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# 1,10-Phenanthrolin-1-ium 1-oxo-2,6,7-trioxa-1-phosphabicyclo[2.2.2]octane-4-carboxylate 1-oxo-2,6,7-trioxa-1-phosphabicyclo[2.2.2]octane-4-carboxylic acid monohydrate

**Zhi Wang, Hong-Jun Zang* and Ming-Lin Guo**

College of Materials and Chemical Engineering, Tianjin Polytechnic University, Tianjin 300160, People's Republic of China
Correspondence e-mail: chemhong@126.com

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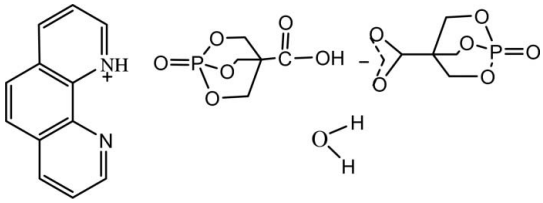
Key indicators: single-crystal X-ray study; $T$ = 294 K; mean $\sigma$(C–C) = 0.005 Å; disorder in main residue; $R$ factor = 0.053; $wR$ factor = 0.159; data-to-parameter ratio = 13.5.

The asymmetric unit of the title salt, $C_{12}H_9N_2^+\cdot C_5H_6O_6P^-\cdot C_5H_7O_6P\cdot H_2O$, contains one 1,10-phenanthrolinium cation, one 1-oxo-2,6,7-trioxa-1-phosphabicyclo[2.2.2]octane-4-carboxylate anion, one 1-oxo-2,6,7-trioxa-1-phospha-bicyclo-[2.2.2]octane-4-carboxylic acid neutral molecule and one water molecule. In the anion, the oxygen atoms of the carboxylate group are disordered over two positions, with site occupation factors *ca* 0.7:0.3. In the crystal structure, intermolecular O—H···O and N—H···O hydrogen-bonding interactions result in the formation of a three-dimensional network.

## Related literature

For related literature, see: Guo (2005); Hensen *et al.* (1998, 2000).



## Experimental

### Crystal data

$C_{12}H_9N_2^+\cdot C_5H_6O_6P^-\cdot C_5H_7O_6P\cdot H_2O$
$M_r$ = 586.37
Monoclinic, $P2_1/c$
$a$ = 9.2841 (18) Å
$b$ = 8.5863 (16) Å
$c$ = 31.124 (6) Å
$\beta$ = 99.917 (5)°
$V$ = 2444.0 (8) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.25 mm$^{-1}$
$T$ = 294 (2) K
0.20 × 0.16 × 0.12 mm

### Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.948, $T_{max}$ = 0.972
13684 measured reflections
5008 independent reflections
2956 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.043

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.159
$S$ = 1.03
5008 reflections
371 parameters
38 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.35 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.32 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O13—H13$B$···O12$^{i}$ | 0.84 | 2.47 | 2.893 (9) | 112 |
| N1—H1···O1$^{ii}$ | 0.90 | 1.91 | 2.765 (3) | 159 |
| O6—H6$C$···O11$^{i}$ | 0.85 | 1.61 | 2.459 (5) | 173 |

Symmetry codes: (i) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x+2, -y+1, -z+2$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2150).

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# supplementary materials

*Acta Cryst.* (2007). E**63**, o3418 [ **doi:10.1107/S1600536807032813** ]

# 1,10-Phenanthrolin-1-ium 1-oxo-2,6,7-trioxa-1-phosphabicyclo[2.2.2]octane-4-carboxylate 1-oxo-2,6,7-trioxa-1-phosphabicyclo[2.2.2]octane-4-carboxylic acid monohydrate

**Z. Wang, H.-J. Zang and M.-L. Guo**

## Comment

Caged bicyclic phosphates are widely used as flame retardants in some polymers. As a low-toxicity and environmentally friendly flame retardant system, the phosphorus-nitrogen combination has been extensively studied. We report here the crystal structure of a new bicyclic phosphate cage compound.

The content of the asymmetric unit of the title salt is shown in Fig. 1. In the 1,10-phenanthrolinium cation, the C—N—C bond angle of 123.0 (3)° at the protonated N1 atom is significantly larger than that at the non-protonated N2 atom (116.4 (3)°). This widening is not unexpected, as analogous differences were already observed in previously reported monoprotonated phenanthrolines (Hensen *et al.*, 1998, 2000; Guo, 2005). In the anion, the oxygen atoms O11 and O12 of the carboxylate group are positionally disordered over two position, with site occupation factors of 0.701 (15) and 0.299 (15) for the major and minor components respectively. In the crystal structure, ions and neutral molecules are linked into an extended three-dimensional network by O—H···O and N—H···O hydrogen bonding interactions (Table 1, Fig. 2).

