171 | −0.0145 | 0.6928 | 0.3385 | 0.060 (4)* |
| H172 | 0.0467 | 0.7986 | 0.3836 | 0.060 (4)* |
| H173 | 0.0779 | 0.7004 | 0.4172 | 0.060 (4)* |
| P2 | 0.24725 (3) | 0.38635 (3) | 0.04178 (2) | 0.01939 (10) |
| O5 | 0.16717 (9) | 0.28576 (7) | 0.00742 (6) | 0.0223 (2) |
| O6 | 0.25685 (9) | 0.42489 (8) | 0.13240 (6) | 0.0246 (2) |
| O7 | 0.21538 (10) | 0.05117 (7) | 0.04486 (6) | 0.0263 (3) |
| O8 | 0.19732 (11) | 0.10585 (9) | −0.07767 (7) | 0.0375 (3) |
| N2 | 0.20689 (11) | 0.21047 (9) | 0.05057 (7) | 0.0210 (3) |
| H2N | 0.1694 (16) | 0.2106 (14) | 0.0977 (12) | 0.039 (5)* |
| C18 | 0.17804 (12) | 0.46636 (11) | −0.01717 (9) | 0.0211 (3) |
| C19 | 0.14058 (13) | 0.55285 (11) | 0.02770 (10) | 0.0250 (3) |
| H19 | 0.1523 | 0.5666 | 0.0868 | 0.030* |
| C20 | 0.08625 (14) | 0.61845 (12) | −0.01490 (11) | 0.0310 (4) |
| H20 | 0.0606 | 0.6771 | 0.0153 | 0.037* |
| C21 | 0.06930 (14) | 0.59849 (13) | −0.10092 (11) | 0.0346 (4) |
| H21 | 0.0326 | 0.6438 | −0.1295 | 0.042* |
| C22 | 0.10585 (15) | 0.51247 (14) | −0.14566 (10) | 0.0344 (4) |
| H22 | 0.0937 | 0.4990 | −0.2047 | 0.041* |
| C23 | 0.16013 (13) | 0.44597 (12) | −0.10403 (9) | 0.0281 (4) |
| H23 | 0.1849 | 0.3871 | −0.1345 | 0.034* |
| C24 | 0.38035 (12) | 0.36019 (11) | 0.01099 (9) | 0.0214 (3) |
| C25 | 0.47290 (14) | 0.40633 (12) | 0.06499 (10) | 0.0288 (4) |
| H25 | 0.4639 | 0.4494 | 0.1157 | 0.035* |
| C26 | 0.57862 (15) | 0.38912 (13) | 0.04411 (12) | 0.0378 (4) |
| H26 | 0.6421 | 0.4199 | 0.0809 | 0.045* |
| C27 | 0.59084 (15) | 0.32707 (14) | −0.03009 (13) | 0.0405 (5) |
| H27 | 0.6631 | 0.3162 | −0.0444 | 0.049* |
| C28 | 0.49962 (15) | 0.28076 (14) | −0.08368 (11) | 0.0377 (4) |
| H28 | 0.5092 | 0.2380 | −0.1344 | 0.045* |
| C29 | 0.39359 (14) | 0.29679 (12) | −0.06343 (9) | 0.0295 (4) |
| H29 | 0.3305 | 0.2647 | −0.1000 | 0.035* |
| C30 | 0.20366 (13) | 0.11913 (11) | −0.00321 (9) | 0.0234 (3) |
| C31 | 0.21882 (16) | −0.05516 (12) | 0.00805 (10) | 0.0333 (4) |
| C32 | 0.1093 (2) | −0.09396 (15) | −0.04397 (13) | 0.0540 (6) |
| H321 | 0.0489 | −0.0800 | −0.0101 | 0.079 (5)* |
| H322 | 0.1093 | −0.1659 | −0.0638 | 0.079 (5)* |
| H323 | 0.0986 | −0.0611 | −0.0917 | 0.079 (5)* |
| C33 | 0.3164 (2) | −0.07041 (16) | −0.04069 (15) | 0.0645 (7) |

| | | | | |
|---|---|---|---|---|
| H331 | 0.3839 | −0.0374 | −0.0060 | 0.064 (4)* |
| H332 | 0.3033 | −0.0418 | −0.0906 | 0.064 (4)* |
| H333 | 0.3253 | −0.1418 | −0.0570 | 0.064 (4)* |
| C34 | 0.2348 (2) | −0.10051 (13) | 0.08505 (12) | 0.0458 (5) |
| H341 | 0.1730 | −0.0859 | 0.1173 | 0.060 (4)* |
| H342 | 0.3046 | −0.0723 | 0.1192 | 0.060 (4)* |
| H343 | 0.2371 | −0.1728 | 0.0684 | 0.060 (4)* |
| P3 | 0.58759 (3) | 1.05685 (3) | 0.38744 (2) | 0.01867 (10) |
| O9 | 0.51367 (8) | 0.95294 (7) | 0.35097 (6) | 0.0216 (2) |
| O10 | 0.58442 (8) | 1.09867 (7) | 0.47662 (6) | 0.0214 (2) |
