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# *N*,*N*′,*N*″,*N*‴-Tetrakis(2-methylphenyl)-oxybis(phosphonic diamide): a redetermination at 150 K with Mo *Kα* radiation

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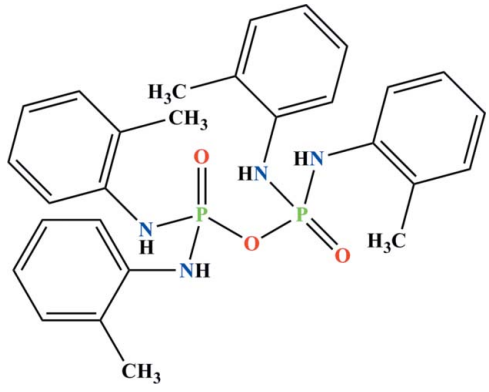
Key indicators: single-crystal X-ray study; $T$ = 150 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.054; $wR$ factor = 0.121; data-to-parameter ratio = 18.4.

The structure of the title compound, $C_{28}H_{32}N_4O_3P_2$, has been redetermined at 150 K, with much improved precision. The structure and molecular packing of the title compound was previously determined using Cu $K\alpha$ radiation, with an $R$ value of 0.0933 [Cameron *et al.* (1978). *Z. Naturforsch. Teil B*, **33**, 728–730]. The *c*-axis length in this structure [13.8401 (8) Å] is almost half that reported in the original study. In the title compound, two $(C_6H_4(2\text{-}CH_3)NH)_2P(O)$ units are bridged *via* an O atom [P—O—P = 133.31 (11)°]. The P atoms adopt a slightly distorted tetrahedral coordination geometry. In the crystal, molecules are linked *via* N—H···OP hydrogen bonds into extended chains parallel to the *c* axis. An intramolecular N—H···O=P hydrogen bond is also found in the molecule.

## Related literature

For the previous determination of this structure, see: Cameron *et al.* (1978). For bond lengths and angles in related structures, see: Pourayoubi *et al.* (2010); Sabbaghi *et al.* (2010).




## Experimental

### Crystal data

$C_{28}H_{32}N_4O_3P_2$
$M_r$ = 534.52
Monoclinic, $P2_1/c$
$a$ = 14.2621 (6) Å
$b$ = 15.7029 (11) Å
$c$ = 13.8401 (8) Å
$\beta$ = 118.915 (4)°
$V$ = 2713.2 (3) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.20 mm$^{-1}$
$T$ = 150 K
0.45 × 0.33 × 0.21 mm

### Data collection

Bruker–Nonius KappaCCD area-detector diffractometer
Absorption correction: Gaussian integration (Coppens & Hamilton, 1970)
$T_{min}$ = 0.942, $T_{max}$ = 0.969
19090 measured reflections
6133 independent reflections
4719 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.047

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.121
$S$ = 1.18
6133 reflections
334 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.38 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.51 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N111—H111···O3$^{i}$ | 0.86 | 2.16 | 2.926 (2) | 149 |
| N112—H112···O3 | 0.86 | 2.16 | 2.906 (2) | 144 |
| N113—H113···O2$^{ii}$ | 0.86 | 1.97 | 2.814 (2) | 167 |

Symmetry codes: (i) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (ii) $x, -y+\frac{1}{2}, z+\frac{1}{2}$.

Data collection: *COLLECT* (Hooft, 1998) and *DENZO* (Otwinowski & Minor, 1997); cell refinement: *COLLECT* and *DENZO*; data reduction: *COLLECT* and *DENZO*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ5092).

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# supporting information

*Acta Cryst.* (2011). E**67**, o450–o451 [doi:10.1107/S1600536811002091]

## *N*,*N*′,*N*″,*N*‴-Tetrakis(2-methylphenyl)oxybis(phosphonic diamide): a redetermination at 150 K with Mo *Kα* radiation

**Mehrdad Pourayoubi, Zdeňka Padělková, Mahnaz Rostami Chaijan and Aleš Růžička**

### S1. Comment

The structure and molecular packing of $\mu$-oxo-bis(phosphenyl-*o*-toluidide) was previously reported at ambient temperature by Cameron *et al.* (1978) using 2595 independent reflections significantly above the background with Cu *Kα* radiation; $R$ = 0.0933. The unit-cell length $c$ and density were respectively reported as 25.26 (3) Å and 1.255 g cm$^{-3}$.