## Experimental

The title salt was prepared by mixing ethanol solutions of 1-oxo-2,6,7-trioxa-1-phospha-bicyclo[2.2.2]octane-4-carboxylic acid (1.94 g in 10 ml) and 1,10-phenanthroline (1.8 g in 10 ml). The solution was stirred at room temperature for 10 min, after which the crystalline product was separated by filtration. The pure product (1.0 g) was heated and dissolved in water (25 ml). Single crystals suitable for X-ray analysis were obtained from this aqueous solution by slow evaporation of the solvent over a period of five days at room temperature.

## Refinement

All H atoms were included in the refinement in the riding model approximation, with N–H = 0.90 Å, C–H = 0.93–0.97 Å, and $U_{iso}$(H) = 1.2 $U_{eq}$(C, N).

## Figures

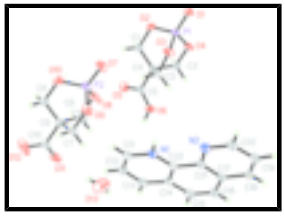

Fig. 1. The structure of the title compound, showing the atom-numbering scheme and displacement ellipsoids drawn at the 30% probability level. Only the major component of disorder is shown.

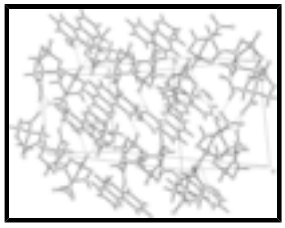

Fig. 2. Packing diagram of the title compound, showing hydrogen-bond interactions as dashed lines.

**1,10-Phenanthrolin-1-ium 1-oxo-2,6,7-trioxa-1-phosphabicyclo[2.2.2]octane-4-carboxylate 1-oxo-2,6,7-trioxa-1-phosphabicyclo[2.2.2]octane-4-carboxylic acid monohydrate**

*Crystal data*

| | |
|---|---|
| $C_{12}H_9N_2^+ \cdot C_5H_6O_6P^- \cdot C_5H_7O_6P \cdot H_2O$ | $F_{000}$ = 1216 |
| $M_r$ = 586.37 | $D_x$ = 1.594 Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation<br>λ = 0.71073 Å |
| Hall symbol: -P 2ybc | Cell parameters from 2791 reflections |
| $a$ = 9.2841 (18) Å | θ = 2.5–26.3º |
| $b$ = 8.5863 (16) Å | μ = 0.25 mm$^{-1}$ |
| $c$ = 31.124 (6) Å | $T$ = 294 (2) K |
| β = 99.917 (5)º | Prism, colorless |
| $V$ = 2444.0 (8) Å$^3$ | 0.20 × 0.16 × 0.12 mm |
| $Z$ = 4 | |

*Data collection*

| | |
|---|---|
| Bruker SMART CCD area detector diffractometer | 5008 independent reflections |
| Radiation source: fine-focus sealed tube | 2956 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int}$ = 0.043 |
| $T$ = 294(2) K | $\theta_{max}$ = 26.4º |
| φ and ω scans | $\theta_{min}$ = 1.3º |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | $h$ = −11→8 |
| $T_{min}$ = 0.948, $T_{max}$ = 0.972 | $k$ = −10→10 |
| 13684 measured reflections | $l$ = −38→36 |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)]$ = 0.053 | H-atom parameters constrained |
| $wR(F^2)$ = 0.159 | $w = 1/[\sigma^2(F_o^2) + (0.0773P)^2 + 0.655P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S$ = 1.03 | $(\Delta/\sigma)_{max}$ = 0.001 |
| 5008 reflections | $\Delta\rho_{max}$ = 0.35 e Å$^{-3}$ |

| | |
|---|---|
| 371 parameters | $\Delta\rho_{min} = -0.32$ e Å$^{-3}$ |
| 38 restraints | Extinction correction: none |
| Primary atom site location: structure-invariant direct methods | |

