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1-Carboxymethyl-3-hydroxypyridinium nitrate–3-hydroxypyridinium-1-acetate (1/1)

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.003 Å
R factor = 0.050
wR factor = 0.181
Data-to-parameter ratio = 13.1

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

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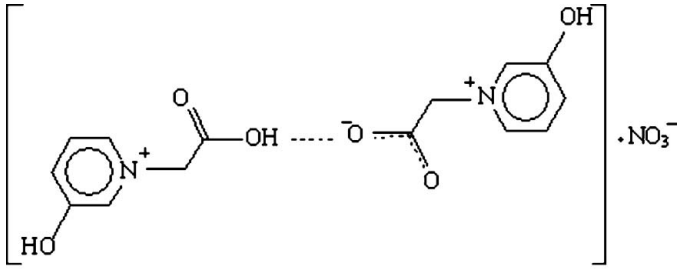
In the crystal structure of the title 1:1 co-crystal, $C_7H_8NO_3^+\cdot NO_3^-\cdot C_7H_7NO_3$, two cations interact with two zwitterions to form a hydrogen-bonded ring, the nitrate anions being located outside of the ring.

Comment

Zwitterionic pyridinioacetate (Szafran *et al.*, 1998) is a betaine that affords a plethora of metal carboxylate complexes (Cambridge Structural Database, Version 5.25; Allen, 2002). With a hydroxy substituent at the 3-position of the pyridine ring, the resulting compound exists in either a zwitterionic or an uncharged configuration (Zhao *et al.*, 2004). We changed experimental conditions to yield a new compound, (I), whose crystal structure is reported here.



(I)

One of the O atoms of the delocalized carboxyl group ($-CO_2^-$) of the 3-hydroxypyridinium-1-acetate zwitterion is linked to the carboxylic acid group of the adjacent cation through a short hydrogen bond [O4···O7 = 2.482 (2) Å] (Fig. 1). This is shorter than that reported in 1-carboxymethyl-3-hydroxypyridinium chloride–3-hydroxypyridinium-1-acetate (1/1) [2.519 (2) Å; Zhao *et al.*, 2004]. The other O atom of the zwitterion forms a hydrogen bond to the 3-hydroxy group of an adjacent cation, giving rise to a cation ring motif with the nitrate anions outside the ring and linked through hydrogen bonds (Fig. 2).

Experimental

An aqueous solution of 3-hydroxypyridine (9.55 g, 0.10 mol) and sodium hydroxide (4.00 g, 0.10 mol) was reacted with an aqueous solution of chloroacetic acid (14.18 g, 0.10 mol) that had been neutralized with sodium hydroxide (6.00 g, 0.15 mol). The pH of the mixture was approximately 9–10. The mixture was refluxed for 5 h and the cooled solution was treated with 0.05 *M* nitric acid to a pH of 2–3. The solution was then filtered and colourless crystals of (I) were obtained after several days. Analysis calculated for $C_{14}H_{15}N_3O_9$: C 45.54, H 4.09, N 11.38%; found: C 45.66, H 4.19, N 11.26%.

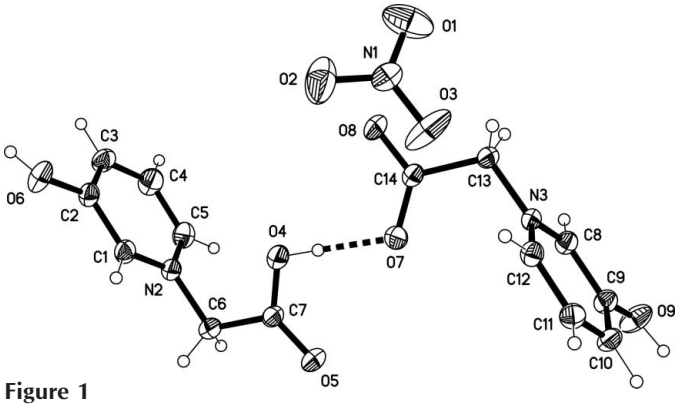


Figure 1
The title compound, showing the atom-numbering scheme, with displacement ellipsoids drawn at the 50% probability level; the O—H···O hydrogen bond is shown as a dashed line.

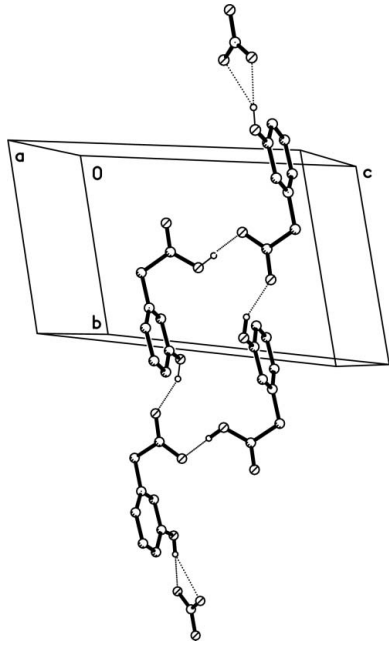


Figure 2
A plot of the layer structure of (I), with hydrogen bonds shown as dashed lines.

