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Bis(2,6-diaminopyridinium) oxalate dihydrate

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.002 Å
R factor = 0.032
wR factor = 0.089
Data-to-parameter ratio = 10.3

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

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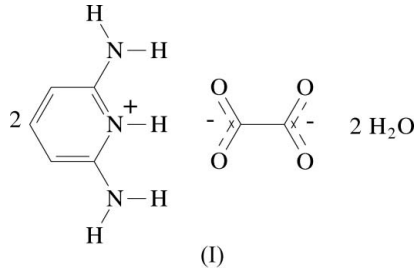
In the title compound, $2C_5H_8N_3^+\cdot C_2O_2^{2-}\cdot 2H_2O$, the 2,6-diaminopyridinium ions are linked to centrosymmetric oxalate ions and water molecules through N—H···O and O—H···O hydrogen bonds, generating edge-fused $[R_2^1(6)R_1^2(5)R_2^2(9)R_4^2(8)R_6^6(17)]$ motifs.

Comment

We have been interested in hydrogen-bonding systems formed by organic amines and carboxylic acids (Büyükgüngör & Odabaşoğlu, 2002, 2003, 2006*a*, 2006*b*; Odabaşoğlu, Büyükgüngör & Lönnecke, 2003; Odabaşoğlu, Büyükgüngör, Turgut *et al.*, 2003; Odabaşoğlu & Büyükgüngör, 2006*a*,*b*). The present work is part of a structural study of compounds of organic ammonium systems with hydrogen-bond donors and we report here the structure of the title compound, (I).



(I)

In (I), the oxalate ion is centrosymmetric (Fig. 1). 2,6-Diaminopyridinium ions are linked to the oxalate ions and water molecules through N—H···O and O—H···O hydrogen bonds (Table 1), forming edge-fused $[R_2^1(6)R_2^2(9)R_1^2(5)]$ motifs

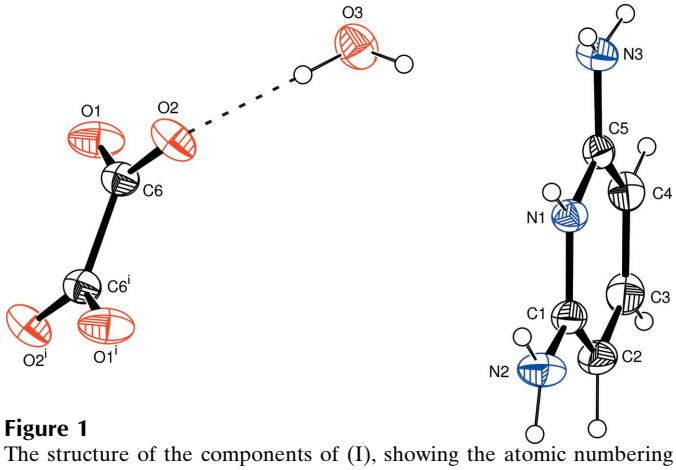


Figure 1
The structure of the components of (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. The dashed line indicates a hydrogen bond. [Symmetry code: (i) $1-x$, $1-y$, $2-z$.]

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(Fig. 2). These motifs are connected by four N—H···O hydrogen bonds, generating $R_6^6(17)$ rings, and water molecules, generating $R_4^2(8)$ hydrogen-bonded rings. The pyridinium rings of the cations are stacked along the *c* axis, and show π–π interactions, the centroid-to-centroid distance being 3.540 (1) Å.

Experimental

The title compound was prepared as described by Büyükgüngör & Odabaşoğlu (2006*b*), using 2,6-diaminopyridine and oxalic acid as starting materials. Equimolar quantities of 2,6-diaminopyridine and oxalic acid were separately dissolved in methanol–water (1:1). The solutions were mixed at 323–333 K and the mixture was left to stand at room temperature, giving crystals suitable for X-ray diffraction by slow evaporation (m.p. 488–490 K, yield 98%).

Crystal data

$2C_5H_8N_3^+\cdot C_2O_4^{2-}\cdot 2H_2O$
M_r = 344.34
Monoclinic, $P2_1/c$
a = 8.0068 (6) Å
b = 15.4788 (8) Å
c = 7.0250 (5) Å
β = 114.531 (6)°
V = 792.06 (9) Å^3
Z = 2
D_x = 1.444 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 296 K
Prism, light yellow
0.65 × 0.44 × 0.28 mm

Data collection

Stoe IPDS-2 diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.936, T_{max} = 0.971
11192 measured reflections
1551 independent reflections
1401 reflections with $I > 2\sigma(I)$
R_{int} = 0.047
θ_{max} = 26.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.089
S = 1.03
1551 reflections
150 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0479P)^2 + 0.1298P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.034 (5)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.882 (15)	1.911 (15)	2.7611 (13)	161.2 (13)
N2—H2A···O2^{i}	0.89 (2)	2.23 (2)	3.0037 (17)	145.2 (16)
N2—H2B···O1^{ii}	0.869 (19)	2.079 (19)	2.9445 (15)	174.0 (16)
N3—H3A···O1^{iii}	0.871 (16)	2.062 (17)	2.9322 (15)	176.7 (14)
N3—H3B···O3^{iv}	0.877 (17)	2.065 (17)	2.9365 (16)	172.3 (15)
O3—H3C···O1^{iii}	0.85 (3)	2.47 (3)	3.1418 (16)	136 (2)
O3—H3C···O2^{i}	0.85 (3)	2.34 (2)	3.1176 (16)	152 (2)
O3—H3D···O2	0.88 (2)	1.98 (2)	2.8493 (15)	168.9 (19)

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

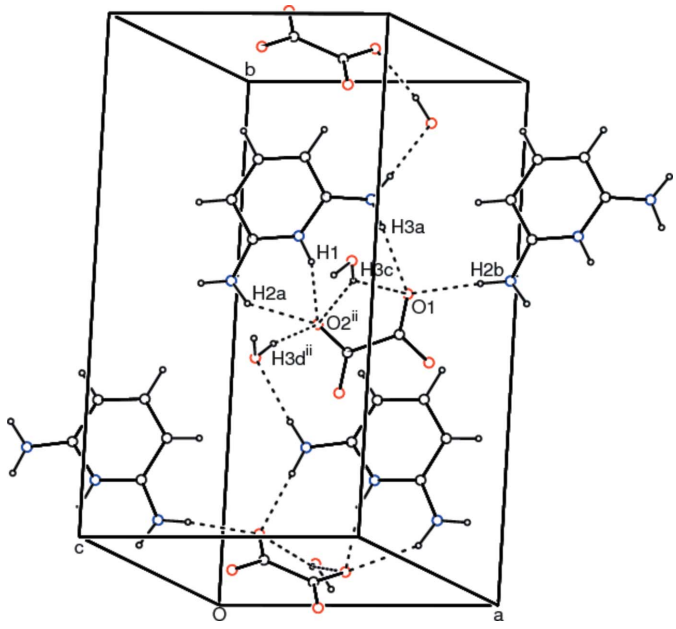

Figure 2
Part of the crystal structure of (I), showing the formation of $R_2^1(6)R_1^2(5)R_2^2(9)$ motifs. Dashed lines indicate hydrogen bonds. [Symmetry code: (ii) $1-x, 1-y, 1-z$.]

All H atoms were located in difference maps and refined freely with an isotropic displacement parameters. The C—H, N—H and O—H bond lengths are 0.95 (2)–0.98 (2), 0.87 (2)–0.89 (2) and 0.85 (3)–0.88 (2) Å, respectively.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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