*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 $F^2 > 2\text{sigma}(F^2)$ 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* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| P1 | 1.15068 (9) | 0.02050 (10) | 0.95898 (3) | 0.0336 (2) | |
| O1 | 1.2593 (2) | 0.0302 (3) | 0.99848 (7) | 0.0451 (6) | |
| O2 | 1.2147 (2) | 0.0221 (3) | 0.91570 (7) | 0.0514 (7) | |
| O3 | 1.0366 (2) | 0.1551 (3) | 0.95336 (7) | 0.0440 (6) | |
| O4 | 1.0576 (2) | −0.1324 (3) | 0.95573 (7) | 0.0460 (6) | |
| O5 | 0.8678 (3) | −0.0305 (3) | 0.81115 (7) | 0.0533 (7) | |
| O6 | 0.7046 (2) | −0.0035 (3) | 0.85531 (7) | 0.0554 (7) | |
| H6C | 0.6335 | −0.0100 | 0.8339 | 0.066* | |
| C1 | 1.1025 (4) | 0.0206 (5) | 0.87599 (10) | 0.0477 (9) | |
| H1A | 1.1220 | −0.0637 | 0.8571 | 0.057* | |
| H1B | 1.1055 | 0.1178 | 0.8603 | 0.057* | |
| C2 | 0.9191 (3) | 0.1378 (4) | 0.91576 (10) | 0.0387 (8) | |
| H2A | 0.9109 | 0.2321 | 0.8983 | 0.046* | |
| H2B | 0.8268 | 0.1210 | 0.9256 | 0.046* | |
| C3 | 0.9521 (4) | −0.1485 (4) | 0.91557 (10) | 0.0426 (8) | |
| H3A | 0.8552 | −0.1652 | 0.9224 | 0.051* | |
| H3B | 0.9772 | −0.2377 | 0.8992 | 0.051* | |
| C4 | 0.9527 (3) | −0.0008 (3) | 0.88813 (9) | 0.0308 (7) | |
| C5 | 0.8348 (4) | −0.0131 (4) | 0.84658 (10) | 0.0370 (8) | |
| P2 | 0.91080 (11) | 0.49646 (13) | 0.82372 (3) | 0.0523 (3) | |
| O7 | 1.0150 (3) | 0.4764 (4) | 0.86362 (9) | 0.0841 (10) | |
| O8 | 0.8420 (4) | 0.3432 (3) | 0.80474 (10) | 0.0960 (11) | |
| O9 | 0.7750 (3) | 0.5977 (4) | 0.82963 (7) | 0.0649 (8) | |
| O10 | 0.9709 (3) | 0.5822 (4) | 0.78669 (8) | 0.0795 (10) | |
| C6 | 0.7333 (5) | 0.3608 (5) | 0.76470 (14) | 0.0862 (13) | |
| H6A | 0.6396 | 0.3205 | 0.7694 | 0.103* | |
| H6B | 0.7638 | 0.3021 | 0.7412 | 0.103* | |
| C7 | 0.6693 (4) | 0.6187 (5) | 0.78974 (10) | 0.0575 (10) | |
| H7A | 0.6594 | 0.7287 | 0.7828 | 0.069* | |