Here, we report on the low temperature X-ray determination of the title compound (Fig. 1) at 150 K using Mo *Kα* radiation, with some improved precision. The unit-cell length $c$ (of 13.8401 (8) Å) and density (of 1.309 g cm$^{-3}$) in this structure are very different from the previously reported values.

In the title compound, two $(C_6H_4(2\text{-}CH_3)NH)_2P(O)$ moieties are bridged *via* an oxygen atom (P1—O1—P2 angle = 133.31 (11)°); the P1—O1 and P2—O1 bond lengths of 1.6014 (16) and 1.6017 (16) Å are standard for the P—O—P moiety (Pourayoubi *et al.*, 2010). The P atoms adopt a slightly distorted tetrahedral environment. The bond angles around the P atoms are in the range of 101.05 (9)° to 116.62 (11)° for P1 and 100.12 (10)° to 117.97 (11)° for P2.

The P1—O2 and P2—O3 bond lengths (1.4681 (17) and 1.4736 (17) Å) and the P—N bond lengths (1.630 (2), 1.6376 (19), 1.612 (2) and 1.634 (2) Å) are standard for this type of compound; for example in $[4\text{-}H_3C—C_6H_4O]P(O)[NHC_6H_4\text{-}2\text{-}CH_3]_2$ (Sabbaghi *et al.*, 2010), P═O = 1.4692 (12) Å and P—N = 1.6268 (15) and 1.6279 (15) Å).

An intramolecular N–H···OP hydrogen bond (N···O = 2.906 (2) Å) is found between the oxygen atom of the P2 phosphoryl group and the N–H hydrogen atom of one of the amide moieties linked to the P1 atom. In the crystal structure, molecules are linked *via* N–H···OP hydrogen bonds (Fig. 2) into extended chains parallel to the $c$ axis (N···O = 2.814 (2) & 2.926 (2) Å).

### S2. Experimental

To a solution of (11.42 mmol) phosphoryl chloride in chloroform (10 ml), a solution of *o*-toluidine (68.52 mmol) in chloroform (10 ml) was added dropwise at 268 K. After 4 h, the solvent was removed in vacuum. Single crystals were obtained from a mixture of chloroform/n-heptane after slow evaporation at room temperature. $^{31}P\{^1H\}$ NMR (202.45 MHz, DMSO-d6, 300.0 K, $H_3PO_4$ external): -5.01 p.p.m. (*s*). $^1H$ NMR (500.13 MHz, DMSO-d6, 300.0 K, TMS): 2.03 (*s*, 12H, $4CH_3$), 6.87–7.06 (*m*, 12H, Ar—H), 7.29 (*m*, 4H, Ar—H), 7.35 p.p.m. (*d*, $^2J$(P,H) = 7.9 Hz, 4H, NH). IR (KBr, cm$^{-1}$): 3746.3, 3413.2, 3300.4, 3188.5, 2927.3, 2854.8, 2358.8, 1675.8, 1599.4, 1496.1, 1402.5, 1226.5, 1111.8, 980.2, 844.9, 750.9.

### S3. Refinement

All the H atoms were discernible in the difference electron density map. However, all the H atoms were positioned geometrically and refined as riding on their parent C or N atoms, with N—H = 0.86 Å, C—H = 0.98 Å for methyl, C—H = 0.93 Å for aromatic hydrogen atoms, U(H) = 1.2$U_{eq}$(C/N) for the amine and U(H) = 1.5$U_{eq}$(C) for methyl H atoms,

respectively.

