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1-Oxo-*N*-(2-pyridyl)-2,6,7-trioxa-1-phosphabicyclo[2.2.2]octane-4-carboxamide

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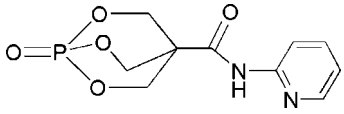
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.004 Å; R factor = 0.039; wR factor = 0.101; data-to-parameter ratio = 13.4.

Inhe title compound, $C_{10}H_{11}N_2O_5P$, intermolecular N—H···O hydrogen bonding links the molecules, forming a zigzag chain running parallel to the [010] direction.

Related literature

For related literature, see: Allen *et al.* (1995); Li *et al.* (2002).



Experimental

Crystal data

$C_{10}H_{11}N_2O_5P$
M_r = 270.18
Monoclinic, $P2_1/n$
a = 5.915 (3) Å
b = 11.960 (6) Å
c = 17.678 (8) Å
β = 90.427 (8)°
V = 1250.5 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 294 (2) K
0.24 × 0.20 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.938, T_{max} = 0.957
6101 measured reflections
2190 independent reflections
1533 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.102
S = 1.02
2190 reflections
163 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.86	2.17	3.020 (3)	172

Symmetry code: (i) $-x+\frac{5}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2280).

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supplementary materials

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1-Oxo-*N*-(2-pyridyl)-2,6,7-trioxa-1-phosphabicyclo[2.2.2]octane-4-carboxamide

G.-F. Chen, J. Hu and G.-C. Ma

Comment

Caged bicyclic phosphates are widely used as flame retardants, resin stabilizers and oil adducts. They have raised great interest and many studies on these compounds have been reported (Allen *et al.*, 1995; Li *et al.*, 2002). We report here the structure of a bicyclic phosphate cage compound, namely *N*-(pyridin-2-yl)-1-oxo-2,6,7-trioxa-1-phosphabicyclo [2.2.2]-octane-4-carboxamide in order to elucidate its molecular conformation.

The asymmetric unit contains one independent molecule in which the trioxa-1-phosphabicyclooctane is linked to the pyridine by a carboxamide chain (Fig. 1). Bond lengths and angles are within normal range.

Intermolecular N—H···O hydrogen bond link the molecule to form a zigzag chain running parallel to the [010] direction (Table 1, Fig. 2).

Experimental

A dry three-necked round-bottom flask was charged with 2-aminopyridine (2 mmol), 1-oxa-1-phosphatrioxabicyclo[2,2,2]-octane-4-chlorocarbinyl (2 mmol) and dichloromethane (20 ml). A dichloromethane solution of triethylamine (2.2 mmol) was added dropwise to the mixture over a period of 0.5 h while stirring at room temperature, then it was refluxed for another 4 h. After removal of the solvent under reduced pressure, the residue was stirred with water, collected by filtration, washed thoroughly with water, and recrystallized from absolute ethanol to give the product as fine white needles. Single crystals were obtained by slow evaporation of a absolute ethanol solution over a period of 20 days at room temperature (yield: 450.0 mg, 83%, m.p. 503–505 K).

Refinement

All H atoms attached to C atoms and N atom were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic) or 0.97 Å (methylene) and N—H = 0.86 Å with U_{iso}(H) = 1.2U_{eq}(C or N).

Figures

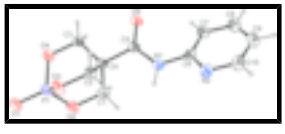

Fig. 1. Molecular view of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level·H atoms are represented as small sphere of arbitrary radii.

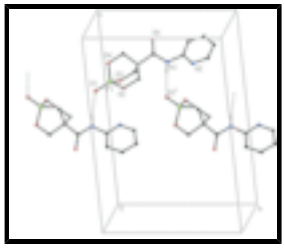

Fig. 2. Partial packing view showing the formation of the zigzag chain. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bondings have been omitted for clarity. [Symmetry code: (i) $5/2 - x$, $1/2 + y$, $3/2 - z$]

1-Oxo-*N*-(2-pyridyl)-2,6,7-trioxa-1-phosphabicyclo[2.2.2]octane-4-carboxamide

Crystal data

$C_{10}H_{11}N_2O_5P$	$F_{000} = 560$
$M_r = 270.18$	$D_x = 1.435$ Mg m^{-3}
Monoclinic, $P2_1/n$	Melting point: 504(1) K
Hall symbol: -P 2yn	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 5.915$ (3) Å	Cell parameters from 1990 reflections
$b = 11.960$ (6) Å	$\theta = 2.9$–25.1°
$c = 17.678$ (8) Å	$\mu = 0.24$ mm^{-1}
$\beta = 90.427$ (8)°	$T = 294$ (2) K
$V = 1250.5$ (10) Å^3	Needle, colorless
$Z = 4$	0.24 × 0.20 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer	2190 independent reflections
Radiation source: fine-focus sealed tube	1533 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.035$
$T = 294$(2) K	$\theta_{max} = 25.0^\circ$
φ and ω scans	$\theta_{min} = 2.1^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -3 \rightarrow 7$
$T_{min} = 0.938$, $T_{max} = 0.957$	$k = -14 \rightarrow 14$
6101 measured reflections	$l = -20 \rightarrow 21$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.039$	H-atom parameters constrained
$wR(F^2) = 0.102$	$w = 1/[\sigma^2(F_o^2) + (0.0426P)^2 + 0.4588P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} < 0.001$
2190 reflections	$\Delta\rho_{max} = 0.21$ e Å^{-3}