| | | | | | |
|---|---|---|---|---|---|
| H7B | 0.5747 | 0.5798 | 0.7940 | 0.069* | |
| C8 | 0.8643 (4) | 0.5989 (6) | 0.74632 (11) | 0.0712 (12) | |
| H8A | 0.8993 | 0.5447 | 0.7228 | 0.085* | |
| H8B | 0.8530 | 0.7081 | 0.7386 | 0.085* | |
| C9 | 0.7185 (4) | 0.5323 (4) | 0.75239 (10) | 0.0440 (8) | |
| C10 | 0.6033 (4) | 0.5554 (5) | 0.71121 (11) | 0.0549 (10) | |
| O11 | 0.5054 (7) | 0.4583 (8) | 0.7037 (2) | 0.0620 (19) | 0.701 (15) |
| O12 | 0.6099 (9) | 0.6825 (11) | 0.6909 (3) | 0.123 (4) | 0.701 (15) |
| O11' | 0.4766 (12) | 0.5159 (19) | 0.7151 (4) | 0.048 (3) | 0.299 (15) |
| O12' | 0.6430 (10) | 0.553 (3) | 0.6759 (3) | 0.075 (7) | 0.299 (15) |
| N1 | 0.5028 (3) | 0.8006 (3) | 0.96061 (8) | 0.0342 (6) | |
| H1 | 0.5655 | 0.8589 | 0.9790 | 0.041* | |
| N2 | 0.5012 (3) | 0.7655 (3) | 1.04740 (8) | 0.0381 (7) | |
| C11 | 0.5104 (4) | 0.8262 (4) | 0.91909 (10) | 0.0386 (8) | |
| H11 | 0.5743 | 0.9009 | 0.9116 | 0.046* | |
| C12 | 0.4235 (4) | 0.7417 (4) | 0.88690 (10) | 0.0422 (8) | |
| H12 | 0.4296 | 0.7583 | 0.8577 | 0.051* | |
| C13 | 0.3281 (4) | 0.6333 (4) | 0.89802 (10) | 0.0399 (8) | |
| H13 | 0.2697 | 0.5762 | 0.8763 | 0.048* | |
| C14 | 0.3182 (3) | 0.6079 (4) | 0.94211 (9) | 0.0339 (7) | |
| C15 | 0.2217 (4) | 0.4975 (4) | 0.95668 (11) | 0.0402 (8) | |
| H15 | 0.1603 | 0.4379 | 0.9363 | 0.048* | |
| C16 | 0.2182 (4) | 0.4786 (4) | 0.99920 (11) | 0.0405 (8) | |
| H16 | 0.1543 | 0.4060 | 1.0078 | 0.049* | |
| C17 | 0.3105 (3) | 0.5678 (4) | 1.03176 (10) | 0.0345 (7) | |
| C18 | 0.3133 (4) | 0.5502 (4) | 1.07702 (11) | 0.0410 (8) | |
| H18 | 0.2517 | 0.4791 | 1.0873 | 0.049* | |
| C19 | 0.4072 (4) | 0.6385 (4) | 1.10530 (10) | 0.0450 (9) | |
| H19 | 0.4103 | 0.6283 | 1.1352 | 0.054* | |
| C20 | 0.4988 (4) | 0.7445 (4) | 1.08938 (10) | 0.0437 (8) | |
| H20 | 0.5617 | 0.8039 | 1.1094 | 0.052* | |
| C21 | 0.4079 (3) | 0.6769 (3) | 1.01926 (9) | 0.0316 (7) | |
| C22 | 0.4103 (3) | 0.6958 (3) | 0.97354 (9) | 0.0307 (7) | |
| O13 | 0.3103 (4) | 0.5046 (4) | 0.79112 (13) | 0.1197 (14) | |
| H13A | 0.3580 | 0.5070 | 0.7702 | 0.144* | |
| H13B | 0.2572 | 0.4255 | 0.7859 | 0.144* | |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| P1 | 0.0254 (4) | 0.0425 (5) | 0.0304 (4) | −0.0021 (4) | −0.0018 (3) | −0.0021 (4) |
| O1 | 0.0347 (13) | 0.0596 (15) | 0.0368 (13) | −0.0045 (11) | −0.0062 (10) | −0.0028 (11) |
| O2 | 0.0258 (12) | 0.0915 (19) | 0.0353 (13) | −0.0075 (13) | 0.0008 (10) | −0.0068 (12) |
| O3 | 0.0435 (13) | 0.0436 (13) | 0.0386 (13) | 0.0066 (11) | −0.0103 (11) | −0.0131 (10) |
| O4 | 0.0449 (13) | 0.0411 (13) | 0.0435 (13) | −0.0065 (11) | −0.0160 (11) | 0.0080 (10) |
| O5 | 0.0522 (16) | 0.0778 (18) | 0.0279 (13) | −0.0068 (14) | 0.0014 (11) | −0.0081 (12) |
| O6 | 0.0284 (13) | 0.096 (2) | 0.0361 (13) | −0.0018 (13) | −0.0098 (10) | −0.0016 (12) |
| C1 | 0.0307 (18) | 0.079 (3) | 0.0318 (18) | −0.0028 (18) | 0.0019 (14) | −0.0056 (17) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C2 | 0.0358 (18) | 0.0419 (19) | 0.0338 (17) | 0.0050 (16) | −0.0065 (14) | 0.0004 (14) |
| C3 | 0.0415 (19) | 0.0357 (18) | 0.0437 (19) | −0.0049 (16) | −0.0126 (16) | −0.0013 (15) |
| C4 | 0.0280 (16) | 0.0373 (17) | 0.0253 (14) | 0.0001 (14) | −0.0009 (13) | −0.0020 (13) |
| C5 | 0.0346 (19) | 0.0387 (19) | 0.0347 (18) | −0.0041 (15) | −0.0020 (15) | 0.0002 (14) |
| P2 | 0.0441 (6) | 0.0700 (7) | 0.0351 (5) | −0.0119 (5) | −0.0156 (4) | 0.0103 (5) |
| O7 | 0.0680 (19) | 0.121 (3) | 0.0496 (17) | −0.0271 (19) | −0.0275 (15) | 0.0276 (16) |
| O8 | 0.114 (2) | 0.0543 (17) | 0.091 (2) | 0.0021 (17) | −0.0649 (19) | 0.0064 (15) |
| O9 | 0.0637 (18) | 0.098 (2) | 0.0291 (13) | −0.0002 (17) | −0.0041 (12) | −0.0061 (13) |
| O10 | 0.0362 (15) | 0.152 (3) | 0.0465 (15) | −0.0141 (18) | −0.0035 (12) | 0.0238 (17) |
| C6 | 0.097 (3) | 0.062 (2) | 0.078 (2) | 0.005 (2) | −0.049 (2) | −0.005 (2) |
| C7 | 0.049 (2) | 0.080 (3) | 0.0385 (19) | 0.008 (2) | −0.0056 (17) | 0.0008 (18) |
| C8 | 0.044 (2) | 0.132 (3) | 0.0354 (19) | 0.002 (2) | −0.0004 (17) | 0.020 (2) |
| C9 | 0.0395 (16) | 0.0621 (18) | 0.0262 (14) | 0.0012 (15) | −0.0059 (13) | 0.0023 (14) |
| C10 | 0.047 (2) | 0.082 (3) | 0.0306 (19) | 0.003 (2) | −0.0077 (17) | 0.0063 (19) |
| O11 | 0.057 (3) | 0.054 (3) | 0.061 (4) | −0.004 (3) | −0.029 (3) | −0.002 (3) |
| O12 | 0.142 (6) | 0.109 (6) | 0.086 (5) | −0.055 (5) | −0.069 (4) | 0.050 (5) |
| O11' | 0.039 (5) | 0.058 (6) | 0.043 (5) | −0.005 (4) | −0.003 (3) | −0.008 (4) |
| O12' | 0.034 (5) | 0.167 (19) | 0.023 (5) | −0.007 (7) | 0.004 (4) | 0.017 (6) |
| N1 | 0.0300 (14) | 0.0383 (15) | 0.0320 (14) | −0.0016 (12) | −0.0013 (11) | −0.0033 (12) |
| N2 | 0.0384 (16) | 0.0431 (16) | 0.0301 (14) | −0.0013 (13) | −0.0012 (12) | −0.0031 (12) |
| C11 | 0.0359 (18) | 0.0440 (19) | 0.0356 (18) | −0.0008 (16) | 0.0054 (15) | 0.0050 (15) |
| C12 | 0.041 (2) | 0.054 (2) | 0.0303 (17) | 0.0072 (18) | 0.0030 (15) | 0.0022 (15) |
| C13 | 0.0374 (18) | 0.046 (2) | 0.0321 (17) | 0.0033 (16) | −0.0059 (15) | −0.0053 (14) |
| C14 | 0.0286 (16) | 0.0368 (18) | 0.0338 (17) | 0.0035 (15) | −0.0022 (14) | −0.0037 (14) |
| C15 | 0.0321 (18) | 0.0385 (19) | 0.0459 (19) | −0.0049 (15) | −0.0049 (15) | −0.0050 (15) |
| C16 | 0.0355 (19) | 0.0371 (19) | 0.048 (2) | −0.0038 (15) | 0.0040 (16) | 0.0040 (15) |
| C17 | 0.0321 (17) | 0.0329 (17) | 0.0376 (18) | 0.0078 (14) | 0.0038 (14) | 0.0015 (14) |
| C18 | 0.044 (2) | 0.0394 (19) | 0.0413 (19) | 0.0061 (16) | 0.0128 (16) | 0.0061 (15) |
| C19 | 0.050 (2) | 0.054 (2) | 0.0322 (18) | 0.0080 (19) | 0.0088 (16) | 0.0034 (16) |
| C20 | 0.042 (2) | 0.051 (2) | 0.0351 (19) | 0.0040 (17) | −0.0004 (16) | −0.0075 (16) |
| C21 | 0.0294 (16) | 0.0341 (17) | 0.0296 (16) | 0.0061 (14) | 0.0003 (13) | −0.0016 (13) |
| C22 | 0.0267 (15) | 0.0322 (16) | 0.0312 (16) | 0.0027 (14) | −0.0011 (13) | 0.0002 (13) |
| O13 | 0.093 (3) | 0.120 (3) | 0.149 (4) | −0.018 (2) | 0.029 (3) | −0.022 (2) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| P1—O1 | 1.452 (2) | C8—H8A | 0.9700 |
| P1—O3 | 1.557 (2) | C8—H8B | 0.9700 |
| P1—O2 | 1.563 (2) | C9—C10 | 1.535 (4) |
| P1—O4 | 1.565 (2) | C10—O12' | 1.218 (8) |
| O2—C1 | 1.473 (4) | C10—O11 | 1.225 (6) |
| O3—C2 | 1.463 (3) | C10—O11' | 1.250 (11) |
| O4—C3 | 1.455 (3) | C10—O12 | 1.268 (6) |
| O5—C5 | 1.204 (4) | N1—C11 | 1.325 (4) |
| O6—C5 | 1.286 (4) | N1—C22 | 1.352 (4) |
| O6—H6C | 0.8548 | N1—H1 | 0.8964 |
| C1—C4 | 1.513 (4) | N2—C20 | 1.323 (4) |
| C1—H1A | 0.9700 | N2—C21 | 1.354 (4) |
| C1—H1B | 0.9700 | C11—C12 | 1.379 (4) |

