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N-(Dicyclopropylmethylamino)-4,5-dihydro-1,3-oxazolium dihydrogenphosphate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.030
wR factor = 0.084
Data-to-parameter ratio = 13.0

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

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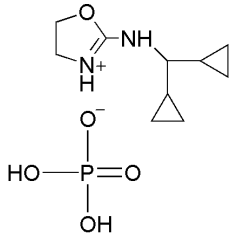
The title compound, $C_{10}H_{17}N_2O^+\cdot H_2PO_4^-$, consists of a rilmenidine cation and a phosphate anion. In the solid state, the inversion-related phosphate anions are linked by O—H···O hydrogen bonds to form a chain along the *a* axis. The cations are connected to the chain *via* N—H···O hydrogen bonds.

Comment

N-(Dicyclopropylmethyl)-4,5-dihydro-2-oxazolamine (or rilmenidine) exhibits good antihypertensive activity (Malen *et al.*, 1978). We report here the structure of rilmenidine phosphate, (I).



(I)

Compound (I) (Fig. 1) contains a rilmenidine cation and a phosphate anion. In the oxazole ring, the N1═C2 [1.3079 (19) Å] distance is longer than the corresponding distance [1.264 (3) Å] reported by Jiang *et al.* (2001); on the other hand, the C2—N6 [1.3055 (19) Å] and C2—O3 [1.3230 (17) Å] distances are shorter [1.367 (2) Å; Jiang *et al.*, 2001]. These changes in bond distance suggest electron delocalization involving atoms N1, C2, O3 and N6. The oxazole ring adopts a half-chair conformation. The geometry of the two cyclopropyl groups agrees with that reported by Gerhard *et al.* (2001). The cyclopropyl substituent often affects the activity of the groups attached to it (Johnson *et al.*, 2000).

In the crystal structure, inversion-related phosphate anions are linked by O—H···O hydrogen bonds to form a chain along the *a* axis (Fig. 2). The cations are connected to the chain *via* N—H···O hydrogen bonds (Table 2). A view of the molecular packing down the *a* axis is shown in Fig. 3.

Experimental

Rilmenidine was synthesized according to the method of Malen *et al.* (1978). An ethanol solution (5 ml) containing rilmenidine (0.018 g, 0.1 mmol) was mixed with an equimolar amount of phosphoric acid and the mixture was placed in a conical flask. After several days, white single crystals were obtained by slow evaporation of ethanol at room temperature.

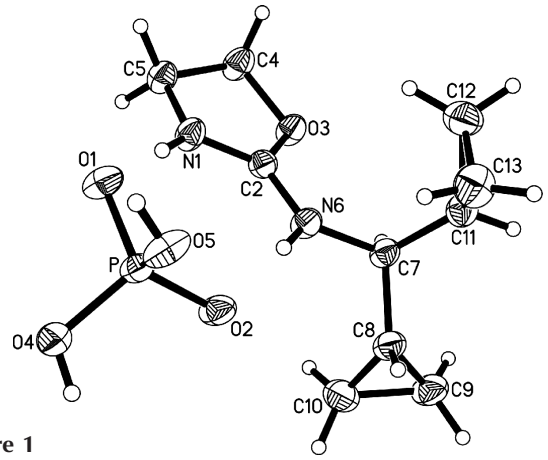


Figure 1
The structure of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. The H atom on C4 is obscured.

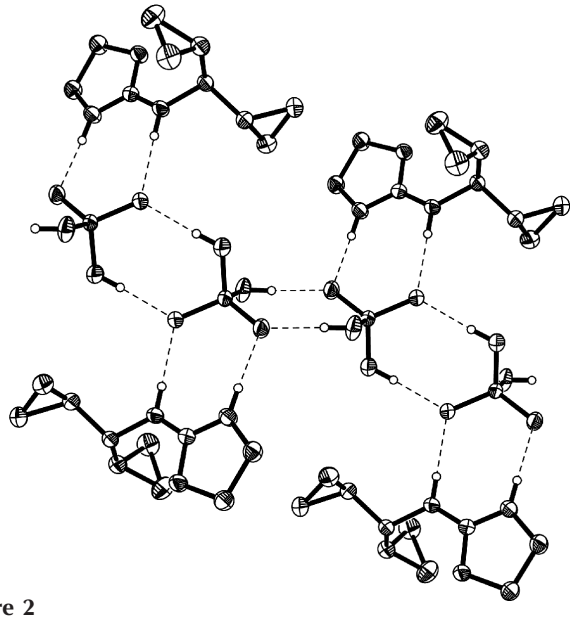


Figure 2
A view of the O—H···O and N—H···O hydrogen-bonded chain. Only H atoms involved in the hydrogen bonds (dashed lines) are shown.

