Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# Diethylammonium anilino(methoxy)-phosphinate

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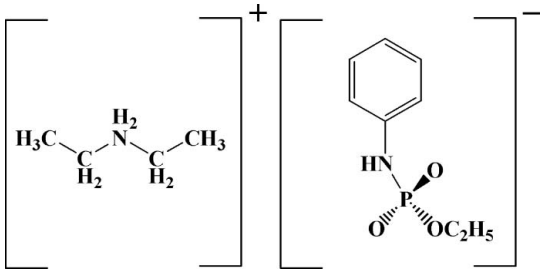
Received 9 August 2008; accepted 12 October 2008

Key indicators: single-crystal X-ray study; $T$ = 173 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.039; $wR$ factor = 0.081; data-to-parameter ratio = 14.9.

The title compound, $[Et_2NH_2][(EtO)PO_2(C_6H_5NH)]$ or $C_4H_{12}N^+\cdot C_8H_{11}NO_3P^-$, is a molecular salt with two anions containing $PO_3N$ groupings and two cations in the asymmetric unit. A network of N—H···O hydrogen bonds link the cations and anions into a two-dimensional network.

## Related literature

For the use of *N*-substituted phosphoramidic acids in the synthesis of pyrophosphate groups, see: Quin & Jankowski (1994). For a corresponding dimer complex with similar P—O and P—N connections, see: Andrianov et al. (1977).



## Experimental

*Crystal data*

$C_4H_{12}N^+\cdot C_8H_{11}NO_3P^-$
$M_r$ = 274.29
Orthorhombic, $Pna2_1$
$a$ = 14.341 (3) Å
$b$ = 12.785 (2) Å
$c$ = 15.997 (3) Å
$V$ = 2933.0 (9) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.19 mm$^{-1}$
$T$ = 173 (2) K
0.40 × 0.20 × 0.08 mm

*Data collection*

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.957, $T_{max}$ = 0.989
5062 measured reflections
4847 independent reflections
4043 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.017

*Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.081
$S$ = 1.03
4847 reflections
325 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.16 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.23 e Å$^{-3}$
Absolute structure: Flack (1983), 2649 Friedel pairs
Flack parameter: 0.06 (4)

**Table 1**
Selected bond lengths (Å).

| | | | |
|---|---|---|---|
| N1—P1 | 1.670 (2) | N2—P2 | 1.651 (2) |
| P1—O1 | 1.4798 (19) | P2—O5 | 1.4897 (18) |
| P1—O3 | 1.4994 (17) | P2—O4 | 1.4983 (19) |
| P1—O2 | 1.5979 (18) | P2—O6 | 1.5969 (17) |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1$B$···O4 | 0.86 | 2.13 | 2.954 (3) | 161 |
| N2—H2$B$···O1 | 0.86 | 2.04 | 2.895 (3) | 173 |
| N3—H3$C$···O4 | 0.90 | 1.88 | 2.742 (3) | 160 |
| N4—H4$A$···O3 | 0.90 | 1.94 | 2.792 (3) | 158 |
| N3—H3$B$···O3$^{i}$ | 0.90 | 1.89 | 2.788 (3) | 174 |
| N4—H4$B$···O5$^{ii}$ | 0.90 | 1.74 | 2.637 (3) | 172 |

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

Data collection: *SMART* (Bruker, 1996); cell refinement: *SAINT* (Bruker, 1996); 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*.

The authors thank the NSFFPC (No. 2003 F006) and the SRP program of the SCUT for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZ2138).

## References

Andrianov, V. G., Kalinin, A. E. & Struchkov, Yu. T. (1977). *Zh. Strukt. Khim.* **18**, 310–317.
Bruker (1996). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Quin, L. D. & Jankowski, S. (1994). *J. Org. Chem.* **59**, 4402–4409.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

# supporting information

*Acta Cryst.* (2008). E**64**, o2171 [doi:10.1107/S1600536808033023]

## Diethylammonium anilino(methoxy)phosphinate

**Zhiyong Fu and Xiaoling Liu**

### S1. Comment

N-substituted phosphoramidic acids are useful agents in the synthesis of pyrophosphate groups (Quin & Jankowski,1994). However the structures of these kinds of materials have not been well characterized. By the use of the O,*N*-substituted phosphoramidic acids as ligands in the preparation of new metal phosphate frameworks, we obtained the title compound as a salt of the O,*N*-substituted phosphoramidic acid.

As shown in Fig. 1, the asymmetric unit of the title compound is composed of two diethyl-amine cations, and two *N*-ethoxyphosphoryl-phenylamide anions. The geometrical parameters of the independent anions are similar. The phosphorus atoms have tetrahedral coordination geometries. The shortest P—O bond lengths correspond to the P=O double bonds. The longest P—O distances are due to the influence of the –OEt group (Andrianov *et al.*,1977). The O—P—O and O—P—N bond angles range from 102.961 (98)–119.158 (108)° and 105.126 (113)–112.197 (117)°, indicating that the geometries of the tetrahedra are slightly distorted.