| | | | |
|---|---|---|---|
| C2—C4 | 1.532 (4) | C11—H11 | 0.9300 |
| C2—H2A | 0.9700 | C12—C13 | 1.369 (5) |
| C2—H2B | 0.9700 | C12—H12 | 0.9300 |
| C3—C4 | 1.529 (4) | C13—C14 | 1.408 (4) |
| C3—H3A | 0.9700 | C13—H13 | 0.9300 |
| C3—H3B | 0.9700 | C14—C22 | 1.404 (4) |
| C4—C5 | 1.547 (4) | C14—C15 | 1.430 (4) |
| P2—O7 | 1.447 (3) | C15—C16 | 1.339 (5) |
| P2—O8 | 1.536 (3) | C15—H15 | 0.9300 |
| P2—O10 | 1.550 (3) | C16—C17 | 1.431 (4) |
| P2—O9 | 1.568 (3) | C16—H16 | 0.9300 |
| O8—C6 | 1.470 (4) | C17—C21 | 1.403 (4) |
| O9—C7 | 1.455 (4) | C17—C18 | 1.413 (4) |
| O10—C8 | 1.467 (4) | C18—C19 | 1.359 (5) |
| C6—C9 | 1.522 (5) | C18—H18 | 0.9300 |
| C6—H6A | 0.9700 | C19—C20 | 1.393 (5) |
| C6—H6B | 0.9700 | C19—H19 | 0.9300 |
| C7—C9 | 1.514 (5) | C20—H20 | 0.9300 |
| C7—H7A | 0.9700 | C21—C22 | 1.436 (4) |
| C7—H7B | 0.9700 | O13—H13A | 0.8488 |
| C8—C9 | 1.511 (5) | O13—H13B | 0.8393 |
| O1—P1—O3 | 114.37 (13) | C9—C8—H8A | 109.7 |
| O1—P1—O2 | 114.69 (14) | O10—C8—H8B | 109.7 |
| O3—P1—O2 | 104.42 (13) | C9—C8—H8B | 109.7 |
| O1—P1—O4 | 113.44 (13) | H8A—C8—H8B | 108.2 |
| O3—P1—O4 | 104.95 (13) | C8—C9—C7 | 107.8 (3) |
| O2—P1—O4 | 103.81 (13) | C8—C9—C6 | 110.7 (4) |
| C1—O2—P1 | 113.86 (19) | C7—C9—C6 | 107.9 (3) |
| C2—O3—P1 | 114.43 (18) | C8—C9—C10 | 110.9 (3) |
| C3—O4—P1 | 114.66 (18) | C7—C9—C10 | 108.6 (3) |
| C5—O6—H6C | 117.4 | C6—C9—C10 | 110.8 (3) |
| O2—C1—C4 | 109.8 (2) | O12'—C10—O11 | 98.3 (8) |
| O2—C1—H1A | 109.7 | O12'—C10—O11' | 121.3 (8) |
| C4—C1—H1A | 109.7 | O12'—C10—O12 | 61.1 (7) |
| O2—C1—H1B | 109.7 | O11—C10—O12 | 126.0 (4) |
| C4—C1—H1B | 109.7 | O11'—C10—O12 | 114.1 (7) |
| H1A—C1—H1B | 108.2 | O12'—C10—C9 | 118.4 (5) |
| O3—C2—C4 | 109.4 (2) | O11—C10—C9 | 117.4 (4) |
| O3—C2—H2A | 109.8 | O11'—C10—C9 | 114.6 (6) |
| C4—C2—H2A | 109.8 | O12—C10—C9 | 116.1 (4) |
| O3—C2—H2B | 109.8 | C11—N1—C22 | 123.0 (3) |
| C4—C2—H2B | 109.8 | C11—N1—H1 | 113.2 |
| H2A—C2—H2B | 108.2 | C22—N1—H1 | 123.8 |
| O4—C3—C4 | 109.5 (2) | C20—N2—C21 | 116.4 (3) |
| O4—C3—H3A | 109.8 | N1—C11—C12 | 119.9 (3) |
| C4—C3—H3A | 109.8 | N1—C11—H11 | 120.0 |
| O4—C3—H3B | 109.8 | C12—C11—H11 | 120.0 |
| C4—C3—H3B | 109.8 | C13—C12—C11 | 119.8 (3) |
| H3A—C3—H3B | 108.2 | C13—C12—H12 | 120.1 |