| O11 | 0.56502 (10) | 0.72252 (8) | 0.39681 (7) | 0.0302 (3) |
| O12 | 0.57720 (12) | 0.77787 (9) | 0.27653 (7) | 0.0420 (3) |
| N3 | 0.55055 (11) | 0.88095 (9) | 0.39871 (7) | 0.0215 (3) |
| H3N | 0.5092 (15) | 0.8805 (14) | 0.4420 (11) | 0.034 (5)* |
| C35 | 0.52414 (12) | 1.13203 (11) | 0.32102 (9) | 0.0206 (3) |
| C36 | 0.48845 (13) | 1.22263 (11) | 0.36013 (9) | 0.0252 (3) |
| H36 | 0.4984 | 1.2414 | 0.4191 | 0.030* |
| C37 | 0.43820 (14) | 1.28491 (12) | 0.31187 (11) | 0.0307 (4) |
| H37 | 0.4139 | 1.3464 | 0.3380 | 0.037* |
| C38 | 0.42358 (14) | 1.25756 (14) | 0.22619 (11) | 0.0344 (4) |
| H38 | 0.3891 | 1.3003 | 0.1937 | 0.041* |
| C39 | 0.45897 (14) | 1.16801 (14) | 0.18714 (10) | 0.0340 (4) |
| H39 | 0.4490 | 1.1499 | 0.1282 | 0.041* |
| C40 | 0.50918 (13) | 1.10465 (12) | 0.23440 (9) | 0.0272 (4) |
| H40 | 0.5330 | 1.0432 | 0.2078 | 0.033* |
| C41 | 0.72615 (12) | 1.03390 (11) | 0.36752 (8) | 0.0209 (3) |
| C42 | 0.81257 (13) | 1.08015 (12) | 0.42822 (9) | 0.0260 (3) |
| H42 | 0.7964 | 1.1220 | 0.4775 | 0.031* |
| C43 | 0.92164 (13) | 1.06508 (13) | 0.41655 (10) | 0.0322 (4) |
| H43 | 0.9802 | 1.0958 | 0.4582 | 0.039* |
| C44 | 0.94527 (14) | 1.00512 (13) | 0.34412 (10) | 0.0328 (4) |
| H44 | 1.0202 | 0.9948 | 0.3364 | 0.039* |
| C45 | 0.86058 (14) | 0.96007 (13) | 0.28310 (10) | 0.0328 (4) |
| H45 | 0.8774 | 0.9198 | 0.2332 | 0.039* |
| C46 | 0.75090 (13) | 0.97382 (12) | 0.29476 (9) | 0.0282 (4) |
| H46 | 0.6926 | 0.9423 | 0.2532 | 0.034* |
| C47 | 0.56398 (14) | 0.79048 (11) | 0.34854 (9) | 0.0265 (4) |
| C48 | 0.59500 (19) | 0.62056 (12) | 0.36590 (12) | 0.0426 (5) |
| C49 | 0.7133 (2) | 0.62491 (16) | 0.34395 (15) | 0.0647 (7) |
| H491 | 0.7159 | 0.6575 | 0.2963 | 0.088 (5)* |
| H492 | 0.7366 | 0.5572 | 0.3297 | 0.088 (5)* |
| H493 | 0.7633 | 0.6627 | 0.3917 | 0.088 (5)* |
| C50 | 0.5102 (3) | 0.56827 (15) | 0.29476 (15) | 0.0728 (8) |
| H501 | 0.4358 | 0.5733 | 0.3115 | 0.079 (5)* |
| H502 | 0.5249 | 0.4980 | 0.2799 | 0.079 (5)* |
| H503 | 0.5148 | 0.5996 | 0.2467 | 0.079 (5)* |
| C51 | 0.5900 (2) | 0.57414 (14) | 0.44159 (14) | 0.0577 (7) |
| H511 | 0.5159 | 0.5790 | 0.4590 | 0.068 (4)* |

| | | | | |
|---|---|---|---|---|
| H512 | 0.6453 | 0.6095 | 0.4870 | 0.068 (4)* |
| H513 | 0.6056 | 0.5040 | 0.4279 | 0.068 (4)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| P1 | 0.0196 (2) | 0.01637 (19) | 0.01840 (16) | 0.00518 (17) | 0.00190 (13) | 0.00567 (13) |
| O1 | 0.0230 (6) | 0.0164 (5) | 0.0252 (5) | 0.0036 (5) | 0.0011 (4) | 0.0088 (4) |
| O2 | 0.0238 (6) | 0.0196 (5) | 0.0199 (4) | 0.0056 (5) | 0.0029 (4) | 0.0059 (4) |
| O4 | 0.0536 (8) | 0.0245 (6) | 0.0235 (5) | 0.0055 (6) | 0.0024 (5) | 0.0031 (4) |
| O3 | 0.0420 (7) | 0.0143 (5) | 0.0257 (5) | 0.0028 (5) | 0.0027 (5) | 0.0045 (4) |