Hydrogen bonds exist between the diethyl-amine cations and the *N*-ethoxyphosphoryl-phenylamide anions. The N—H···O connections result a two dimensional packing motif (Fig. 2).

### S2. Experimental

A solution of aniline (12.5 mmol) and 12.5 mmol of $Et_2NH$ in 15 ml of ether was added to a solution of 12.5 mmol of ethyl phosphorodichloridate in 15 ml of ether. After 20 h, the solution was filtered and the filtrate was evaporated to give a powder. The powder was dissolved in 30 ml of an acetone-water mixture (1:1) containing 1 g of NaOH. After 10 min, the solvent was evaporated and the residue dried *in vacuo*. Recrystalization of the precipitate from a chloroform solution yielded crystals of the title compound.

### S3. Refinement

H atoms were positioned geometrically and refined using a riding model, with N—H = 0.86–0.90 Å, C—H = 0.93–0.97 Å and with $U_{iso}$(H) = 1.2 (1.5 for methyl groups) times $U_{eq}$(C). >.

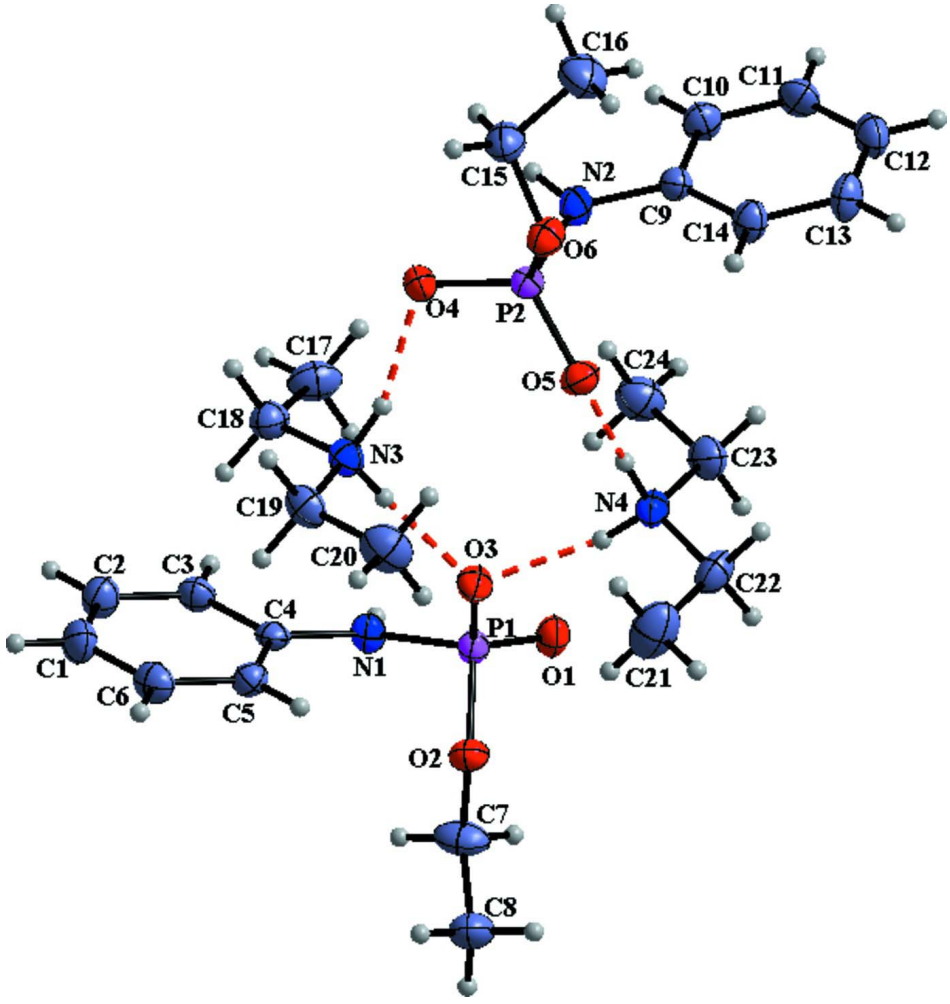



**Figure 1**

The molecular structure of (I), with atom labels and 50% probability displacement ellipsoids for non-H atoms. The red dashed lines indicate the hydrogen bonds.