| | | | |
|---|---|---|---|
| C1—C4—C3 | 109.4 (3) | C11—C12—H12 | 120.1 |
| C1—C4—C2 | 109.3 (3) | C12—C13—C14 | 120.4 (3) |
| C3—C4—C2 | 108.1 (3) | C12—C13—H13 | 119.8 |
| C1—C4—C5 | 110.4 (2) | C14—C13—H13 | 119.8 |
| C3—C4—C5 | 109.9 (2) | C22—C14—C13 | 117.5 (3) |
| C2—C4—C5 | 109.8 (2) | C22—C14—C15 | 118.4 (3) |
| O5—C5—O6 | 126.8 (3) | C13—C14—C15 | 124.2 (3) |
| O5—C5—C4 | 121.3 (3) | C16—C15—C14 | 121.1 (3) |
| O6—C5—C4 | 112.0 (3) | C16—C15—H15 | 119.4 |
| O7—P2—O8 | 113.61 (17) | C14—C15—H15 | 119.4 |
| O7—P2—O10 | 114.93 (16) | C15—C16—C17 | 121.4 (3) |
| O8—P2—O10 | 107.47 (19) | C15—C16—H16 | 119.3 |
| O7—P2—O9 | 113.40 (18) | C17—C16—H16 | 119.3 |
| O8—P2—O9 | 103.32 (18) | C21—C17—C18 | 116.5 (3) |
| O10—P2—O9 | 102.94 (17) | C21—C17—C16 | 119.8 (3) |
| C6—O8—P2 | 114.7 (2) | C18—C17—C16 | 123.7 (3) |
| C7—O9—P2 | 114.0 (2) | C19—C18—C17 | 119.1 (3) |
| C8—O10—P2 | 114.0 (2) | C19—C18—H18 | 120.5 |
| O8—C6—C9 | 109.4 (3) | C17—C18—H18 | 120.5 |
| O8—C6—H6A | 109.8 | C18—C19—C20 | 119.8 (3) |
| C9—C6—H6A | 109.8 | C18—C19—H19 | 120.1 |
| O8—C6—H6B | 109.8 | C20—C19—H19 | 120.1 |
| C9—C6—H6B | 109.8 | N2—C20—C19 | 123.7 (3) |
| H6A—C6—H6B | 108.2 | N2—C20—H20 | 118.1 |
| O9—C7—C9 | 110.2 (3) | C19—C20—H20 | 118.1 |
| O9—C7—H7A | 109.6 | N2—C21—C17 | 124.5 (3) |
| C9—C7—H7A | 109.6 | N2—C21—C22 | 117.4 (3) |
| O9—C7—H7B | 109.6 | C17—C21—C22 | 118.1 (3) |
| C9—C7—H7B | 109.6 | N1—C22—C14 | 119.5 (3) |
| H7A—C7—H7B | 108.1 | N1—C22—C21 | 119.3 (3) |
| O10—C8—C9 | 110.0 (3) | C14—C22—C21 | 121.2 (3) |
| O10—C8—H8A | 109.7 | H13A—O13—H13B | 103.8 |
| O1—P1—O2—C1 | −176.9 (2) | O8—C6—C9—C7 | −60.0 (5) |
| O3—P1—O2—C1 | −51.0 (3) | O8—C6—C9—C10 | −178.8 (4) |
| O4—P1—O2—C1 | 58.7 (3) | C8—C9—C10—O12' | 32.4 (13) |
| O1—P1—O3—C2 | −174.5 (2) | C7—C9—C10—O12' | 150.7 (13) |
| O2—P1—O3—C2 | 59.3 (2) | C6—C9—C10—O12' | −91.0 (13) |
| O4—P1—O3—C2 | −49.6 (2) | C8—C9—C10—O11 | 150.2 (7) |
| O1—P1—O4—C3 | −177.4 (2) | C7—C9—C10—O11 | −91.5 (7) |
| O3—P1—O4—C3 | 57.0 (2) | C6—C9—C10—O11 | 26.8 (7) |
| O2—P1—O4—C3 | −52.3 (2) | C8—C9—C10—O11' | −173.8 (10) |
| P1—O2—C1—C4 | −6.5 (4) | C7—C9—C10—O11' | −55.5 (10) |
| P1—O3—C2—C4 | −7.5 (3) | C6—C9—C10—O11' | 62.8 (10) |
| P1—O4—C3—C4 | −4.5 (3) | C8—C9—C10—O12 | −37.3 (8) |
| O2—C1—C4—C3 | −55.4 (3) | C7—C9—C10—O12 | 81.0 (8) |
| O2—C1—C4—C2 | 62.7 (3) | C6—C9—C10—O12 | −160.7 (8) |
| O2—C1—C4—C5 | −176.4 (3) | C22—N1—C11—C12 | −1.3 (5) |
| O4—C3—C4—C1 | 61.9 (3) | N1—C11—C12—C13 | 0.9 (5) |
| O4—C3—C4—C2 | −57.0 (3) | C11—C12—C13—C14 | 0.2 (5) |