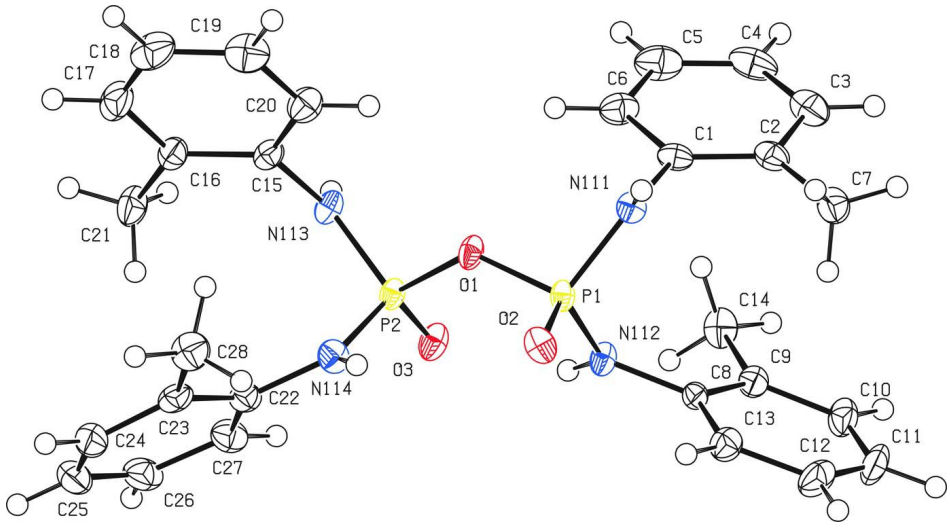


**Figure 1**

Molecular structure and atom labeling scheme for title compound with displacement ellipsoids at the 50% probability level.

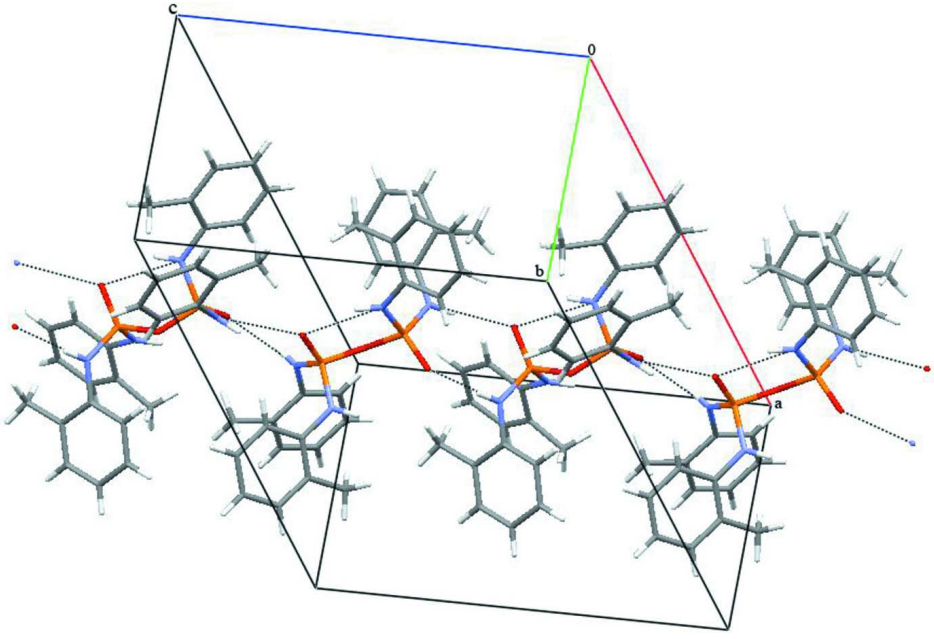


**Figure 2**

A view of H-bonded chain in the crystal network of title compound. N–H···OP hydrogen bonds are shown as dashed lines.

### *N*-{[bis(2-methylanilino)phosphoryloxy](2- methylanilino)phosphoryl]}-2-methylaniline

*Crystal data*

$C_{28}H_{32}N_4O_3P_2$
$M_r$ = 534.52

Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc

$a$ = 14.2621 (6) Å
$b$ = 15.7029 (11) Å
$c$ = 13.8401 (8) Å
$\beta$ = 118.915 (4)°
$V$ = 2713.2 (3) Å$^3$
$Z$ = 4
$F$(000) = 1128

$D_x$ = 1.309 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
$\theta$ = 1–27.5°
$\mu$ = 0.20 mm$^{-1}$
$T$ = 150 K
Needle, colourless
0.45 × 0.33 × 0.21 mm

*Data collection*

Bruker–Nonius KappaCCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 9.091 pixels mm$^{-1}$
$\varphi$ and $\omega$ scans to fill the Ewald sphere
Absorption correction: integration Gaussian integration (Coppens & Hamilton, 1970)

$T_{min}$ = 0.942, $T_{max}$ = 0.969
19090 measured reflections
6133 independent reflections
4719 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.047
$\theta_{max}$ = 27.5°, $\theta_{min}$ = 1.6°
$h$ = −17→18
$k$ = −20→18
$l$ = −17→17