| N1 | 0.0293 (7) | 0.0143 (6) | 0.0206 (5) | 0.0050 (6) | 0.0024 (5) | 0.0068 (4) |
| C1 | 0.0230 (8) | 0.0187 (7) | 0.0246 (6) | 0.0066 (7) | 0.0055 (5) | 0.0093 (5) |
| C2 | 0.0270 (9) | 0.0265 (8) | 0.0344 (8) | 0.0044 (8) | 0.0038 (6) | 0.0080 (6) |
| C3 | 0.0231 (9) | 0.0338 (10) | 0.0584 (11) | 0.0032 (9) | 0.0102 (8) | 0.0151 (8) |
| C4 | 0.0325 (10) | 0.0335 (10) | 0.0659 (12) | 0.0109 (9) | 0.0270 (9) | 0.0169 (9) |
| C5 | 0.0482 (12) | 0.0278 (9) | 0.0425 (9) | 0.0108 (9) | 0.0259 (8) | 0.0073 (7) |
| C6 | 0.0338 (9) | 0.0258 (8) | 0.0286 (7) | 0.0063 (8) | 0.0086 (6) | 0.0051 (6) |
| C7 | 0.0187 (7) | 0.0186 (7) | 0.0231 (6) | 0.0032 (7) | 0.0009 (5) | 0.0085 (5) |
| C8 | 0.0201 (8) | 0.0187 (7) | 0.0254 (6) | 0.0030 (7) | 0.0021 (5) | 0.0071 (5) |
| C9 | 0.0189 (8) | 0.0192 (7) | 0.0348 (8) | 0.0037 (7) | 0.0023 (6) | 0.0088 (6) |
| C10 | 0.0201 (8) | 0.0244 (8) | 0.0364 (8) | 0.0030 (7) | −0.0021 (6) | 0.0157 (6) |
| C11 | 0.0264 (8) | 0.0299 (8) | 0.0265 (7) | 0.0032 (7) | −0.0021 (6) | 0.0130 (6) |
| C12 | 0.0256 (8) | 0.0250 (8) | 0.0244 (7) | 0.0048 (7) | 0.0002 (6) | 0.0078 (6) |
| C13 | 0.0263 (8) | 0.0182 (7) | 0.0254 (7) | 0.0014 (7) | 0.0051 (6) | 0.0054 (5) |
| C14 | 0.0455 (11) | 0.0134 (7) | 0.0332 (8) | 0.0051 (8) | 0.0038 (7) | 0.0031 (6) |
| C15 | 0.0547 (13) | 0.0258 (9) | 0.0499 (11) | −0.0040 (10) | −0.0042 (9) | 0.0032 (8) |
| C16 | 0.0629 (14) | 0.0209 (8) | 0.0424 (10) | 0.0056 (9) | 0.0029 (9) | 0.0106 (7) |
| C17 | 0.0632 (14) | 0.0338 (10) | 0.0518 (11) | 0.0211 (10) | 0.0206 (10) | 0.0083 (8) |
| P2 | 0.0207 (2) | 0.0181 (2) | 0.02074 (16) | 0.00477 (18) | 0.00170 (13) | 0.00693 (13) |
| O5 | 0.0228 (6) | 0.0195 (5) | 0.0265 (5) | 0.0044 (5) | 0.0007 (4) | 0.0101 (4) |
| O6 | 0.0302 (6) | 0.0220 (5) | 0.0228 (5) | 0.0041 (5) | 0.0044 (4) | 0.0060 (4) |
| O7 | 0.0382 (7) | 0.0150 (5) | 0.0262 (5) | 0.0041 (5) | 0.0048 (4) | 0.0045 (4) |
| O8 | 0.0616 (9) | 0.0279 (6) | 0.0230 (5) | 0.0044 (6) | 0.0055 (5) | 0.0044 (4) |
| N2 | 0.0278 (7) | 0.0155 (6) | 0.0220 (5) | 0.0047 (6) | 0.0034 (5) | 0.0088 (4) |
| C18 | 0.0173 (7) | 0.0204 (7) | 0.0276 (7) | 0.0009 (7) | 0.0017 (5) | 0.0108 (6) |
| C19 | 0.0198 (8) | 0.0199 (8) | 0.0367 (8) | 0.0026 (7) | 0.0025 (6) | 0.0098 (6) |
| C20 | 0.0224 (9) | 0.0208 (8) | 0.0515 (10) | 0.0034 (8) | −0.0006 (7) | 0.0139 (7) |
| C21 | 0.0245 (9) | 0.0302 (9) | 0.0526 (10) | 0.0003 (8) | −0.0067 (7) | 0.0245 (8) |
| C22 | 0.0311 (9) | 0.0400 (10) | 0.0340 (8) | 0.0011 (9) | −0.0067 (7) | 0.0188 (7) |
| C23 | 0.0270 (9) | 0.0288 (8) | 0.0297 (7) | 0.0039 (8) | −0.0007 (6) | 0.0108 (6) |
| C24 | 0.0219 (8) | 0.0192 (7) | 0.0252 (6) | 0.0034 (7) | 0.0016 (5) | 0.0102 (5) |
| C25 | 0.0265 (9) | 0.0251 (8) | 0.0347 (8) | 0.0009 (8) | −0.0022 (6) | 0.0100 (6) |
| C26 | 0.0214 (9) | 0.0312 (10) | 0.0607 (12) | −0.0020 (8) | −0.0033 (8) | 0.0152 (8) |