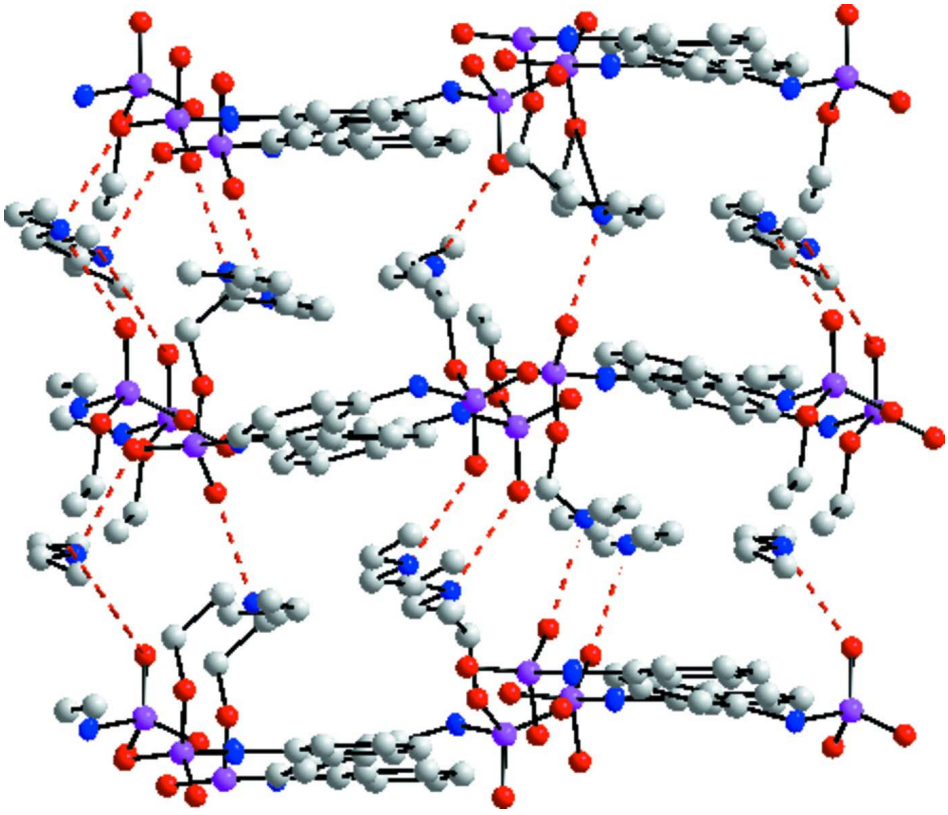


**Figure 2**

The packing of (I), viewed down the *b* axis, showing the N—H···O hydrogen bonds between the diethylamine cations and the *N*-ethoxy phosphoryl-phenylamide anions. H-atoms have been omitted for clarity.

### Diethylammonium anilino(methoxy)phosphinate

*Crystal data*

$C_4H_{12}N^+·C_8H_{11}NO_3P^-$
$M_r = 274.29$
Orthorhombic, $Pna2_1$
Hall symbol: P2c-2n
$a = 14.341$ (3) Å
$b = 12.785$ (2) Å
$c = 15.997$ (3) Å
$V = 2933.0$ (9) Å$^3$
$Z = 8$
$F(000) = 1184$
$D_x = 1.242$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4847 reflections
$\theta = 3.3$–25.0°
$\mu = 0.19$ mm$^{-1}$
$T = 173$ K
Needle-like, colorless
0.4 × 0.2 × 0.08 mm

*Data collection*

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.957$, $T_{max} = 0.989$
5062 measured reflections
4847 independent reflections
4043 reflections with $I > 2\sigma(I)$
$R_{int} = 0.002$
$\theta_{max} = 25.0°$, $\theta_{min} = 3.3°$
$h = -17 \rightarrow 17$
$k = -15 \rightarrow 15$
$l = -19 \rightarrow 18$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.081$
$S = 1.03$
4847 reflections
325 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0363P)^2 + 0.5497P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.16$ e Å$^{-3}$
$\Delta\rho_{min} = -0.23$ e Å$^{-3}$
Absolute structure: Flack (1983), 2649 Friedel pairs
Absolute structure parameter: 0.06 (4)