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.121
$S$ = 1.18
6133 reflections
334 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
$w = 1/[\sigma^2(F_o^2) + (0.026P)^2 + 3.5968P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.38 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.51 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)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| P1 | 0.71234 (5) | 0.23508 (4) | 0.23550 (5) | 0.01571 (14) |
| P2 | 0.81104 (5) | 0.21568 (4) | 0.47636 (5) | 0.01629 (14) |
| O3 | 0.71315 (13) | 0.18618 (12) | 0.47678 (13) | 0.0220 (4) |
| O2 | 0.77228 (13) | 0.18044 (11) | 0.19840 (14) | 0.0222 (4) |
| N111 | 0.67768 (15) | 0.32469 (13) | 0.16770 (15) | 0.0171 (4) |
| H111 | 0.7047 | 0.3375 | 0.1258 | 0.021* |
| N112 | 0.60850 (15) | 0.19217 (13) | 0.23720 (15) | 0.0181 (4) |
| H112 | 0.6126 | 0.1794 | 0.2995 | 0.022* |
| O1 | 0.78382 (12) | 0.26154 (11) | 0.36239 (12) | 0.0195 (4) |

| | | | | |
|---|---|---|---|---|
| N113 | 0.87451 (15) | 0.28468 (13) | 0.57247 (15) | 0.0183 (4) |
| H113 | 0.8426 | 0.3037 | 0.6073 | 0.022* |
| N114 | 0.89635 (16) | 0.14390 (13) | 0.48176 (16) | 0.0207 (4) |
| H114 | 0.9035 | 0.1363 | 0.4242 | 0.025* |
| C15 | 0.97931 (18) | 0.31677 (16) | 0.6050 (2) | 0.0192 (5) |
| C8 | 0.51193 (18) | 0.17513 (15) | 0.13812 (18) | 0.0171 (5) |
| C14 | 0.4100 (2) | 0.25236 (18) | 0.2194 (2) | 0.0263 (6) |
| H14A | 0.3375 | 0.2691 | 0.1963 | 0.032* |
| H14B | 0.4543 | 0.3021 | 0.2366 | 0.032* |
| H14C | 0.4356 | 0.2169 | 0.2837 | 0.032* |
| C10 | 0.3220 (2) | 0.18702 (18) | 0.0296 (2) | 0.0268 (6) |
| H10 | 0.2563 | 0.2049 | 0.0211 | 0.032* |
| C27 | 0.9154 (2) | 0.06277 (17) | 0.6406 (2) | 0.0259 (6) |
| H27 | 0.8443 | 0.0744 | 0.6200 | 0.031* |
| C23 | 1.0662 (2) | 0.07556 (16) | 0.6064 (2) | 0.0231 (5) |
| C1 | 0.60452 (19) | 0.38319 (15) | 0.1753 (2) | 0.0193 (5) |
| C2 | 0.5062 (2) | 0.40163 (16) | 0.0836 (2) | 0.0226 (5) |
| C9 | 0.41430 (19) | 0.20379 (16) | 0.12797 (19) | 0.0206 (5) |
| C24 | 1.1247 (2) | 0.02480 (17) | 0.6984 (2) | 0.0294 (6) |
| H24 | 1.1947 | 0.0104 | 0.7174 | 0.035* |
| C21 | 1.0415 (2) | 0.25452 (19) | 0.7949 (2) | 0.0297 (6) |
| H21A | 1.1022 | 0.2605 | 0.8673 | 0.036* |
| H21B | 1.0307 | 0.1954 | 0.7750 | 0.036* |
| H21C | 0.9789 | 0.2768 | 0.7950 | 0.036* |
| C16 | 1.06130 (19) | 0.30251 (16) | 0.7133 (2) | 0.0219 (5) |
| C6 | 0.6332 (2) | 0.42124 (17) | 0.2774 (2) | 0.0267 (6) |
| H6 | 0.7003 | 0.4104 | 0.3374 | 0.032* |
| C22 | 0.9598 (2) | 0.09322 (16) | 0.5770 (2) | 0.0214 (5) |
| C11 | 0.3249 (2) | 0.1444 (2) | −0.0562 (2) | 0.0326 (7) |
| H11 | 0.2619 | 0.1348 | −0.1218 | 0.039* |
| C12 | 0.4213 (2) | 0.11623 (19) | −0.0450 (2) | 0.0305 (6) |
| H12 | 0.4234 | 0.0873 | −0.1025 | 0.037* |
| C3 | 0.4368 (2) | 0.45692 (18) | 0.0985 (2) | 0.0309 (6) |
| H3 | 0.3708 | 0.4705 | 0.0386 | 0.037* |
| C13 | 0.5154 (2) | 0.13111 (17) | 0.0530 (2) | 0.0241 (5) |
| H13 | 0.5806 | 0.1117 | 0.0615 | 0.029* |
| C25 | 1.0816 (2) | −0.00486 (18) | 0.7628 (2) | 0.0327 (6) |
| H25 | 1.1228 | −0.0381 | 0.8247 | 0.039* |
| C19 | 1.1003 (2) | 0.39397 (19) | 0.5620 (3) | 0.0331 (6) |
| H19 | 1.1132 | 0.4239 | 0.5116 | 0.040* |
| C7 | 0.4739 (2) | 0.36301 (18) | −0.0263 (2) | 0.0264 (6) |
| H7A | 0.5312 | 0.3690 | −0.0435 | 0.032* |
| H7B | 0.4112 | 0.3914 | −0.0816 | 0.032* |
| H7C | 0.4585 | 0.3037 | −0.0247 | 0.032* |
| C20 | 0.9989 (2) | 0.36202 (18) | 0.5309 (2) | 0.0261 (6) |
| H20 | 0.9437 | 0.3714 | 0.4595 | 0.031* |
| C26 | 0.9776 (2) | 0.01502 (17) | 0.7348 (2) | 0.0300 (6) |
| H26 | 0.9489 | −0.0035 | 0.7788 | 0.036* |