| C27 | 0.0235 (9) | 0.0393 (11) | 0.0653 (12) | 0.0088 (9) | 0.0141 (8) | 0.0204 (9) |
| C28 | 0.0336 (10) | 0.0390 (10) | 0.0439 (10) | 0.0122 (9) | 0.0139 (8) | 0.0090 (8) |
| C29 | 0.0275 (9) | 0.0328 (9) | 0.0283 (7) | 0.0083 (8) | 0.0028 (6) | 0.0051 (6) |
| C30 | 0.0245 (8) | 0.0201 (7) | 0.0257 (7) | 0.0007 (7) | 0.0031 (6) | 0.0052 (5) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C31 | 0.0506 (11) | 0.0162 (7) | 0.0346 (8) | 0.0081 (8) | 0.0127 (7) | 0.0034 (6) |
| C32 | 0.0709 (16) | 0.0287 (10) | 0.0549 (12) | −0.0107 (11) | −0.0033 (11) | 0.0008 (9) |
| C33 | 0.0868 (18) | 0.0432 (12) | 0.0805 (16) | 0.0340 (13) | 0.0522 (14) | 0.0213 (11) |
| C34 | 0.0753 (15) | 0.0216 (9) | 0.0448 (10) | 0.0126 (10) | 0.0128 (10) | 0.0122 (7) |
| P3 | 0.0186 (2) | 0.01860 (19) | 0.02017 (16) | 0.00437 (17) | 0.00114 (13) | 0.00722 (13) |
| O9 | 0.0215 (6) | 0.0196 (5) | 0.0252 (5) | 0.0028 (5) | −0.0006 (4) | 0.0098 (4) |
| O10 | 0.0218 (5) | 0.0215 (5) | 0.0219 (5) | 0.0041 (5) | 0.0022 (4) | 0.0067 (4) |
| O11 | 0.0423 (7) | 0.0173 (5) | 0.0343 (6) | 0.0073 (5) | 0.0120 (5) | 0.0078 (4) |
| O12 | 0.0706 (10) | 0.0278 (6) | 0.0289 (6) | 0.0067 (7) | 0.0131 (6) | 0.0042 (5) |
| N3 | 0.0261 (7) | 0.0170 (6) | 0.0239 (6) | 0.0046 (6) | 0.0038 (5) | 0.0090 (5) |
| C35 | 0.0161 (7) | 0.0222 (7) | 0.0259 (7) | 0.0008 (7) | 0.0012 (5) | 0.0121 (5) |
| C36 | 0.0219 (8) | 0.0223 (8) | 0.0327 (7) | 0.0031 (7) | −0.0002 (6) | 0.0106 (6) |
| C37 | 0.0233 (9) | 0.0239 (8) | 0.0481 (10) | 0.0021 (8) | −0.0002 (7) | 0.0180 (7) |
| C38 | 0.0241 (9) | 0.0386 (10) | 0.0465 (10) | 0.0015 (8) | −0.0030 (7) | 0.0284 (8) |
| C39 | 0.0282 (9) | 0.0477 (11) | 0.0302 (8) | 0.0017 (9) | −0.0015 (6) | 0.0213 (7) |
| C40 | 0.0239 (8) | 0.0325 (9) | 0.0267 (7) | 0.0032 (8) | 0.0016 (6) | 0.0105 (6) |
| C41 | 0.0205 (8) | 0.0208 (7) | 0.0236 (6) | 0.0038 (7) | 0.0024 (5) | 0.0100 (5) |
| C42 | 0.0239 (8) | 0.0286 (8) | 0.0262 (7) | 0.0022 (7) | 0.0030 (6) | 0.0069 (6) |
| C43 | 0.0205 (8) | 0.0391 (10) | 0.0365 (8) | 0.0010 (8) | 0.0006 (6) | 0.0088 (7) |
| C44 | 0.0233 (9) | 0.0362 (10) | 0.0432 (9) | 0.0085 (8) | 0.0104 (7) | 0.0135 (7) |
| C45 | 0.0321 (9) | 0.0342 (9) | 0.0348 (8) | 0.0105 (8) | 0.0127 (7) | 0.0063 (7) |
| C46 | 0.0262 (8) | 0.0305 (9) | 0.0277 (7) | 0.0065 (8) | 0.0027 (6) | 0.0046 (6) |
| C47 | 0.0307 (9) | 0.0201 (8) | 0.0291 (7) | 0.0021 (7) | 0.0043 (6) | 0.0055 (6) |
| C48 | 0.0671 (14) | 0.0170 (8) | 0.0494 (10) | 0.0134 (9) | 0.0228 (9) | 0.0092 (7) |
| C49 | 0.0791 (17) | 0.0465 (13) | 0.0849 (17) | 0.0339 (13) | 0.0472 (14) | 0.0244 (12) |
| C50 | 0.124 (3) | 0.0217 (10) | 0.0651 (15) | −0.0023 (14) | 0.0042 (15) | −0.0025 (10) |
| C51 | 0.0944 (19) | 0.0274 (10) | 0.0640 (13) | 0.0241 (12) | 0.0337 (13) | 0.0215 (9) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| P1—O2 | 1.4808 (9) | C24—C29 | 1.395 (2) |