*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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| N1 | 0.50725 (13) | 0.16964 (16) | 0.52519 (13) | 0.0230 (5) |
| H1B | 0.5374 | 0.2242 | 0.5414 | 0.028* |
| N4 | 0.30305 (14) | −0.01440 (16) | 0.74774 (12) | 0.0265 (5) |
| H4A | 0.3436 | −0.0013 | 0.7061 | 0.032* |
| H4B | 0.2451 | −0.0069 | 0.7270 | 0.032* |
| C1 | 0.4316 (2) | 0.1694 (2) | 0.27143 (19) | 0.0359 (7) |
| H1A | 0.4151 | 0.1696 | 0.2152 | 0.043* |
| C2 | 0.45479 (18) | 0.2624 (2) | 0.31172 (18) | 0.0336 (7) |
| H2A | 0.4541 | 0.3251 | 0.2823 | 0.040* |
| C3 | 0.47881 (17) | 0.2616 (2) | 0.39546 (16) | 0.0269 (6) |
| H3A | 0.4937 | 0.3242 | 0.4219 | 0.032* |
| C4 | 0.48107 (16) | 0.1682 (2) | 0.44095 (16) | 0.0220 (6) |
| C5 | 0.45765 (17) | 0.0754 (2) | 0.39961 (16) | 0.0242 (6) |
| H5A | 0.4583 | 0.0122 | 0.4285 | 0.029* |
| C6 | 0.43349 (18) | 0.0772 (2) | 0.31564 (18) | 0.0315 (7) |
| H6A | 0.4183 | 0.0150 | 0.2888 | 0.038* |
| C7 | 0.63999 (18) | −0.0209 (2) | 0.5543 (2) | 0.0407 (7) |
| H7A | 0.6703 | 0.0043 | 0.6046 | 0.049* |
| H7B | 0.6556 | 0.0261 | 0.5087 | 0.049* |
| C8 | 0.6729 (2) | −0.1300 (2) | 0.53435 (19) | 0.0415 (7) |
| H8A | 0.7393 | −0.1297 | 0.5266 | 0.062* |
| H8B | 0.6432 | −0.1540 | 0.4841 | 0.062* |
| H8C | 0.6572 | −0.1760 | 0.5797 | 0.062* |

| | | | | |
|---|---|---|---|---|
| C21 | 0.3111 (3) | −0.1992 (2) | 0.7038 (2) | 0.0540 (9) |
| H21A | 0.3195 | −0.2693 | 0.7240 | 0.081* |
| H21B | 0.3596 | −0.1828 | 0.6645 | 0.081* |
| H21C | 0.2516 | −0.1935 | 0.6769 | 0.081* |
| C22 | 0.3154 (2) | −0.1243 (2) | 0.77572 (17) | 0.0351 (7) |
| H22A | 0.2671 | −0.1417 | 0.8157 | 0.042* |
| H22B | 0.3752 | −0.1312 | 0.8036 | 0.042* |
| C23 | 0.3172 (2) | 0.0639 (2) | 0.81480 (18) | 0.0359 (7) |
| H23A | 0.3785 | 0.0545 | 0.8391 | 0.043* |
| H23B | 0.2713 | 0.0529 | 0.8585 | 0.043* |
| C24 | 0.3086 (2) | 0.1737 (2) | 0.7821 (2) | 0.0434 (8) |
| H24A | 0.3185 | 0.2224 | 0.8269 | 0.065* |
| H24B | 0.2474 | 0.1838 | 0.7591 | 0.065* |
| H24C | 0.3544 | 0.1851 | 0.7393 | 0.065* |
| P1 | 0.48758 (4) | 0.07915 (5) | 0.59834 (4) | 0.02230 (16) |
| O1 | 0.52914 (12) | 0.12003 (13) | 0.67646 (11) | 0.0297 (4) |
| O2 | 0.54000 (11) | −0.02352 (13) | 0.56616 (11) | 0.0259 (4) |
| O3 | 0.38708 (11) | 0.04682 (12) | 0.59813 (12) | 0.0272 (4) |
| N2 | 0.55256 (14) | 0.33041 (17) | 0.73990 (14) | 0.0283 (5) |
| H2B | 0.5502 | 0.2687 | 0.7187 | 0.034* |
| N3 | 0.72854 (14) | 0.40960 (16) | 0.50343 (13) | 0.0267 (5) |
| H3B | 0.7820 | 0.4225 | 0.5310 | 0.032* |
| H3C | 0.6826 | 0.4046 | 0.5416 | 0.032* |
| C9 | 0.55185 (17) | 0.3353 (2) | 0.82782 (16) | 0.0233 (6) |
| C10 | 0.55401 (18) | 0.2428 (2) | 0.87430 (17) | 0.0302 (7) |
| H10A | 0.5560 | 0.1789 | 0.8466 | 0.036* |
| C11 | 0.55331 (19) | 0.2445 (2) | 0.96026 (18) | 0.0343 (7) |
| H11A | 0.5552 | 0.1820 | 0.9899 | 0.041* |
| C12 | 0.54987 (18) | 0.3381 (2) | 1.00280 (18) | 0.0346 (7) |
| H12A | 0.5492 | 0.3391 | 1.0609 | 0.042* |
| C13 | 0.5474 (2) | 0.4306 (2) | 0.95808 (18) | 0.0336 (7) |
| H13A | 0.5451 | 0.4941 | 0.9863 | 0.040* |
| C14 | 0.54840 (19) | 0.4294 (2) | 0.87124 (17) | 0.0298 (7) |
| H14A | 0.5467 | 0.4922 | 0.8419 | 0.036* |
| C15 | 0.37515 (18) | 0.4351 (2) | 0.66248 (18) | 0.0357 (7) |
| H15A | 0.3879 | 0.3613 | 0.6542 | 0.043* |
| H15B | 0.3479 | 0.4624 | 0.6115 | 0.043* |
| C16 | 0.3085 (2) | 0.4486 (3) | 0.7328 (2) | 0.0479 (8) |
| H16A | 0.2516 | 0.4121 | 0.7205 | 0.072* |
| H16B | 0.2955 | 0.5216 | 0.7404 | 0.072* |
| H16C | 0.3353 | 0.4207 | 0.7831 | 0.072* |
| C17 | 0.7673 (2) | 0.2234 (2) | 0.5187 (2) | 0.0412 (7) |
| H17A | 0.7725 | 0.1585 | 0.4889 | 0.062* |
| H17B | 0.7220 | 0.2164 | 0.5625 | 0.062* |
| H17C | 0.8267 | 0.2413 | 0.5424 | 0.062* |
| C18 | 0.73711 (19) | 0.3085 (2) | 0.45931 (17) | 0.0315 (6) |
| H18A | 0.7824 | 0.3149 | 0.4146 | 0.038* |
| H18B | 0.6775 | 0.2900 | 0.4347 | 0.038* |