| | | | | |
|---|---|---|---|---|
| C28 | 1.1164 (2) | 0.11288 (19) | 0.5422 (2) | 0.0308 (6) |
| H28A | 1.1902 | 0.0960 | 0.5754 | 0.037* |
| H28B | 1.1120 | 0.1739 | 0.5429 | 0.037* |
| H28C | 1.0791 | 0.0928 | 0.4674 | 0.037* |
| C18 | 1.1820 (2) | 0.3807 (2) | 0.6680 (3) | 0.0354 (7) |
| H18 | 1.2501 | 0.4021 | 0.6897 | 0.043* |
| C5 | 0.5626 (3) | 0.47486 (18) | 0.2898 (3) | 0.0347 (7) |
| H5 | 0.5812 | 0.4991 | 0.3581 | 0.042* |
| C17 | 1.1622 (2) | 0.33534 (19) | 0.7422 (2) | 0.0314 (6) |
| H17 | 1.2181 | 0.3266 | 0.8133 | 0.038* |
| C4 | 0.4648 (2) | 0.49159 (18) | 0.2005 (3) | 0.0344 (7) |
| H4 | 0.4168 | 0.5270 | 0.2087 | 0.041* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| P1 | 0.0135 (3) | 0.0217 (3) | 0.0119 (3) | 0.0002 (2) | 0.0062 (2) | 0.0018 (2) |
| P2 | 0.0149 (3) | 0.0225 (3) | 0.0113 (3) | −0.0025 (2) | 0.0062 (2) | 0.0001 (2) |
| O3 | 0.0189 (8) | 0.0333 (10) | 0.0141 (8) | −0.0056 (8) | 0.0083 (7) | −0.0009 (7) |
| O2 | 0.0214 (8) | 0.0264 (9) | 0.0232 (9) | 0.0059 (7) | 0.0144 (7) | 0.0043 (7) |
| N111 | 0.0195 (9) | 0.0215 (10) | 0.0146 (9) | −0.0007 (8) | 0.0115 (8) | 0.0009 (8) |
| N112 | 0.0163 (9) | 0.0267 (11) | 0.0119 (9) | −0.0035 (8) | 0.0073 (8) | 0.0021 (8) |
| O1 | 0.0163 (8) | 0.0280 (9) | 0.0113 (7) | −0.0049 (7) | 0.0045 (6) | 0.0020 (7) |
| N113 | 0.0150 (9) | 0.0261 (11) | 0.0150 (9) | −0.0024 (8) | 0.0081 (8) | −0.0046 (8) |
| N114 | 0.0260 (11) | 0.0239 (11) | 0.0148 (9) | 0.0019 (9) | 0.0119 (8) | 0.0005 (8) |
| C15 | 0.0151 (11) | 0.0206 (12) | 0.0221 (12) | −0.0010 (10) | 0.0090 (9) | −0.0053 (10) |
| C8 | 0.0178 (11) | 0.0167 (11) | 0.0140 (10) | −0.0019 (9) | 0.0055 (9) | 0.0029 (9) |
| C14 | 0.0216 (12) | 0.0355 (15) | 0.0246 (13) | 0.0017 (11) | 0.0134 (10) | 0.0018 (11) |
| C10 | 0.0167 (12) | 0.0343 (15) | 0.0249 (13) | −0.0031 (11) | 0.0063 (10) | 0.0033 (11) |
| C27 | 0.0308 (14) | 0.0234 (13) | 0.0265 (13) | 0.0003 (11) | 0.0163 (11) | 0.0009 (11) |
| C23 | 0.0253 (13) | 0.0198 (13) | 0.0237 (12) | −0.0030 (10) | 0.0115 (11) | −0.0085 (10) |
| C1 | 0.0245 (12) | 0.0156 (11) | 0.0247 (12) | −0.0012 (10) | 0.0172 (10) | 0.0011 (10) |
| C2 | 0.0249 (12) | 0.0182 (12) | 0.0280 (13) | 0.0002 (10) | 0.0154 (11) | 0.0044 (10) |
| C9 | 0.0206 (12) | 0.0228 (13) | 0.0182 (11) | −0.0031 (10) | 0.0092 (10) | 0.0030 (10) |
| C24 | 0.0285 (14) | 0.0237 (14) | 0.0276 (14) | 0.0045 (11) | 0.0068 (11) | −0.0027 (11) |
| C21 | 0.0266 (13) | 0.0372 (16) | 0.0179 (12) | −0.0018 (12) | 0.0049 (10) | −0.0012 (11) |
| C16 | 0.0172 (11) | 0.0228 (13) | 0.0227 (12) | 0.0026 (10) | 0.0072 (10) | −0.0052 (10) |
| C6 | 0.0338 (14) | 0.0227 (13) | 0.0274 (13) | −0.0032 (12) | 0.0178 (12) | −0.0040 (11) |
| C22 | 0.0265 (13) | 0.0183 (12) | 0.0184 (11) | −0.0008 (10) | 0.0101 (10) | −0.0029 (10) |
| C11 | 0.0237 (13) | 0.0411 (17) | 0.0192 (12) | −0.0111 (13) | −0.0007 (10) | −0.0013 (12) |
| C12 | 0.0367 (15) | 0.0334 (15) | 0.0184 (12) | −0.0098 (13) | 0.0109 (11) | −0.0079 (11) |
