Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Ammonium 6-carboxypyridine-2-carboxylate

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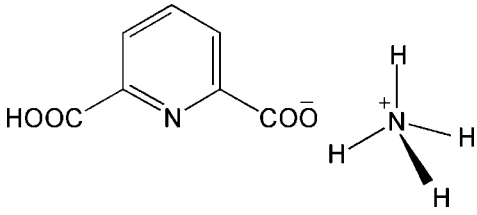
Received 8 October 2007; accepted 23 October 2007

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.001 Å; R factor = 0.034; wR factor = 0.101; data-to-parameter ratio = 18.7.

In the title compound, $NH_4^+\cdot C_7H_4NO_4^-$, the cations and anions are situated on twofold rotation axes. The carboxyl and carboxylate groups are thus disordered; this is consistent with the short and strong O—H···O hydrogen bond across an inversion centre between these groups in neighbouring anions. Ion pairing, and intermolecular O—H···O and N—H···O hydrogen bonds contribute to the crystal packing stability.

Related literature

For related crystal structures, see: Teunissen *et al.* (1993); Lazzarotto *et al.* (2005); Casanova *et al.* (2006). For crystal structures of similar compounds with the same anionic fragment synthesized by our group, see: Aghabozorg *et al.* (2005); Aghabozorg, Ghadermazi & Attar Gharamaleki (2006); Aghabozorg, Ghadermazi, Manteghi & Nakhjavan (2006); Aghabozorg, Ghadermazi & Ramezanipour (2006); Sheshmani *et al.* (2006).



Experimental

Crystal data

$H_4N^+\cdot C_7H_4NO_4^-$
M_r = 184.15
Monoclinic, $C2/c$
a = 10.7033 (11) Å
b = 12.0722 (12) Å
c = 7.2121 (7) Å
β = 125.651 (2)°
V = 757.24 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 100 (2) K
0.50 × 0.50 × 0.50 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*APEX2*; Bruker, 2005)
T_{min} = 0.936, T_{max} = 0.936
3303 measured reflections
1158 independent reflections
1081 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.102
S = 1.05
1158 reflections
62 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H1···O2^{i}	0.84	1.64	2.434 (1)	158
N2—H2···O1^{ii}	0.90	2.07	2.897 (1)	153
N2—H3···O1	0.90	2.06	2.930 (1)	163

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The authors acknowledge financial support from the Teacher Training University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2318).

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 © 2007 International Union of Crystallography doi:10.1107/S1600536807052610

supplementary materials

Acta Cryst. (2007). E**63**, o4454 [**doi:10.1107/S1600536807052610**]

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Comment

There are some instances of ion pairing between ammonium ion and a counter ion, such as a proton transfer compound containing ammonium cation and an anionic complex $[Co(CO)_4]^-$ (Casanova *et al.*, 2006), supramolecular complexes of *p-tert*-butylcalix[6]arene and ammonium cations (Lazzarotto *et al.*, 2005). Another example is proton transfer from acidic zeolites to NH_3 and the interaction of NH_4^+ cation with the zeolite lattice (Teunissen *et al.*, 1993).

In continuation of our study of proton transfer compounds containing pyridine-2,6-dicarboxylate ion (Aghabozorg *et al.*, 2005; Aghabozorg, Ghadermazi & Attar Gharamaleki, 2006; Aghabozorg, Ghadermazi, Manteghi & Nakhjavan, 2006; Aghabozorg, Ghadermazi & Ramezanipour, 2006; Sheshmani *et al.*, 2006), we present here the crystal structure of the title compound (I).

In (I), the cations and anions are situated on twofold rotational symmetry axes (Fig. 1), that causes the disorder of the acidic H atom. Various types of intermolecular hydrogen bonds (Table 1) are observed in (I), which form the layered supramolecular structure (Fig. 2).

Experimental

A 150 ml solution of 4 mmol (668 mg) pyridine-2,6-dicarboxylic acid and a 20 ml solution of 4 mmol (560 mg) hexamethylenetetramine in THF were mixed. The resulting white precipitate with 90% yield was recrystallized in water to shiny colourless cubic crystals (m.p.: 330°C) after four weeks.

Refinement

All H atoms were found in Fourier difference map, placed in idealized postions (C—H 0.93 Å, N—H 0.90 Å, O—H 0.84 Å) and refined as riding, with U_{eq}(H) = 1.2Ueq of the parent atom. For disordered by symmetry atom H2, the occupancy was fixed to 0.5.