| | | | | |
|---|---|---|---|---|
| C19 | 0.70839 (19) | 0.4987 (2) | 0.44723 (17) | 0.0326 (6) |
| H19A | 0.6487 | 0.4876 | 0.4201 | 0.039* |
| H19B | 0.7559 | 0.5023 | 0.4041 | 0.039* |
| C20 | 0.7060 (2) | 0.6010 (2) | 0.4943 (2) | 0.0475 (8) |
| H20A | 0.6928 | 0.6570 | 0.4562 | 0.071* |
| H20B | 0.7653 | 0.6128 | 0.5204 | 0.071* |
| H20C | 0.6583 | 0.5981 | 0.5364 | 0.071* |
| P2 | 0.55732 (4) | 0.42782 (5) | 0.67233 (4) | 0.02279 (16) |
| O4 | 0.56495 (11) | 0.37700 (13) | 0.58821 (11) | 0.0282 (4) |
| O5 | 0.62869 (12) | 0.50728 (13) | 0.69568 (10) | 0.0306 (4) |
| O6 | 0.46099 (11) | 0.48965 (13) | 0.68041 (12) | 0.0294 (4) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0269 (11) | 0.0204 (12) | 0.0219 (11) | −0.0046 (9) | −0.0028 (9) | −0.0019 (9) |
| N4 | 0.0218 (12) | 0.0335 (13) | 0.0241 (11) | 0.0008 (9) | −0.0012 (9) | 0.0045 (10) |
| C1 | 0.0436 (17) | 0.0395 (18) | 0.0247 (15) | 0.0050 (14) | −0.0059 (13) | −0.0015 (14) |
| C2 | 0.0404 (16) | 0.0300 (16) | 0.0304 (16) | 0.0006 (13) | −0.0034 (12) | 0.0025 (14) |
| C3 | 0.0305 (14) | 0.0226 (15) | 0.0276 (16) | 0.0018 (12) | 0.0013 (11) | −0.0045 (12) |
| C4 | 0.0181 (13) | 0.0249 (15) | 0.0231 (15) | 0.0032 (11) | 0.0024 (10) | −0.0017 (11) |
| C5 | 0.0255 (14) | 0.0204 (14) | 0.0268 (16) | −0.0006 (11) | −0.0014 (11) | −0.0014 (12) |
| C6 | 0.0319 (16) | 0.0342 (16) | 0.0284 (16) | −0.0013 (12) | −0.0040 (12) | −0.0077 (14) |
| C7 | 0.0223 (15) | 0.0437 (18) | 0.0561 (19) | 0.0006 (13) | 0.0027 (13) | −0.0137 (15) |
| C8 | 0.0409 (17) | 0.0400 (18) | 0.0435 (18) | 0.0134 (14) | 0.0066 (14) | −0.0004 (14) |
| C21 | 0.073 (2) | 0.0354 (18) | 0.054 (2) | −0.0014 (17) | −0.0147 (17) | 0.0019 (16) |
| C22 | 0.0366 (16) | 0.0322 (16) | 0.0365 (17) | 0.0011 (13) | −0.0049 (13) | 0.0116 (14) |
| C23 | 0.0386 (17) | 0.0401 (17) | 0.0291 (15) | 0.0011 (13) | −0.0048 (13) | −0.0042 (14) |
| C24 | 0.0464 (18) | 0.0368 (17) | 0.0470 (19) | −0.0006 (14) | 0.0049 (15) | −0.0052 (14) |
| P1 | 0.0261 (4) | 0.0211 (3) | 0.0197 (3) | 0.0005 (3) | −0.0011 (3) | 0.0006 (3) |
| O1 | 0.0411 (11) | 0.0250 (9) | 0.0230 (10) | 0.0006 (8) | −0.0046 (9) | 0.0006 (9) |
| O2 | 0.0239 (10) | 0.0239 (10) | 0.0298 (11) | 0.0019 (7) | 0.0000 (7) | −0.0027 (8) |
| O3 | 0.0225 (9) | 0.0320 (10) | 0.0272 (10) | −0.0021 (7) | 0.0011 (9) | 0.0028 (9) |
| N2 | 0.0400 (13) | 0.0201 (12) | 0.0247 (13) | −0.0001 (10) | 0.0015 (10) | −0.0041 (9) |
| N3 | 0.0250 (12) | 0.0307 (13) | 0.0245 (12) | 0.0010 (9) | −0.0009 (9) | 0.0021 (10) |
| C9 | 0.0230 (14) | 0.0255 (15) | 0.0213 (15) | −0.0023 (11) | −0.0003 (11) | −0.0002 (12) |
| C10 | 0.0362 (16) | 0.0238 (15) | 0.0306 (17) | −0.0010 (12) | 0.0000 (12) | −0.0010 (13) |
| C11 | 0.0428 (18) | 0.0298 (16) | 0.0304 (17) | −0.0056 (14) | −0.0008 (12) | 0.0090 (13) |
| C12 | 0.0383 (17) | 0.0454 (19) | 0.0202 (16) | −0.0005 (13) | −0.0033 (12) | 0.0007 (13) |