| C3 | 0.0314 (14) | 0.0262 (15) | 0.0383 (15) | 0.0041 (12) | 0.0193 (13) | 0.0078 (12) |
| C13 | 0.0246 (13) | 0.0260 (14) | 0.0210 (12) | −0.0015 (11) | 0.0104 (10) | −0.0014 (10) |
| C25 | 0.0423 (16) | 0.0217 (14) | 0.0250 (13) | 0.0037 (12) | 0.0090 (12) | 0.0032 (11) |
| C19 | 0.0330 (15) | 0.0341 (16) | 0.0426 (16) | −0.0031 (13) | 0.0264 (14) | 0.0005 (13) |
| C7 | 0.0255 (13) | 0.0281 (14) | 0.0241 (13) | 0.0016 (11) | 0.0107 (11) | 0.0038 (11) |
| C20 | 0.0244 (13) | 0.0295 (14) | 0.0257 (13) | −0.0042 (11) | 0.0130 (11) | −0.0025 (11) |
| C26 | 0.0429 (16) | 0.0222 (13) | 0.0267 (14) | −0.0017 (12) | 0.0182 (13) | 0.0024 (11) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C28 | 0.0271 (14) | 0.0330 (15) | 0.0343 (15) | −0.0002 (12) | 0.0164 (12) | −0.0020 (12) |
| C18 | 0.0222 (13) | 0.0353 (16) | 0.0519 (18) | −0.0066 (12) | 0.0203 (13) | −0.0101 (14) |
| C5 | 0.0517 (18) | 0.0266 (15) | 0.0364 (16) | −0.0027 (14) | 0.0297 (15) | −0.0062 (12) |
| C17 | 0.0177 (12) | 0.0350 (16) | 0.0341 (15) | 0.0005 (12) | 0.0066 (11) | −0.0063 (12) |
| C4 | 0.0455 (17) | 0.0195 (14) | 0.0502 (18) | 0.0071 (13) | 0.0327 (15) | 0.0024 (13) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| P1—O2 | 1.4681 (17) | C2—C7 | 1.489 (4) |
| P1—O1 | 1.6014 (16) | C24—C25 | 1.385 (4) |
| P1—N111 | 1.630 (2) | C24—H24 | 0.9300 |
| P1—N112 | 1.6376 (19) | C21—C16 | 1.494 (4) |
| P2—O3 | 1.4736 (17) | C21—H21A | 0.9601 |
| P2—O1 | 1.6017 (16) | C21—H21B | 0.9599 |
| P2—N113 | 1.612 (2) | C21—H21C | 0.9601 |
| P2—N114 | 1.634 (2) | C16—C17 | 1.392 (4) |
| N111—C1 | 1.432 (3) | C6—C5 | 1.385 (4) |
| N111—H111 | 0.8600 | C6—H6 | 0.9300 |
| N112—C8 | 1.422 (3) | C11—C12 | 1.379 (4) |
| N112—H112 | 0.8599 | C11—H11 | 0.9299 |
| N113—C15 | 1.428 (3) | C12—C13 | 1.391 (4) |
| N113—H113 | 0.8600 | C12—H12 | 0.9300 |
| N114—C22 | 1.427 (3) | C3—C4 | 1.380 (4) |
| N114—H114 | 0.8600 | C3—H3 | 0.9299 |
| C15—C20 | 1.383 (4) | C13—H13 | 0.9300 |
| C15—C16 | 1.403 (3) | C25—C26 | 1.375 (4) |
| C8—C13 | 1.387 (3) | C25—H25 | 0.9300 |
| C8—C9 | 1.404 (3) | C19—C18 | 1.379 (4) |
| C14—C9 | 1.504 (3) | C19—C20 | 1.388 (4) |
| C14—H14A | 0.9600 | C19—H19 | 0.9300 |
| C14—H14B | 0.9600 | C7—H7A | 0.9601 |
| C14—H14C | 0.9599 | C7—H7B | 0.9600 |
| C10—C11 | 1.382 (4) | C7—H7C | 0.9601 |
| C10—C9 | 1.387 (3) | C20—H20 | 0.9301 |
| C10—H10 | 0.9300 | C26—H26 | 0.9300 |
| C27—C26 | 1.389 (4) | C28—H28A | 0.9601 |
| C27—C22 | 1.393 (4) | C28—H28B | 0.9600 |
| C27—H27 | 0.9300 | C28—H28C | 0.9600 |
| C23—C24 | 1.388 (4) | C18—C17 | 1.386 (4) |
| C23—C22 | 1.396 (3) | C18—H18 | 0.9300 |
| C23—C28 | 1.504 (4) | C5—C4 | 1.369 (4) |
| C1—C2 | 1.393 (3) | C5—H5 | 0.9300 |
| C1—C6 | 1.401 (3) | C17—H17 | 0.9300 |
| C2—C3 | 1.404 (4) | C4—H4 | 0.9300 |
| | | | |
| O2—P1—O1 | 111.45 (10) | C16—C21—H21C | 109.4 |
| O2—P1—N111 | 111.65 (10) | H21A—C21—H21C | 109.5 |
| O1—P1—N111 | 105.22 (10) | H21B—C21—H21C | 109.5 |