| P1—O1 | 1.6144 (11) | C25—C26 | 1.392 (2) |
| P1—C7 | 1.7835 (13) | C25—H25 | 0.9500 |
| P1—C1 | 1.7871 (15) | C26—C27 | 1.381 (3) |
| O1—N1 | 1.4430 (14) | C26—H26 | 0.9500 |
| O4—C13 | 1.2020 (17) | C27—C28 | 1.379 (3) |
| O3—C13 | 1.3400 (17) | C27—H27 | 0.9501 |
| O3—C14 | 1.4757 (17) | C28—C29 | 1.390 (2) |
| N1—C13 | 1.3883 (18) | C28—H28 | 0.9501 |
| N1—H1N | 0.886 (17) | C29—H29 | 0.9500 |
| C1—C2 | 1.392 (2) | C31—C32 | 1.511 (3) |
| C1—C6 | 1.396 (2) | C31—C33 | 1.514 (3) |
| C2—C3 | 1.394 (2) | C31—C34 | 1.518 (2) |
| C2—H2 | 0.9500 | C32—H321 | 0.9801 |
| C3—C4 | 1.378 (3) | C32—H322 | 0.9800 |
| C3—H3 | 0.9500 | C32—H323 | 0.9799 |
| C4—C5 | 1.374 (3) | C33—H331 | 0.9803 |
| C4—H4 | 0.9500 | C33—H332 | 0.9802 |

| | | | |
|---|---|---|---|
| C5—C6 | 1.396 (2) | C33—H333 | 0.9804 |
| C5—H5 | 0.9501 | C34—H341 | 0.9802 |
| C6—H6 | 0.9501 | C34—H342 | 0.9801 |
| C7—C12 | 1.3992 (18) | C34—H343 | 0.9801 |
| C7—C8 | 1.3995 (18) | P3—O10 | 1.4813 (10) |
| C8—C9 | 1.3939 (18) | P3—O9 | 1.6187 (11) |
| C8—H8 | 0.9502 | P3—C35 | 1.7853 (13) |
| C9—C10 | 1.389 (2) | P3—C41 | 1.7895 (15) |
| C9—H9 | 0.9500 | O9—N3 | 1.4367 (14) |
| C10—C11 | 1.391 (2) | O11—C47 | 1.3393 (18) |
| C10—H10 | 0.9500 | O11—C48 | 1.4752 (18) |
| C11—C12 | 1.3864 (18) | O12—C47 | 1.2007 (18) |
| C11—H11 | 0.9500 | N3—C47 | 1.3837 (18) |
| C12—H12 | 0.9501 | N3—H3N | 0.923 (18) |
| C14—C15 | 1.515 (3) | C35—C40 | 1.3961 (19) |
| C14—C17 | 1.518 (3) | C35—C36 | 1.4026 (19) |
| C14—C16 | 1.518 (2) | C36—C37 | 1.3942 (19) |
| C15—H151 | 0.9800 | C36—H36 | 0.9500 |
| C15—H152 | 0.9802 | C37—C38 | 1.381 (2) |
| C15—H153 | 0.9802 | C37—H37 | 0.9500 |
| C16—H161 | 0.9802 | C38—C39 | 1.389 (2) |
| C16—H162 | 0.9801 | C38—H38 | 0.9500 |
| C16—H163 | 0.9800 | C39—C40 | 1.395 (2) |
| C17—H171 | 0.9805 | C39—H39 | 0.9499 |
| C17—H172 | 0.9807 | C40—H40 | 0.9502 |
| C17—H173 | 0.9804 | C41—C46 | 1.3954 (19) |
| P2—O6 | 1.4783 (10) | C41—C42 | 1.399 (2) |
| P2—O5 | 1.6162 (11) | C42—C43 | 1.385 (2) |
| P2—C18 | 1.7836 (14) | C42—H42 | 0.9500 |
| P2—C24 | 1.7890 (15) | C43—C44 | 1.386 (2) |
| O5—N2 | 1.4374 (14) | C43—H43 | 0.9500 |
| O7—C30 | 1.3400 (17) | C44—C45 | 1.383 (2) |
| O7—C31 | 1.4748 (18) | C44—H44 | 0.9501 |
| O8—C30 | 1.2018 (17) | C45—C46 | 1.389 (2) |
| N2—C30 | 1.3867 (18) | C45—H45 | 0.9501 |
| N2—H2N | 0.95 (2) | C46—H46 | 0.9499 |
| C18—C23 | 1.3976 (19) | C48—C50 | 1.509 (3) |
| C18—C19 | 1.4019 (19) | C48—C51 | 1.516 (3) |
| C19—C20 | 1.3932 (19) | C48—C49 | 1.528 (3) |
| C19—H19 | 0.9501 | C49—H491 | 0.9801 |
| C20—C21 | 1.384 (2) | C49—H492 | 0.9800 |
| C20—H20 | 0.9500 | C49—H493 | 0.9801 |
| C21—C22 | 1.391 (2) | C50—H501 | 0.9803 |
| C21—H21 | 0.9500 | C50—H502 | 0.9802 |
