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1,10-Phenanthrolinium–6-carboxypyridine-2-carboxylate–1,10-phenanthroline–pyridine-2,6-dicarboxylic acid–ethanol–water (1/1/1/1/1/1)

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.060
wR factor = 0.141
Data-to-parameter ratio = 12.3

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

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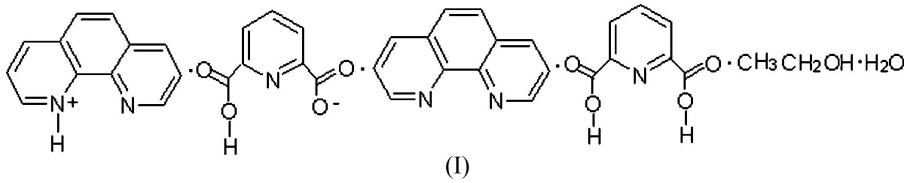
In the title compound, $C_{12}H_9N_2^+\cdot C_7H_5O_4N^-\cdot C_{12}H_8N_2\cdot C_7H_4O_4N\cdot C_2H_6O\cdot H_2O$, the cations are stacked along the *b* axis to form a column-like structure. The anions, ethanol molecules and water molecules are linked *via* O—H···O and O—H···N hydrogen bonds to form a zigzag sheet-like structure. The cationic columns and anionic sheets are alternately arranged along the *c* axis.

Comment

The asymmetric unit of (I), contains a $(C_{12}H_8N_2\cdot C_{12}H_9N_2)^+$ cation, a $(C_7H_5O_4N\cdot C_7H_4O_4N)^-$ anion, a solvent ethanol molecule and a water molecule (Fig. 1). The N3—H3*A*···N5^{i} hydrogen-bonded (see Table 2 for symmetry code) 1,10-phenanthrolinium and 1,10-phenanthroline molecules form a cation, and the O4—H4···O5^{iii} hydrogen-bonded pyridine-2,6-dicarboxylic acid and 6-carboxypyridine-2-carboxylate molecules form an anion. The C—O bond lengths of the carboxylate group [C14—O5 = 1.289 (4) Å and C14—O6 = 1.222 (5) Å] are not equal as the O4—H4···O5^{iii} [O···O = 2.440 (4) Å] hydrogen bond is stronger than the O9—H9···O6^{v} [O···O = 2.768 (5) Å] hydrogen bond. The other bond lengths and angles in the cation are comparable to corresponding values found in its complexes (Fu, Sun *et al.*, 2004; Fu, Wang & Shen, 2004; Fu, Wang, Shen & Zhang, 2004; Fu, Fu & Yu, 2005), and those in the anion of (I) are consistent with the values found in its complexes (Fu, Wang & Liu, 2004; Fu, Wang & Sun, 2005).



(I)

In the crystal structure of (I), the cations are stacked along the *b* axis to form a column-like structure. The anions and ethanol and water molecules are linked *via* O—H···O and O—H···N hydrogen bonds (Table 2) to form a zigzag sheet-like structure. The cationic columns and anionic sheets are alternately arranged along the *c* axis (Fig. 2).

Experimental

Phenanthroline (1 mmol) and dipicolinic acid (1 mmol) were dissolved in a 1:1 alcohol and distilled water solution (20 ml), which was allowed to stand in air. After 14 d, colourless prism-shaped crystals separated. These were collected, washed with water and dried in a vacuum over $CaCl_2$ (yield 41%). Elemental analysis found: C

63.17, H 4.44, N 10.98%; calculated for $C_{40}H_{34}N_6O_{10}$: C 63.32, H 4.51, N 11.08%.

Crystal data

$C_{12}H_9N_2^+\cdot C_7H_4NO_4^-\cdot C_{12}H_8N_2\cdot C_7H_5NO_4\cdot C_2H_6O\cdot H_2O$	D_x = 1.409 Mg m^{-3}
M_r = 758.73	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 1469 reflections
a = 20.802 (7) Å	θ = 2.6–20.2°
b = 7.861 (3) Å	μ = 0.10 mm^{-1}
c = 24.488 (6) Å	T = 293 (2) K
β = 116.72 (2)°	Prism, colourless
V = 3577 (2) Å^3	0.39 × 0.13 × 0.11 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	6314 independent reflections
φ and ω scans	2335 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)	R_{int} = 0.111
T_{min} = 0.961, T_{max} = 0.989	θ_{max} = 25.0°
18072 measured reflections	$h = -24 \rightarrow 20$
	$k = -8 \rightarrow 9$
	$l = -29 \rightarrow 29$

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.060	$w = 1/[\sigma^2(F_o^2) + (0.0316P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.141	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.00	$\Delta\rho_{max}$ = 0.32 e Å^{-3}
6314 reflections	$\Delta\rho_{min}$ = −0.28 e Å^{-3}
512 parameters	