Figures

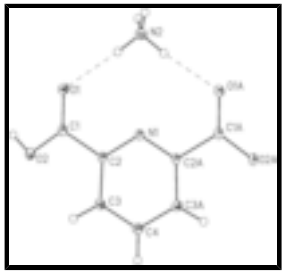

Fig. 1. View of (I) showing the atomic numbering and 50% probability displacement ellipsoids [symmetry code: (A) 2 - *x, y*, 1/2 − *z*]. In the anion, only one position of teh disordered atom H2 is shown. Dashed lines indicate hydrogen bonds.

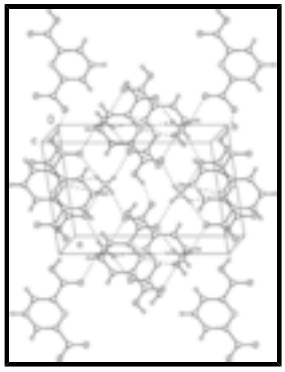

Fig. 2. A portion of the crystal packing viewed along *c* axis. Hydrogen bonds are shown with dashed lines. In the anions, only one position of the disordered hydroxyl H-atom is shown.

Ammonium 6-carboxypyridine-2-carboxylate

Crystal data

$H_4N^+ \cdot C_7H_4NO_4^-$	F_{000} = 384
M_r = 184.15	D_x = 1.615 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 1992 reflections
a = 10.7033 (11) Å	θ = 2.9–34.8º
b = 12.0722 (12) Å	μ = 0.14 mm^{-1}
c = 7.2121 (7) Å	*T* = 100 (2) K
β = 125.651 (2)º	Cube, colourless
V = 757.24 (13) Å^3	0.50 × 0.50 × 0.50 mm
Z = 4	

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	1158 independent reflections
Radiation source: fine-focus sealed tube	1081 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.020
T = 100(2) K	θ_{max} = 30.5º
φ and ω scans	θ_{min} = 2.9º
Absorption correction: multi-scan (APEX2; Bruker, 2005)	*h* = −15→14
T_{min} = 0.936, T_{max} = 0.936	*k* = −16→17
3303 measured reflections	*l* = −10→10

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.034	$w = 1/[\sigma^2(F_o^2) + (0.0629P)^2 + 0.3876P]$ where $P = (F_o^2 + 2F_c^2)/3$

$wR(F^2) = 0.102$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.05$	$\Delta\rho_{max} = 0.55$ e Å^{-3}
1158 reflections	$\Delta\rho_{min} = -0.22$ e Å^{-3}
62 parameters	Extinction correction: SHELXTL (Sheldrick, 1998), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.016 (4)
Secondary atom site location: difference Fourier map	

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}	Occ. (<1)
O1	0.73809 (7)	0.61754 (5)	0.13711 (11)	0.01439 (18)	
O2	0.62331 (7)	0.45370 (5)	0.08777 (12)	0.01787 (19)	
H1	0.5518	0.5000	0.0356	0.027*	0.50
N1	1.0000	0.50758 (8)	0.2500	0.0100 (2)	
C1	0.73808 (9)	0.51498 (7)	0.14117 (13)	0.01132 (19)	
C2	0.87885 (8)	0.44959 (6)	0.20657 (12)	0.00989 (19)	
C3	0.87534 (9)	0.33428 (7)	0.21115 (14)	0.01224 (19)	
H3A	0.7893	0.2967	0.1881	0.015*	
C4	1.0000	0.27524 (9)	0.2500	0.0135 (2)	
H4A	1.0000	0.1966	0.2500	0.016*	
N2	1.0000	0.75422 (8)	0.2500	0.0144 (2)	
H2	0.9523	0.7984	0.1263	0.017*	
H3	0.9317	0.7089	0.2460	0.017*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0108 (3)	0.0103 (3)	0.0208 (3)	0.00095 (19)	0.0085 (3)	−0.0002 (2)
O2	0.0092 (3)	0.0123 (3)	0.0316 (4)	−0.0001 (2)	0.0115 (3)	0.0008 (2)
N1	0.0080 (4)	0.0092 (4)	0.0121 (4)	0.000	0.0054 (4)	0.000
C1	0.0087 (4)	0.0116 (4)	0.0136 (4)	0.0003 (2)	0.0064 (3)	0.0001 (2)
C2	0.0076 (3)	0.0095 (4)	0.0117 (4)	0.0001 (2)	0.0052 (3)	0.0000 (2)
C3	0.0094 (3)	0.0102 (4)	0.0156 (4)	−0.0011 (2)	0.0064 (3)	0.0001 (2)
C4	0.0112 (5)	0.0087 (4)	0.0180 (5)	0.000	0.0070 (4)	0.000

N2	0.0119 (4)	0.0107 (4)	0.0183 (5)	0.000	0.0076 (4)	0.000

Geometric parameters (Å, °)