| C13 | 0.0422 (18) | 0.0308 (17) | 0.0279 (16) | 0.0054 (13) | −0.0027 (12) | −0.0089 (13) |
| C14 | 0.0374 (16) | 0.0256 (15) | 0.0264 (16) | 0.0020 (12) | −0.0050 (12) | 0.0008 (12) |
| C15 | 0.0281 (14) | 0.0456 (17) | 0.0334 (16) | −0.0015 (13) | −0.0007 (14) | −0.0054 (14) |
| C16 | 0.0383 (18) | 0.060 (2) | 0.045 (2) | −0.0006 (16) | 0.0093 (15) | −0.0028 (16) |
| C17 | 0.0384 (16) | 0.0291 (16) | 0.056 (2) | −0.0014 (13) | 0.0010 (14) | 0.0036 (14) |
| C18 | 0.0276 (15) | 0.0327 (16) | 0.0341 (16) | −0.0017 (12) | 0.0008 (11) | −0.0017 (13) |
| C19 | 0.0342 (16) | 0.0357 (17) | 0.0277 (14) | 0.0065 (13) | 0.0007 (11) | 0.0078 (13) |
| C20 | 0.054 (2) | 0.0384 (18) | 0.050 (2) | 0.0042 (15) | 0.0052 (16) | 0.0090 (15) |
| P2 | 0.0246 (3) | 0.0225 (3) | 0.0213 (3) | −0.0017 (3) | 0.0002 (3) | −0.0004 (3) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O4 | 0.0334 (10) | 0.0304 (10) | 0.0208 (10) | −0.0043 (8) | 0.0031 (8) | −0.0011 (9) |
| O5 | 0.0292 (10) | 0.0307 (10) | 0.0320 (11) | −0.0062 (8) | −0.0044 (8) | 0.0011 (8) |
| O6 | 0.0264 (9) | 0.0299 (10) | 0.0319 (10) | 0.0012 (8) | −0.0018 (8) | −0.0045 (9) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N1—C4 | 1.399 (3) | N2—C9 | 1.408 (3) |
| N1—P1 | 1.670 (2) | N2—P2 | 1.651 (2) |
| N1—H1B | 0.8600 | N2—H2B | 0.8600 |
| N4—C23 | 1.481 (3) | N3—C18 | 1.478 (3) |
| N4—C22 | 1.485 (3) | N3—C19 | 1.480 (3) |
| N4—H4A | 0.9000 | N3—H3B | 0.9000 |
| N4—H4B | 0.9000 | N3—H3C | 0.9000 |
| C1—C6 | 1.374 (4) | C9—C14 | 1.391 (4) |
| C1—C2 | 1.393 (4) | C9—C10 | 1.397 (4) |
| C1—H1A | 0.9300 | C10—C11 | 1.375 (4) |
| C2—C3 | 1.383 (4) | C10—H10A | 0.9300 |
| C2—H2A | 0.9300 | C11—C12 | 1.378 (4) |
| C3—C4 | 1.399 (4) | C11—H11A | 0.9300 |
| C3—H3A | 0.9300 | C12—C13 | 1.382 (4) |
| C4—C5 | 1.399 (3) | C12—H12A | 0.9300 |
| C5—C6 | 1.387 (4) | C13—C14 | 1.389 (4) |
| C5—H5A | 0.9300 | C13—H13A | 0.9300 |
| C6—H6A | 0.9300 | C14—H14A | 0.9300 |
| C7—O2 | 1.447 (3) | C15—O6 | 1.444 (3) |
| C7—C8 | 1.507 (4) | C15—C16 | 1.487 (4) |
| C7—H7A | 0.9700 | C15—H15A | 0.9700 |
| C7—H7B | 0.9700 | C15—H15B | 0.9700 |
| C8—H8A | 0.9600 | C16—H16A | 0.9600 |
| C8—H8B | 0.9600 | C16—H16B | 0.9600 |
| C8—H8C | 0.9600 | C16—H16C | 0.9600 |
| C21—C22 | 1.498 (4) | C17—C18 | 1.507 (4) |
| C21—H21A | 0.9600 | C17—H17A | 0.9600 |
| C21—H21B | 0.9600 | C17—H17B | 0.9600 |
| C21—H21C | 0.9600 | C17—H17C | 0.9600 |
| C22—H22A | 0.9700 | C18—H18A | 0.9700 |
| C22—H22B | 0.9700 | C18—H18B | 0.9700 |
| C23—C24 | 1.504 (4) | C19—C20 | 1.509 (4) |
| C23—H23A | 0.9700 | C19—H19A | 0.9700 |
| C23—H23B | 0.9700 | C19—H19B | 0.9700 |
| C24—H24A | 0.9600 | C20—H20A | 0.9600 |
| C24—H24B | 0.9600 | C20—H20B | 0.9600 |
| C24—H24C | 0.9600 | C20—H20C | 0.9600 |
| P1—O1 | 1.4798 (19) | P2—O5 | 1.4897 (18) |
| P1—O3 | 1.4994 (17) | P2—O4 | 1.4983 (19) |
| P1—O2 | 1.5979 (18) | P2—O6 | 1.5969 (17) |
| | | | |
| C4—N1—P1 | 128.36 (18) | C9—N2—P2 | 128.40 (19) |