| C22—C23 | 1.393 (2) | C50—H503 | 0.9801 |
| C22—H22 | 0.9500 | C51—H511 | 0.9804 |
| C23—H23 | 0.9500 | C51—H512 | 0.9803 |
| C24—C25 | 1.393 (2) | C51—H513 | 0.9803 |

| | | | |
|---|---|---|---|
| O2—P1—O1 | 115.19 (6) | C25—C26—H26 | 120.1 |
| O2—P1—C7 | 112.68 (6) | C28—C27—C26 | 120.93 (16) |
| O1—P1—C7 | 99.91 (6) | C28—C27—H27 | 119.5 |
| O2—P1—C1 | 112.18 (6) | C26—C27—H27 | 119.5 |
| O1—P1—C1 | 105.79 (6) | C27—C28—C29 | 119.93 (16) |
| C7—P1—C1 | 110.27 (7) | C27—C28—H28 | 120.1 |
| N1—O1—P1 | 107.68 (8) | C29—C28—H28 | 120.0 |
| C13—O3—C14 | 120.86 (11) | C28—C29—C24 | 119.56 (16) |
| C13—N1—O1 | 111.09 (10) | C28—C29—H29 | 120.2 |
| C13—N1—H1N | 114.4 (11) | C24—C29—H29 | 120.2 |
| O1—N1—H1N | 107.8 (11) | O8—C30—O7 | 127.79 (14) |
| C2—C1—C6 | 119.87 (14) | O8—C30—N2 | 125.97 (14) |
| C2—C1—P1 | 118.31 (11) | O7—C30—N2 | 106.14 (11) |
| C6—C1—P1 | 121.80 (12) | O7—C31—C32 | 109.70 (14) |
| C1—C2—C3 | 119.72 (15) | O7—C31—C33 | 110.27 (16) |
| C1—C2—H2 | 120.1 | C32—C31—C33 | 112.80 (18) |
| C3—C2—H2 | 120.2 | O7—C31—C34 | 101.63 (12) |
| C4—C3—C2 | 120.01 (17) | C32—C31—C34 | 110.71 (18) |
| C4—C3—H3 | 120.1 | C33—C31—C34 | 111.16 (16) |
| C2—C3—H3 | 119.9 | C31—C32—H321 | 109.5 |
| C5—C4—C3 | 120.74 (16) | C31—C32—H322 | 109.4 |
| C5—C4—H4 | 119.7 | H321—C32—H322 | 109.5 |
| C3—C4—H4 | 119.6 | C31—C32—H323 | 109.5 |
| C4—C5—C6 | 120.10 (16) | H321—C32—H323 | 109.5 |
| C4—C5—H5 | 120.0 | H322—C32—H323 | 109.5 |
| C6—C5—H5 | 119.9 | C31—C33—H331 | 109.4 |
| C1—C6—C5 | 119.55 (17) | C31—C33—H332 | 109.5 |
| C1—C6—H6 | 120.2 | H331—C33—H332 | 109.5 |
| C5—C6—H6 | 120.3 | C31—C33—H333 | 109.6 |
| C12—C7—C8 | 120.03 (12) | H331—C33—H333 | 109.4 |
| C12—C7—P1 | 122.08 (10) | H332—C33—H333 | 109.5 |
| C8—C7—P1 | 117.89 (10) | C31—C34—H341 | 109.5 |
| C9—C8—C7 | 119.44 (13) | C31—C34—H342 | 109.5 |
| C9—C8—H8 | 120.3 | H341—C34—H342 | 109.5 |
| C7—C8—H8 | 120.2 | C31—C34—H343 | 109.4 |
| C10—C9—C8 | 120.33 (13) | H341—C34—H343 | 109.5 |
| C10—C9—H9 | 119.9 | H342—C34—H343 | 109.5 |
| C8—C9—H9 | 119.8 | O10—P3—O9 | 115.30 (6) |
| C9—C10—C11 | 120.09 (13) | O10—P3—C35 | 112.54 (6) |
| C9—C10—H10 | 119.9 | O9—P3—C35 | 99.52 (6) |
| C11—C10—H10 | 120.0 | O10—P3—C41 | 111.02 (6) |
| C12—C11—C10 | 120.22 (14) | O9—P3—C41 | 106.13 (6) |
| C12—C11—H11 | 119.9 | C35—P3—C41 | 111.72 (7) |
| C10—C11—H11 | 119.9 | N3—O9—P3 | 108.44 (8) |
| C11—C12—C7 | 119.88 (13) | C47—O11—C48 | 120.81 (12) |
| C11—C12—H12 | 120.1 | C47—N3—O9 | 111.97 (11) |
| C7—C12—H12 | 120.0 | C47—N3—H3N | 117.6 (12) |

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| O4—C13—O3 | 128.10 (14) | O9—N3—H3N | 110.6 (11) |
| O4—C13—N1 | 126.01 (14) | C40—C35—C36 | 120.06 (13) |
| O3—C13—N1 | 105.80 (11) | C40—C35—P3 | 123.37 (11) |
| O3—C14—C15 | 109.68 (13) | C36—C35—P3 | 116.57 (11) |