| | | | |
|---|---|---|---|
| O2—P1—N112 | 116.62 (11) | C17—C16—C15 | 117.3 (2) |
| O1—P1—N112 | 101.05 (9) | C17—C16—C21 | 121.1 (2) |
| N111—P1—N112 | 109.81 (10) | C15—C16—C21 | 121.6 (2) |
| O3—P2—O1 | 111.48 (9) | C5—C6—C1 | 120.6 (3) |
| O3—P2—N113 | 111.30 (10) | C5—C6—H6 | 119.7 |
| O1—P2—N113 | 106.40 (10) | C1—C6—H6 | 119.7 |
| O3—P2—N114 | 117.97 (11) | C27—C22—C23 | 120.6 (2) |
| O1—P2—N114 | 100.12 (10) | C27—C22—N114 | 119.8 (2) |
| N113—P2—N114 | 108.51 (11) | C23—C22—N114 | 119.6 (2) |
| C1—N111—P1 | 122.72 (15) | C12—C11—C10 | 120.1 (2) |
| C1—N111—H111 | 118.7 | C12—C11—H11 | 119.9 |
| P1—N111—H111 | 118.6 | C10—C11—H11 | 120.0 |
| C8—N112—P1 | 121.64 (15) | C11—C12—C13 | 119.7 (3) |
| C8—N112—H112 | 119.2 | C11—C12—H12 | 120.2 |
| P1—N112—H112 | 119.2 | C13—C12—H12 | 120.2 |
| P1—O1—P2 | 133.31 (11) | C4—C3—C2 | 121.2 (3) |
| C15—N113—P2 | 125.59 (16) | C4—C3—H3 | 119.3 |
| C15—N113—H113 | 117.2 | C2—C3—H3 | 119.5 |
| P2—N113—H113 | 117.2 | C8—C13—C12 | 119.9 (2) |
| C22—N114—P2 | 123.54 (17) | C8—C13—H13 | 120.0 |
| C22—N114—H114 | 118.2 | C12—C13—H13 | 120.1 |
| P2—N114—H114 | 118.3 | C26—C25—C24 | 119.7 (3) |
| C20—C15—C16 | 120.8 (2) | C26—C25—H25 | 120.2 |
| C20—C15—N113 | 120.2 (2) | C24—C25—H25 | 120.1 |
| C16—C15—N113 | 119.0 (2) | C18—C19—C20 | 119.5 (3) |
| C13—C8—C9 | 121.0 (2) | C18—C19—H19 | 120.2 |
| C13—C8—N112 | 119.8 (2) | C20—C19—H19 | 120.3 |
| C9—C8—N112 | 119.2 (2) | C2—C7—H7A | 109.5 |
| C9—C14—H14A | 109.5 | C2—C7—H7B | 109.5 |
| C9—C14—H14B | 109.4 | H7A—C7—H7B | 109.5 |
| H14A—C14—H14B | 109.5 | C2—C7—H7C | 109.4 |
| C9—C14—H14C | 109.5 | H7A—C7—H7C | 109.5 |
| H14A—C14—H14C | 109.5 | H7B—C7—H7C | 109.5 |
| H14B—C14—H14C | 109.5 | C15—C20—C19 | 120.6 (3) |
| C11—C10—C9 | 121.8 (2) | C15—C20—H20 | 119.8 |
| C11—C10—H10 | 119.1 | C19—C20—H20 | 119.5 |
| C9—C10—H10 | 119.1 | C25—C26—C27 | 120.0 (3) |
| C26—C27—C22 | 119.9 (3) | C25—C26—H26 | 120.0 |
| C26—C27—H27 | 120.1 | C27—C26—H26 | 120.0 |
| C22—C27—H27 | 120.0 | C23—C28—H28A | 109.6 |
| C24—C23—C22 | 117.9 (2) | C23—C28—H28B | 109.3 |
| C24—C23—C28 | 121.3 (2) | H28A—C28—H28B | 109.5 |
| C22—C23—C28 | 120.8 (2) | C23—C28—H28C | 109.5 |
| C2—C1—C6 | 120.3 (2) | H28A—C28—H28C | 109.5 |
| C2—C1—N111 | 121.0 (2) | H28B—C28—H28C | 109.5 |
| C6—C1—N111 | 118.7 (2) | C19—C18—C17 | 119.8 (3) |
| C1—C2—C3 | 117.8 (2) | C19—C18—H18 | 120.0 |
| C1—C2—C7 | 121.6 (2) | C17—C18—H18 | 120.1 |