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|---|---|---|---|
| C4—N1—H1B | 115.8 | C9—N2—H2B | 115.8 |
| P1—N1—H1B | 115.8 | P2—N2—H2B | 115.8 |
| C23—N4—C22 | 113.87 (19) | C18—N3—C19 | 113.6 (2) |
| C23—N4—H4A | 108.8 | C18—N3—H3B | 108.9 |
| C22—N4—H4A | 108.8 | C19—N3—H3B | 108.9 |
| C23—N4—H4B | 108.8 | C18—N3—H3C | 108.9 |
| C22—N4—H4B | 108.8 | C19—N3—H3C | 108.9 |
| H4A—N4—H4B | 107.7 | H3B—N3—H3C | 107.7 |
| C6—C1—C2 | 119.3 (3) | C14—C9—C10 | 117.9 (2) |
| C6—C1—H1A | 120.4 | C14—C9—N2 | 122.5 (2) |
| C2—C1—H1A | 120.4 | C10—C9—N2 | 119.6 (2) |
| C3—C2—C1 | 120.1 (3) | C11—C10—C9 | 121.2 (3) |
| C3—C2—H2A | 120.0 | C11—C10—H10A | 119.4 |
| C1—C2—H2A | 120.0 | C9—C10—H10A | 119.4 |
| C2—C3—C4 | 121.1 (3) | C10—C11—C12 | 120.6 (3) |
| C2—C3—H3A | 119.5 | C10—C11—H11A | 119.7 |
| C4—C3—H3A | 119.5 | C12—C11—H11A | 119.7 |
| C3—C4—N1 | 119.7 (2) | C11—C12—C13 | 119.2 (3) |
| C3—C4—C5 | 118.2 (2) | C11—C12—H12A | 120.4 |
| N1—C4—C5 | 122.1 (2) | C13—C12—H12A | 120.4 |
| C6—C5—C4 | 120.2 (2) | C12—C13—C14 | 120.6 (3) |
| C6—C5—H5A | 119.9 | C12—C13—H13A | 119.7 |
| C4—C5—H5A | 119.9 | C14—C13—H13A | 119.7 |
| C1—C6—C5 | 121.2 (3) | C13—C14—C9 | 120.6 (3) |
| C1—C6—H6A | 119.4 | C13—C14—H14A | 119.7 |
| C5—C6—H6A | 119.4 | C9—C14—H14A | 119.7 |
| O2—C7—C8 | 108.4 (2) | O6—C15—C16 | 110.0 (2) |
| O2—C7—H7A | 110.0 | O6—C15—H15A | 109.7 |
| C8—C7—H7A | 110.0 | C16—C15—H15A | 109.7 |
| O2—C7—H7B | 110.0 | O6—C15—H15B | 109.7 |
| C8—C7—H7B | 110.0 | C16—C15—H15B | 109.7 |
| H7A—C7—H7B | 108.4 | H15A—C15—H15B | 108.2 |
| C7—C8—H8A | 109.5 | C15—C16—H16A | 109.5 |
| C7—C8—H8B | 109.5 | C15—C16—H16B | 109.5 |
| H8A—C8—H8B | 109.5 | H16A—C16—H16B | 109.5 |
| C7—C8—H8C | 109.5 | C15—C16—H16C | 109.5 |
| H8A—C8—H8C | 109.5 | H16A—C16—H16C | 109.5 |
| H8B—C8—H8C | 109.5 | H16B—C16—H16C | 109.5 |
| C22—C21—H21A | 109.5 | C18—C17—H17A | 109.5 |
| C22—C21—H21B | 109.5 | C18—C17—H17B | 109.5 |
| H21A—C21—H21B | 109.5 | H17A—C17—H17B | 109.5 |
| C22—C21—H21C | 109.5 | C18—C17—H17C | 109.5 |
| H21A—C21—H21C | 109.5 | H17A—C17—H17C | 109.5 |
| H21B—C21—H21C | 109.5 | H17B—C17—H17C | 109.5 |
| N4—C22—C21 | 111.6 (2) | N3—C18—C17 | 110.7 (2) |
| N4—C22—H22A | 109.3 | N3—C18—H18A | 109.5 |
| C21—C22—H22A | 109.3 | C17—C18—H18A | 109.5 |
| N4—C22—H22B | 109.3 | N3—C18—H18B | 109.5 |