| | | | |
|---|---|---|---|
| O4—C3—C4—C5 | −176.9 (3) | C12—C13—C14—C22 | −0.8 (5) |
| O3—C2—C4—C1 | −54.9 (3) | C12—C13—C14—C15 | 179.9 (3) |
| O3—C2—C4—C3 | 64.1 (3) | C22—C14—C15—C16 | 0.4 (5) |
| O3—C2—C4—C5 | −176.1 (2) | C13—C14—C15—C16 | 179.7 (3) |
| C1—C4—C5—O5 | 12.0 (4) | C14—C15—C16—C17 | 0.0 (5) |
| C3—C4—C5—O5 | −108.7 (3) | C15—C16—C17—C21 | −0.4 (5) |
| C2—C4—C5—O5 | 132.6 (3) | C15—C16—C17—C18 | −178.7 (3) |
| C1—C4—C5—O6 | −168.5 (3) | C21—C17—C18—C19 | 0.4 (4) |
| C3—C4—C5—O6 | 70.9 (3) | C16—C17—C18—C19 | 178.7 (3) |
| C2—C4—C5—O6 | −47.9 (4) | C17—C18—C19—C20 | 0.0 (5) |
| O7—P2—O8—C6 | 178.7 (3) | C21—N2—C20—C19 | 0.0 (5) |
| O10—P2—O8—C6 | −53.0 (4) | C18—C19—C20—N2 | −0.3 (5) |
| O9—P2—O8—C6 | 55.4 (4) | C20—N2—C21—C17 | 0.5 (4) |
| O7—P2—O9—C7 | −180.0 (3) | C20—N2—C21—C22 | −179.0 (3) |
| O8—P2—O9—C7 | −56.5 (3) | C18—C17—C21—N2 | −0.7 (4) |
| O10—P2—O9—C7 | 55.2 (3) | C16—C17—C21—N2 | −179.1 (3) |
| O7—P2—O10—C8 | 179.0 (3) | C18—C17—C21—C22 | 178.8 (3) |
| O8—P2—O10—C8 | 51.5 (3) | C16—C17—C21—C22 | 0.4 (4) |
| O9—P2—O10—C8 | −57.2 (3) | C11—N1—C22—C14 | 0.6 (4) |
| P2—O8—C6—C9 | 0.3 (5) | C11—N1—C22—C21 | −179.2 (3) |
| P2—O9—C7—C9 | 1.1 (4) | C13—C14—C22—N1 | 0.5 (4) |
| P2—O10—C8—C9 | 2.4 (5) | C15—C14—C22—N1 | 179.8 (3) |
| O10—C8—C9—C7 | 58.6 (4) | C13—C14—C22—C21 | −179.7 (3) |
| O10—C8—C9—C6 | −59.2 (5) | C15—C14—C22—C21 | −0.4 (4) |
| O10—C8—C9—C10 | 177.4 (3) | N2—C21—C22—N1 | −0.7 (4) |
| O9—C7—C9—C8 | −60.6 (4) | C17—C21—C22—N1 | 179.8 (3) |
| O9—C7—C9—C6 | 59.0 (4) | N2—C21—C22—C14 | 179.5 (3) |
| O9—C7—C9—C10 | 179.2 (3) | C17—C21—C22—C14 | 0.0 (4) |
| O8—C6—C9—C8 | 57.7 (5) | | |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O13—H13B···O12$^{i}$ | 0.84 | 2.47 | 2.893 (9) | 112 |
| N1—H1···O1$^{ii}$ | 0.90 | 1.91 | 2.765 (3) | 159 |
| O6—H6C···O11$^{i}$ | 0.85 | 1.61 | 2.459 (5) | 173 |