| | | | |
|---|---|---|---|
| C3—C2—C7 | 120.6 (2) | C4—C5—C6 | 119.2 (3) |
| C10—C9—C8 | 117.5 (2) | C4—C5—H5 | 120.3 |
| C10—C9—C14 | 121.1 (2) | C6—C5—H5 | 120.5 |
| C8—C9—C14 | 121.3 (2) | C18—C17—C16 | 122.0 (3) |
| C25—C24—C23 | 121.8 (3) | C18—C17—H17 | 118.8 |
| C25—C24—H24 | 119.2 | C16—C17—H17 | 119.2 |
| C23—C24—H24 | 119.0 | C5—C4—C3 | 120.8 (3) |
| C16—C21—H21A | 109.5 | C5—C4—H4 | 119.5 |
| C16—C21—H21B | 109.4 | C3—C4—H4 | 119.6 |
| H21A—C21—H21B | 109.5 | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N111—H111···O3$^{i}$ | 0.86 | 2.16 | 2.926 (2) | 149 |
| N112—H112···O3 | 0.86 | 2.16 | 2.906 (2) | 144 |
| N113—H113···O2$^{ii}$ | 0.86 | 1.97 | 2.814 (2) | 167 |

Symmetry codes: (i) $x$, $-y+1/2$, $z-1/2$; (ii) $x$, $-y+1/2$, $z+1/2$.