| | | | |
|---|---|---|---|
| C21—C22—H22B | 109.3 | C17—C18—H18B | 109.5 |
| H22A—C22—H22B | 108.0 | H18A—C18—H18B | 108.1 |
| N4—C23—C24 | 111.6 (2) | N3—C19—C20 | 111.6 (2) |
| N4—C23—H23A | 109.3 | N3—C19—H19A | 109.3 |
| C24—C23—H23A | 109.3 | C20—C19—H19A | 109.3 |
| N4—C23—H23B | 109.3 | N3—C19—H19B | 109.3 |
| C24—C23—H23B | 109.3 | C20—C19—H19B | 109.3 |
| H23A—C23—H23B | 108.0 | H19A—C19—H19B | 108.0 |
| C23—C24—H24A | 109.5 | C19—C20—H20A | 109.5 |
| C23—C24—H24B | 109.5 | C19—C20—H20B | 109.5 |
| H24A—C24—H24B | 109.5 | H20A—C20—H20B | 109.5 |
| C23—C24—H24C | 109.5 | C19—C20—H20C | 109.5 |
| H24A—C24—H24C | 109.5 | H20A—C20—H20C | 109.5 |
| H24B—C24—H24C | 109.5 | H20B—C20—H20C | 109.5 |
| O1—P1—O3 | 119.11 (11) | O5—P2—O4 | 118.09 (10) |
| O1—P1—O2 | 111.87 (10) | O5—P2—O6 | 103.68 (10) |
| O3—P1—O2 | 103.01 (9) | O4—P2—O6 | 110.52 (10) |
| O1—P1—N1 | 106.21 (10) | O5—P2—N2 | 112.25 (11) |
| O3—P1—N1 | 110.60 (10) | O4—P2—N2 | 105.31 (11) |
| O2—P1—N1 | 105.30 (10) | O6—P2—N2 | 106.54 (10) |
| C7—O2—P1 | 119.30 (16) | C15—O6—P2 | 118.84 (16) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*B*···O4 | 0.86 | 2.13 | 2.954 (3) | 161 |
| N2—H2*B*···O1 | 0.86 | 2.04 | 2.895 (3) | 173 |
| N3—H3*C*···O4 | 0.90 | 1.88 | 2.742 (3) | 160 |
| N4—H4*A*···O3 | 0.90 | 1.94 | 2.792 (3) | 158 |
| N3—H3*B*···O3$^{i}$ | 0.90 | 1.89 | 2.788 (3) | 174 |
| N4—H4*B*···O5$^{ii}$ | 0.90 | 1.74 | 2.637 (3) | 172 |

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