| O3—C14—C17 | 109.88 (15) | C37—C36—C35 | 119.50 (14) |
| C15—C14—C17 | 112.75 (16) | C37—C36—H36 | 120.2 |
| O3—C14—C16 | 101.85 (12) | C35—C36—H36 | 120.3 |
| C15—C14—C16 | 111.39 (16) | C38—C37—C36 | 120.25 (15) |
| C17—C14—C16 | 110.74 (15) | C38—C37—H37 | 119.9 |
| C14—C15—H151 | 109.4 | C36—C37—H37 | 119.9 |
| C14—C15—H152 | 109.5 | C37—C38—C39 | 120.48 (14) |
| H151—C15—H152 | 109.5 | C37—C38—H38 | 119.8 |
| C14—C15—H153 | 109.5 | C39—C38—H38 | 119.8 |
| H151—C15—H153 | 109.5 | C38—C39—C40 | 120.07 (15) |
| H152—C15—H153 | 109.5 | C38—C39—H39 | 120.0 |
| C14—C16—H161 | 109.5 | C40—C39—H39 | 120.0 |
| C14—C16—H162 | 109.5 | C39—C40—C35 | 119.63 (15) |
| H161—C16—H162 | 109.5 | C39—C40—H40 | 120.2 |
| C14—C16—H163 | 109.5 | C35—C40—H40 | 120.2 |
| H161—C16—H163 | 109.5 | C46—C41—C42 | 119.44 (14) |
| H162—C16—H163 | 109.5 | C46—C41—P3 | 123.19 (11) |
| C14—C17—H171 | 109.5 | C42—C41—P3 | 117.37 (11) |
| C14—C17—H172 | 109.6 | C43—C42—C41 | 120.11 (14) |
| H171—C17—H172 | 109.4 | C43—C42—H42 | 120.0 |
| C14—C17—H173 | 109.5 | C41—C42—H42 | 119.9 |
| H171—C17—H173 | 109.4 | C42—C43—C44 | 119.96 (15) |
| H172—C17—H173 | 109.4 | C42—C43—H43 | 120.0 |
| O6—P2—O5 | 115.40 (6) | C44—C43—H43 | 120.0 |
| O6—P2—C18 | 112.66 (6) | C45—C44—C43 | 120.48 (15) |
| O5—P2—C18 | 99.07 (6) | C45—C44—H44 | 119.8 |
| O6—P2—C24 | 110.56 (6) | C43—C44—H44 | 119.8 |
| O5—P2—C24 | 106.56 (6) | C44—C45—C46 | 119.92 (15) |
| C18—P2—C24 | 112.02 (7) | C44—C45—H45 | 120.1 |
| N2—O5—P2 | 108.19 (8) | C46—C45—H45 | 120.0 |
| C30—O7—C31 | 121.02 (11) | C45—C46—C41 | 120.08 (15) |
| C30—N2—O5 | 111.24 (10) | C45—C46—H46 | 120.0 |
| C30—N2—H2N | 115.3 (12) | C41—C46—H46 | 119.9 |
| O5—N2—H2N | 109.6 (11) | O12—C47—O11 | 127.51 (14) |
| C23—C18—C19 | 120.06 (13) | O12—C47—N3 | 125.58 (15) |
| C23—C18—P2 | 123.04 (11) | O11—C47—N3 | 106.82 (12) |
| C19—C18—P2 | 116.90 (11) | O11—C48—C50 | 110.23 (16) |
| C20—C19—C18 | 119.55 (14) | O11—C48—C51 | 101.75 (14) |
| C20—C19—H19 | 120.3 | C50—C48—C51 | 110.8 (2) |
| C18—C19—H19 | 120.2 | O11—C48—C49 | 109.18 (16) |
| C21—C20—C19 | 120.30 (15) | C50—C48—C49 | 113.8 (2) |
| C21—C20—H20 | 119.9 | C51—C48—C49 | 110.39 (18) |
| C19—C20—H20 | 119.8 | C48—C49—H491 | 109.5 |
| C20—C21—C22 | 120.31 (14) | C48—C49—H492 | 109.4 |

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| C20—C21—H21 | 119.8 | H491—C49—H492 | 109.5 |
| C22—C21—H21 | 119.9 | C48—C49—H493 | 109.5 |
| C21—C22—C23 | 120.13 (15) | H491—C49—H493 | 109.5 |
| C21—C22—H22 | 119.9 | H492—C49—H493 | 109.5 |
| C23—C22—H22 | 119.9 | C48—C50—H501 | 109.4 |
| C22—C23—C18 | 119.64 (15) | C48—C50—H502 | 109.6 |
| C22—C23—H23 | 120.2 | H501—C50—H502 | 109.5 |
| C18—C23—H23 | 120.2 | C48—C50—H503 | 109.4 |
| C25—C24—C29 | 120.18 (14) | H501—C50—H503 | 109.5 |
| C25—C24—P2 | 117.08 (11) | H502—C50—H503 | 109.5 |