163 parameters	$\Delta\rho_{min} = -0.26$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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}
P1	1.17487 (12)	0.19592 (5)	0.80268 (4)	0.0441 (2)
O1	1.2807 (4)	0.09392 (14)	0.77026 (11)	0.0651 (6)
O2	1.0133 (3)	0.26256 (15)	0.74607 (9)	0.0573 (5)
O3	1.3517 (3)	0.28690 (14)	0.83324 (10)	0.0526 (5)
O4	1.0184 (3)	0.17106 (13)	0.87459 (10)	0.0600 (6)
O5	0.7430 (3)	0.47232 (14)	0.95608 (9)	0.0497 (5)
N1	0.9131 (3)	0.58464 (16)	0.86551 (11)	0.0444 (5)
H1	0.9923	0.5819	0.8250	0.053*
N2	0.9907 (4)	0.77507 (18)	0.86938 (12)	0.0536 (6)
C1	0.9069 (4)	0.3651 (2)	0.77778 (12)	0.0404 (6)
H1A	0.7436	0.3589	0.7745	0.048*
H1B	0.9530	0.4300	0.7488	0.048*
C2	1.2475 (4)	0.38963 (19)	0.86565 (13)	0.0374 (6)
H2A	1.2950	0.4546	0.8370	0.045*
H2B	1.2974	0.3992	0.9176	0.045*
C3	0.9103 (4)	0.27196 (18)	0.90771 (13)	0.0389 (6)
H3A	0.9560	0.2795	0.9603	0.047*
H3B	0.7472	0.2639	0.9059	0.047*
C4	0.9817 (4)	0.37935 (18)	0.86264 (11)	0.0319 (5)
C5	0.8658 (4)	0.48371 (19)	0.89979 (12)	0.0349 (5)
C6	0.8439 (4)	0.6941 (2)	0.89033 (12)	0.0397 (6)
C7	0.6406 (5)	0.7139 (2)	0.93044 (14)	0.0478 (7)
H7	0.5436	0.6558	0.9433	0.057*
C8	0.5930 (6)	0.8248 (2)	0.94968 (17)	0.0658 (8)
H8	0.4611	0.8419	0.9756	0.079*
C9	0.7444 (6)	0.9108 (2)	0.92991 (17)	0.0742 (10)
H9	0.7161	0.9847	0.9433	0.089*
C10	0.9379 (6)	0.8820 (2)	0.88971 (17)	0.0639 (8)
H10	1.0366	0.9389	0.8760	0.077*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0488 (4)	0.0409 (4)	0.0429 (4)	−0.0003 (3)	0.0156 (3)	−0.0054 (3)
O1	0.0826 (14)	0.0475 (11)	0.0658 (12)	0.0070 (10)	0.0318 (11)	−0.0084 (9)
O2	0.0677 (12)	0.0643 (12)	0.0397 (9)	0.0073 (11)	−0.0019 (9)	−0.0171 (9)
O3	0.0337 (9)	0.0532 (11)	0.0712 (12)	0.0068 (8)	0.0079 (9)	−0.0149 (9)
O4	0.0803 (14)	0.0377 (10)	0.0627 (12)	0.0021 (10)	0.0353 (11)	0.0008 (8)
O5	0.0502 (10)	0.0512 (10)	0.0480 (10)	0.0028 (9)	0.0241 (9)	0.0011 (8)
N1	0.0511 (13)	0.0429 (12)	0.0393 (11)	0.0051 (10)	0.0182 (10)	0.0021 (9)
N2	0.0591 (15)	0.0451 (13)	0.0566 (14)	−0.0007 (12)	0.0080 (12)	0.0053 (10)
C1	0.0368 (13)	0.0495 (14)	0.0348 (12)	−0.0031 (12)	0.0014 (10)	−0.0018 (11)
C2	0.0305 (12)	0.0400 (13)	0.0418 (13)	−0.0024 (11)	0.0035 (10)	−0.0031 (10)
C3	0.0389 (13)	0.0423 (13)	0.0356 (12)	0.0001 (11)	0.0102 (11)	−0.0003 (10)
C4	0.0265 (11)	0.0394 (12)	0.0299 (11)	−0.0038 (10)	0.0045 (9)	0.0001 (10)
C5	0.0290 (12)	0.0426 (13)	0.0332 (12)	−0.0017 (11)	0.0032 (10)	−0.0003 (10)
C6	0.0463 (15)	0.0413 (13)	0.0315 (12)	0.0075 (12)	−0.0010 (11)	0.0021 (11)
C7	0.0483 (15)	0.0492 (16)	0.0459 (14)	0.0093 (13)	0.0062 (12)	0.0015 (12)
C8	0.074 (2)	0.0610 (19)	0.0628 (18)	0.0213 (17)	0.0173 (16)	−0.0014 (15)
C9	0.111 (3)	0.0441 (17)	0.067 (2)	0.0182 (19)	0.011 (2)	−0.0057 (15)
C10	0.081 (2)	0.0439 (16)	0.0671 (19)	−0.0003 (16)	0.0041 (17)	0.0030 (14)