Crystal data

$C_7H_8NO_3^+\cdot NO_3^-\cdot C_7H_7NO_3$	$Z = 2$
$M_r = 369.29$	$D_x = 1.562$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 7.0054$ (14) Å	Cell parameters from 5868 reflections
$b = 8.2661$ (17) Å	$\theta = 3.0$–$26.4°$
$c = 14.445$ (3) Å	$\mu = 0.13$ mm^{-1}
$\alpha = 74.50$ (3)°	$T = 295$ (2) K
$\beta = 77.09$ (3)°	Prism, colourless
$\gamma = 88.37$ (3)°	0.39 × 0.26 × 0.18 mm
$V = 785.2$ (3) Å^3	

Data collection

Rigaku R-AXIS RAPID diffractometer	3206 independent reflections
ω scans	2417 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)	$R_{int} = 0.020$
$T_{min} = 0.959$, $T_{max} = 0.976$	$\theta_{max} = 26.5°$
6964 measured reflections	$h = -8 \rightarrow 8$
	$k = -10 \rightarrow 10$
	$l = -18 \rightarrow 18$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1204P)^2 + 0.0897P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.050$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.181$	$\Delta\rho_{max} = 0.44$ e Å^{-3}
$S = 1.06$	$\Delta\rho_{min} = -0.48$ e Å^{-3}
3206 reflections	
244 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1
Selected geometric parameters (Å, °).

N2—C1	1.336 (3)	O8—C14	1.228 (2)
N2—C5	1.346 (3)	O9—C9	1.340 (3)
N2—C6	1.477 (2)	C1—C2	1.384 (3)
N3—C8	1.335 (2)	C2—C3	1.380 (3)
N3—C12	1.346 (2)	C3—C4	1.377 (3)
N3—C13	1.477 (2)	C4—C5	1.373 (3)
O1—N1	1.218 (3)	C6—C7	1.511 (3)
O2—N1	1.229 (3)	C8—C9	1.390 (3)
O3—N1	1.221 (2)	C9—C10	1.387 (3)
O4—C7	1.293 (3)	C10—C11	1.374 (3)
O5—C7	1.206 (2)	C11—C12	1.370 (3)
O6—C2	1.338 (2)	C13—C14	1.519 (3)
O7—C14	1.266 (2)		
N2—C1—C2	120.53 (18)	O8—C14—C13	115.92 (16)
N2—C5—C4	119.3 (2)	O9—C9—C10	124.90 (17)
N2—C6—C7	113.26 (16)	O9—C9—C8	116.67 (17)
N3—C8—C9	120.67 (17)	C1—N2—C5	121.77 (17)
N3—C12—C11	119.52 (17)	C1—N2—C6	119.24 (17)
N3—C13—C14	112.42 (14)	C3—C2—C1	118.67 (19)
O1—N1—O3	122.9 (2)	C4—C3—C2	119.48 (18)
O1—N1—O2	119.0 (2)	C5—N2—C6	118.94 (18)
O3—N1—O2	118.2 (2)	C5—C4—C3	120.3 (2)
O4—C7—C6	114.23 (16)	C8—N3—C12	121.56 (15)
O5—C7—O4	126.4 (2)	C8—N3—C13	118.63 (15)
O5—C7—C6	119.37 (18)	C12—N3—C13	119.76 (16)
O6—C2—C3	123.78 (18)	C10—C9—C8	118.43 (18)
O6—C2—C1	117.55 (17)	C11—C10—C9	119.24 (17)
O7—C14—C13	117.32 (15)	C12—C11—C10	120.58 (17)
O8—C14—O7	126.76 (18)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H15···O7	0.86 (2)	1.62 (2)	2.482 (2)	178 (3)
O6—H16···O8^{i}	0.85 (2)	1.83 (2)	2.619 (2)	154 (3)
O9—H17···O1^{ii}	0.85 (3)	1.96 (3)	2.772 (3)	159 (3)
O9—H17···O2^{ii}	0.85 (3)	2.44 (2)	3.155 (3)	142 (3)

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

The acid and hydroxy H atoms were located and refined isotropically, with the O—H distance restrained to 0.85 (1) Å. All other H atoms were placed in calculated positions, with aromatic C—H = 0.93 Å and aliphatic C—H = 0.97 Å, and were included in the refinement in the riding-model approximation, with U_{iso}(H) = $1.5U_{eq}$(C).

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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