Symmetry codes: (i) −*x*+1, *y*−1/2, −*z*+3/2; (ii) −*x*+2, −*y*+1, −*z*+2.

**Fig. 1**

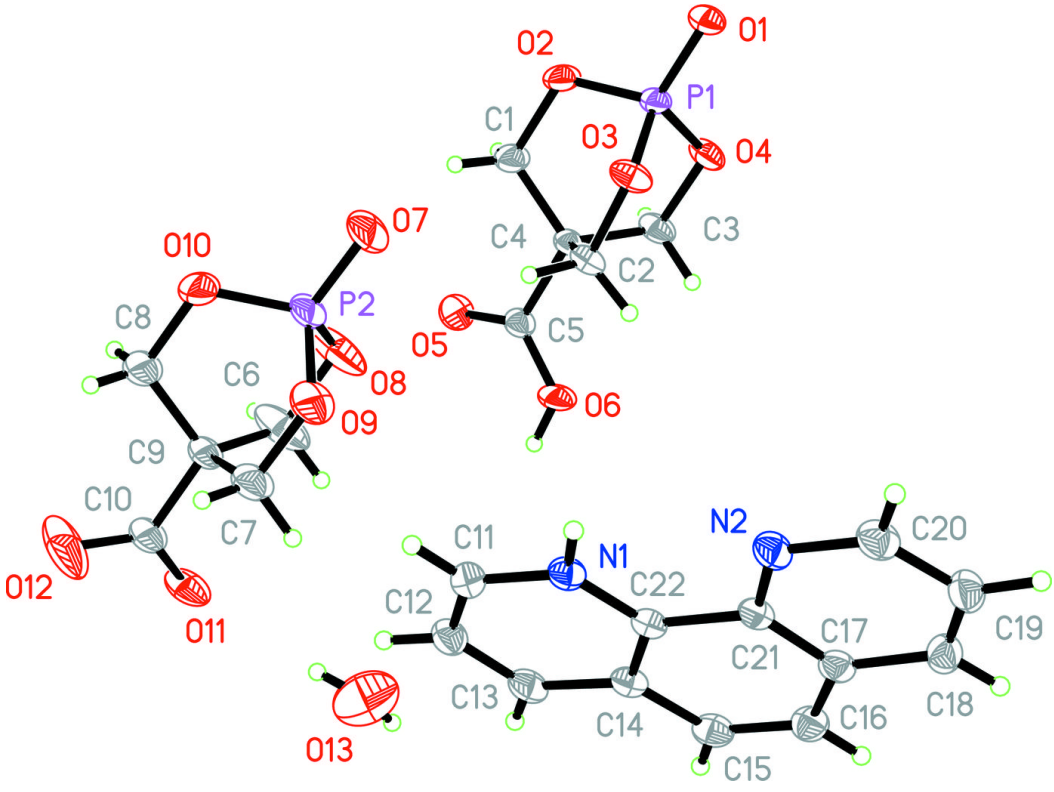
O1
O2
P1
C1
O3
O4
C4
C3
C2
O7
O10
C8
P2
O5
C5
C6
O8
O6
O9
C9
C10
C7
O12
O11
C11
N1
N2
C20
C12
C22
C17
C19
C21
C13
C18
C14
C16
O13
C15

**Fig. 2**