Geometric parameters (Å, °)

P1—O1	1.4879 (18)	C2—C4	1.577 (3)
P1—O2	1.592 (2)	C2—H2A	0.9700
P1—O3	1.6003 (19)	C2—H2B	0.9700
P1—O4	1.6058 (18)	C3—C4	1.571 (3)
O2—C1	1.490 (3)	C3—H3A	0.9700
O3—C2	1.491 (3)	C3—H3B	0.9700
O4—C3	1.488 (3)	C4—C5	1.571 (3)
O5—C5	1.244 (2)	C6—C7	1.421 (4)
N1—C5	1.380 (3)	C7—C8	1.399 (4)
N1—C6	1.441 (3)	C7—H7	0.9300
N1—H1	0.8600	C8—C9	1.410 (4)
N2—C6	1.354 (3)	C8—H8	0.9300
N2—C10	1.365 (3)	C9—C10	1.395 (4)
C1—C4	1.570 (3)	C9—H9	0.9300
C1—H1A	0.9700	C10—H10	0.9300
C1—H1B	0.9700		
O1—P1—O2	114.87 (11)	O4—C3—H3B	109.6
O1—P1—O3	114.31 (11)	C4—C3—H3B	109.6
O2—P1—O3	105.03 (10)	H3A—C3—H3B	108.1
O1—P1—O4	113.57 (10)	C1—C4—C5	111.41 (18)
O2—P1—O4	104.09 (11)	C1—C4—C3	108.71 (18)
O3—P1—O4	103.75 (10)	C5—C4—C3	108.52 (16)
C1—O2—P1	115.39 (14)	C1—C4—C2	108.34 (17)

C2—O3—P1	114.78 (14)	C5—C4—C2	111.24 (17)
C3—O4—P1	114.41 (13)	C3—C4—C2	108.56 (18)
C5—N1—C6	127.01 (18)	O5—C5—N1	124.6 (2)
C5—N1—H1	116.5	O5—C5—C4	120.5 (2)
C6—N1—H1	116.5	N1—C5—C4	114.89 (18)
C6—N2—C10	116.7 (2)	N2—C6—C7	124.3 (2)
O2—C1—C4	109.39 (18)	N2—C6—N1	112.5 (2)
O2—C1—H1A	109.8	C7—C6—N1	123.2 (2)
C4—C1—H1A	109.8	C8—C7—C6	116.9 (3)
O2—C1—H1B	109.8	C8—C7—H7	121.6
C4—C1—H1B	109.8	C6—C7—H7	121.6
H1A—C1—H1B	108.2	C7—C8—C9	120.2 (3)
O3—C2—C4	109.72 (17)	C7—C8—H8	119.9
O3—C2—H2A	109.7	C9—C8—H8	119.9
C4—C2—H2A	109.7	C10—C9—C8	118.1 (3)
O3—C2—H2B	109.7	C10—C9—H9	120.9
C4—C2—H2B	109.7	C8—C9—H9	120.9
H2A—C2—H2B	108.2	N2—C10—C9	123.8 (3)
O4—C3—C4	110.22 (17)	N2—C10—H10	118.1
O4—C3—H3A	109.6	C9—C10—H10	118.1
C4—C3—H3A	109.6		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.86	2.17	3.020 (3)	172

Symmetry codes: (i) −*x*+5/2, *y*+1/2, −*z*+3/2.

Fig. 1

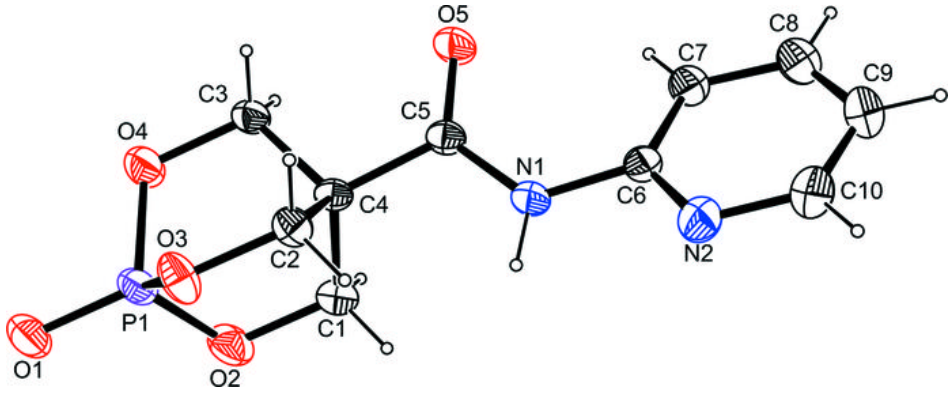

Fig. 2