Crystal data

$C_{10}H_{17}N_2O^+\cdot H_2O_4P^-$
M_r = 278.24
Triclinic, $P\overline{1}$
a = 8.3212 (8) Å
b = 8.9609 (9) Å
c = 9.8813 (10) Å
α = 97.070 (2)°
β = 101.475 (2)°
γ = 108.355 (2)°
V = 671.54 (12) Å^3
Z = 2
D_x = 1.376 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2726 reflections
θ = 2.4–28.6°
μ = 0.22 mm^{-1}
T = 293 (2) K
Block, white
0.48 × 0.48 × 0.46 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
3425 measured reflections
2342 independent reflections
2120 reflections with $I > 2\sigma(I)$
R_{int} = 0.011
θ_{max} = 25.1°
$h = -9 \rightarrow 8$
$k = -10 \rightarrow 10$
$l = -11 \rightarrow 11$

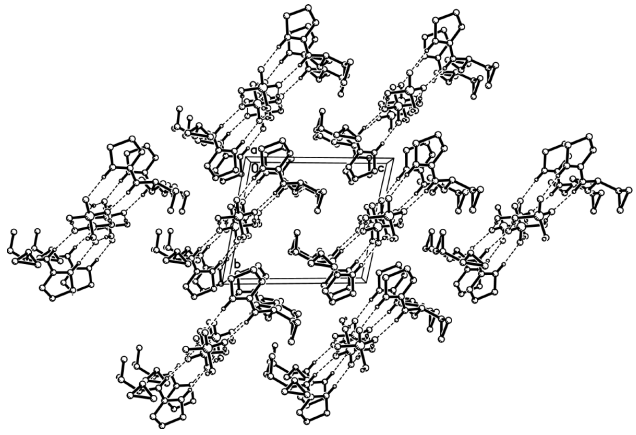


Figure 3
Packing of (I), viewed down the *a* axis. Dashed lines indicate hydrogen bonds.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.084
S = 1.06
2342 reflections
180 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0421P)^2 + 0.1859P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Extinction correction: *SHELXTL97*
Extinction coefficient: 0.041 (4)

Table 1
Selected geometric parameters (Å, °).

P—O1	1.5043 (11)	O3—C4	1.4679 (18)
P—O2	1.5053 (11)	N1—C2	1.3079 (19)
P—O5	1.5599 (13)	N1—C5	1.457 (2)
P—O4	1.5637 (13)	N6—C2	1.3055 (19)
O3—C2	1.3230 (17)	N6—C7	1.4755 (19)
C5—N1—C2—O3	4.99 (19)	C2—N1—C5—C4	−14.60 (18)
C4—O3—C2—N1	7.60 (18)	O3—C4—C5—N1	17.88 (16)
C2—O3—C4—C5	−16.29 (17)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O1	0.85 (2)	1.87 (2)	2.724 (2)	175 (2)
N6—H6N···O2	0.85 (2)	1.92 (2)	2.759 (2)	173 (2)
O4—H4O···O2^{i}	0.81 (1)	1.80 (1)	2.608 (2)	175 (2)
O5—H5O···O1^{ii}	0.82 (2)	1.77 (2)	2.584 (2)	175 (2)

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

H atoms attached to the hydroxy O and amine N atoms were located in a difference Fourier map and refined isotropically; the O4—H4O distance was restrained to 0.81 (1) Å. The remaining H atoms were placed in calculated positions and refined using a riding model, with C—H = 0.97 and 0.98 Å, and U_{iso}(H) = 1.2U_{eq}(carrier atom).

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics:

SHELXTL (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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