| C29—C24—P2 | 122.74 (12) | C48—C51—H511 | 109.6 |
| C26—C25—C24 | 119.62 (16) | C48—C51—H512 | 109.4 |
| C26—C25—H25 | 120.2 | H511—C51—H512 | 109.4 |
| C24—C25—H25 | 120.2 | C48—C51—H513 | 109.5 |
| C27—C26—C25 | 119.77 (16) | H511—C51—H513 | 109.4 |
| C27—C26—H26 | 120.2 | H512—C51—H513 | 109.5 |

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| O2—P1—O1—N1 | 58.18 (9) | C18—P2—C24—C25 | 106.65 (13) |
| C7—P1—O1—N1 | 179.16 (8) | O6—P2—C24—C29 | 160.14 (12) |
| C1—P1—O1—N1 | −66.33 (9) | O5—P2—C24—C29 | 34.01 (14) |
| P1—O1—N1—C13 | 142.58 (11) | C18—P2—C24—C29 | −73.30 (14) |
| O2—P1—C1—C2 | 26.47 (14) | C31—O7—C30—O8 | −1.8 (3) |
| O1—P1—C1—C2 | 152.85 (12) | C31—O7—C30—N2 | −178.31 (14) |
| C7—P1—C1—C2 | −100.00 (13) | O5—N2—C30—O8 | 18.3 (2) |
| O2—P1—C1—C6 | −155.13 (12) | O5—N2—C30—O7 | −165.15 (12) |
| O1—P1—C1—C6 | −28.76 (14) | C30—O7—C31—C32 | −63.8 (2) |
| C7—P1—C1—C6 | 78.40 (14) | C30—O7—C31—C33 | 61.0 (2) |
| O2—P1—C7—C12 | −171.40 (12) | C30—O7—C31—C34 | 179.00 (15) |
| O1—P1—C7—C12 | 65.83 (14) | O10—P3—O9—N3 | −57.91 (9) |
| C1—P1—C7—C12 | −45.21 (15) | C35—P3—O9—N3 | −178.52 (8) |
| O2—P1—C7—C8 | 8.16 (15) | C41—P3—O9—N3 | 65.44 (9) |
| O1—P1—C7—C8 | −114.61 (12) | P3—O9—N3—C47 | −133.94 (12) |
| C1—P1—C7—C8 | 134.35 (12) | O10—P3—C35—C40 | −174.95 (12) |
| C14—O3—C13—O4 | −2.6 (3) | O9—P3—C35—C40 | −52.35 (14) |
| C14—O3—C13—N1 | 174.07 (14) | C41—P3—C35—C40 | 59.37 (15) |
| O1—N1—C13—O4 | −16.6 (2) | O10—P3—C35—C36 | 4.97 (15) |
| O1—N1—C13—O3 | 166.69 (11) | O9—P3—C35—C36 | 127.57 (12) |
| C13—O3—C14—C15 | 64.6 (2) | C41—P3—C35—C36 | −120.72 (13) |
| C13—O3—C14—C17 | −59.9 (2) | O10—P3—C41—C46 | 164.23 (12) |
| C13—O3—C14—C16 | −177.33 (15) | O9—P3—C41—C46 | 38.23 (14) |
| O6—P2—O5—N2 | −57.27 (9) | C35—P3—C41—C46 | −69.26 (15) |
| C18—P2—O5—N2 | −177.79 (8) | O10—P3—C41—C42 | −16.56 (14) |
| C24—P2—O5—N2 | 65.88 (9) | O9—P3—C41—C42 | −142.56 (12) |
| P2—O5—N2—C30 | −138.18 (11) | C35—P3—C41—C42 | 109.96 (13) |
| O6—P2—C18—C23 | 179.38 (13) | C48—O11—C47—O12 | 6.2 (3) |
| O5—P2—C18—C23 | −58.11 (14) | C48—O11—C47—N3 | −170.61 (15) |
| C24—P2—C18—C23 | 53.97 (16) | O9—N3—C47—O12 | 21.4 (2) |
| O6—P2—C18—C19 | −0.87 (15) | O9—N3—C47—O11 | −161.75 (12) |

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| O5—P2—C18—C19 | 121.64 (13) | C47—O11—C48—C50 | −64.9 (2) |
| C24—P2—C18—C19 | −126.28 (13) | C47—O11—C48—C51 | 177.42 (17) |
| O6—P2—C24—C25 | −19.90 (14) | C47—O11—C48—C49 | 60.7 (2) |
| O5—P2—C24—C25 | −146.03 (12) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*N*···O6 | 0.886 (17) | 1.836 (17) | 2.7138 (16) | 170.0 (15) |
| N2—H2*N*···O2 | 0.95 (2) | 1.83 (2) | 2.7688 (16) | 168.4 (17) |
| N3—H3*N*···O10$^{i}$ | 0.923 (18) | 1.855 (19) | 2.7666 (16) | 169.0 (16) |

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