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.196 (5)	O4—C7	1.281 (4)
O2—C1	1.309 (5)	O7—C8	1.321 (5)
O3—C7	1.213 (5)	O8—C8	1.206 (5)
O1—C1—O2	124.2 (4)	O8—C8—O7	120.9 (5)
O1—C1—C2	122.7 (4)	O8—C8—C9	123.1 (5)
O2—C1—C2	113.1 (4)	O7—C8—C9	116.0 (5)
O3—C7—O4	126.5 (5)	O6—C14—O5	125.0 (4)
O3—C7—C6	121.0 (4)	O6—C14—C13	120.7 (4)
O4—C7—C6	112.5 (4)	O5—C14—C13	114.3 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···N5^{i}	0.86	2.08	2.863 (5)	152
O2—H2···O10^{ii}	0.82	1.74	2.549 (4)	170
O4—H4···O5^{iii}	0.82	1.64	2.440 (4)	164
O7—H7···O9^{iv}	0.82	1.93	2.686 (5)	154
O7—H7···N2	0.82	2.17	2.662 (4)	118
O9—H9···O6^{v}	0.82	1.96	2.768 (5)	169
O9—H9···N2^{v}	0.82	2.41	2.850 (5)	114
O10—H1···O1	0.87 (2)	2.02 (2)	2.869 (5)	167 (4)
O10—H6···O3^{vi}	0.84 (2)	2.23 (4)	2.876 (4)	134 (4)
O10—H6···N1^{vi}	0.84 (2)	2.35 (3)	3.041 (5)	140 (4)

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

The water H atoms were located in difference Fourier maps, and were refined with O—H and H···H distance restraints of 0.85 (2) and 1.35 (2) Å, respectively, and with fixed U_{iso} value of 0.08 Å^2. All other H atoms were placed in idealized positions and constrained to ride on their parent atoms, with O—H = 0.82 Å, N—H = 0.86 Å, C—H = 0.93–0.97 Å, and U_{iso}(H) = 1.5U_{eq}(C,O) for methyl and hydroxy H atoms, and 1.2U_{eq}(C,N) for other H atoms.

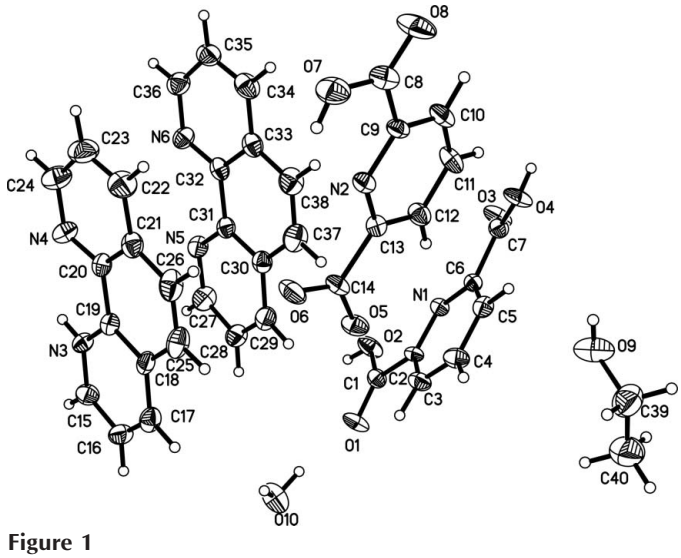


Figure 1
The asymmetric unit of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

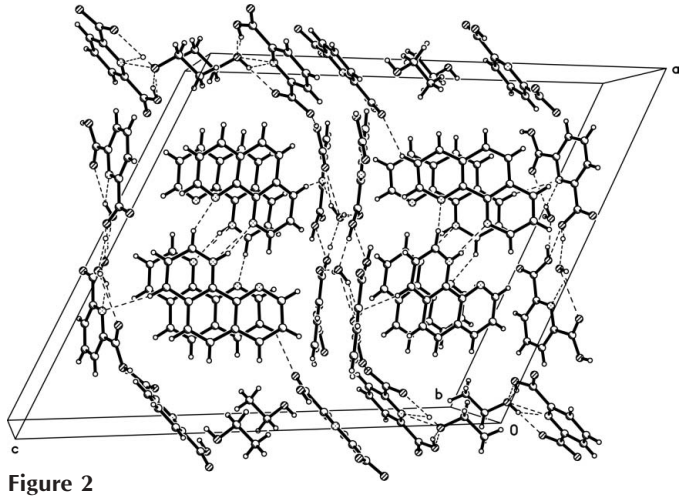


Figure 2
Crystal packing of (I), showing the hydrogen-bonded interactions as dashed lines.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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