O1—C1	1.2385 (10)	C3—C4	1.3896 (9)
O2—C1	1.2857 (9)	C3—H3A	0.9500
O2—H1	0.8389	C4—C3^{i}	1.3896 (9)
N1—C2	1.3402 (9)	C4—H4A	0.9500
N1—C2^{i}	1.3402 (9)	N2—H2	0.8999
C1—C2	1.5125 (11)	N2—H3	0.9000
C2—C3	1.3934 (11)		
C1—O2—H1	102.4	C4—C3—C2	118.76 (7)
C2—N1—C2^{i}	117.01 (9)	C4—C3—H3A	120.6
O1—C1—O2	125.55 (7)	C2—C3—H3A	120.6
O1—C1—C2	121.02 (7)	C3^{i}—C4—C3	118.29 (10)
O2—C1—C2	113.41 (7)	C3^{i}—C4—H4A	120.9
N1—C2—C3	123.51 (7)	C3—C4—H4A	120.9
N1—C2—C1	116.90 (7)	H2—N2—H3	110.3
C3—C2—C1	119.54 (7)		
C2^{i}—N1—C2—C3	1.91 (5)	O2—C1—C2—C3	−2.04 (10)
C2^{i}—N1—C2—C1	−175.78 (7)	N1—C2—C3—C4	−3.76 (11)
O1—C1—C2—N1	−2.72 (10)	C1—C2—C3—C4	173.87 (6)
O2—C1—C2—N1	175.75 (6)	C2—C3—C4—C3^{i}	1.74 (5)
O1—C1—C2—C3	179.50 (7)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H1···O2^{ii}	0.84	1.64	2.434 (1)	158
N2—H2···O1^{iii}	0.90	2.07	2.897 (1)	153
N2—H3···O1	0.90	2.06	2.930 (1)	163
N2—H3···N1	0.90	2.53	2.977 (1)	111

Symmetry codes: (ii) −*x*+1, −*y*+1, −*z*; (iii) −*x*+3/2, −*y*+3/2, −*z*.

Fig. 1

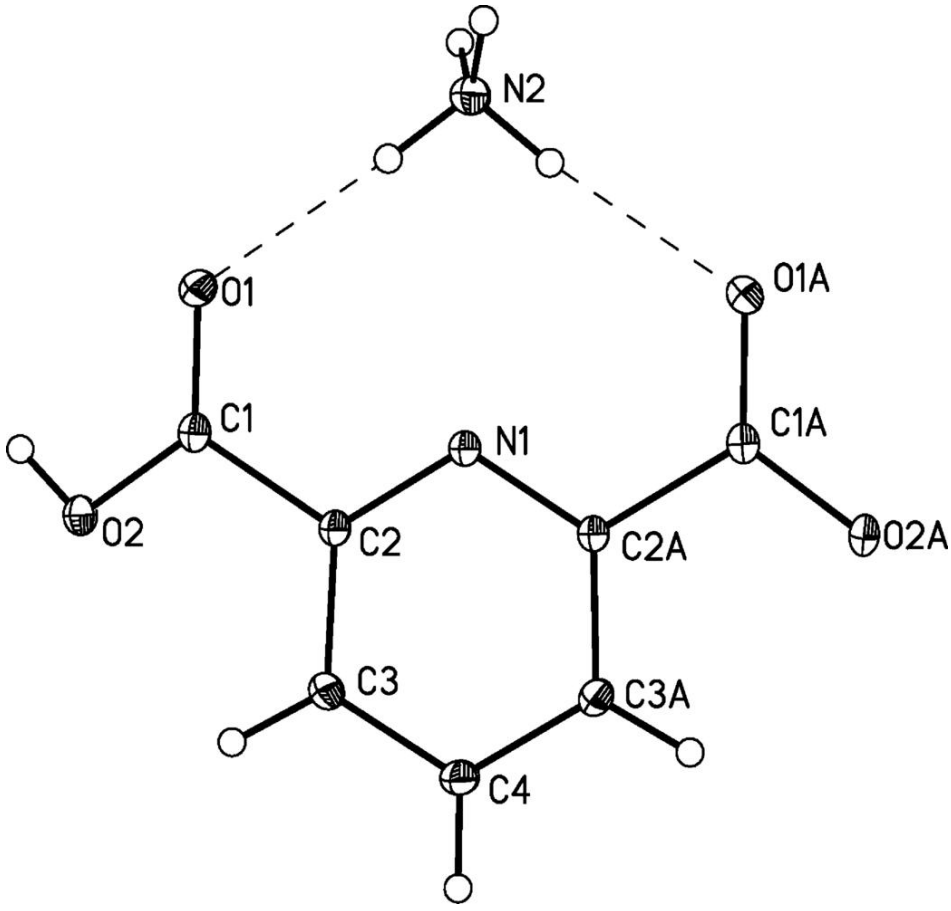
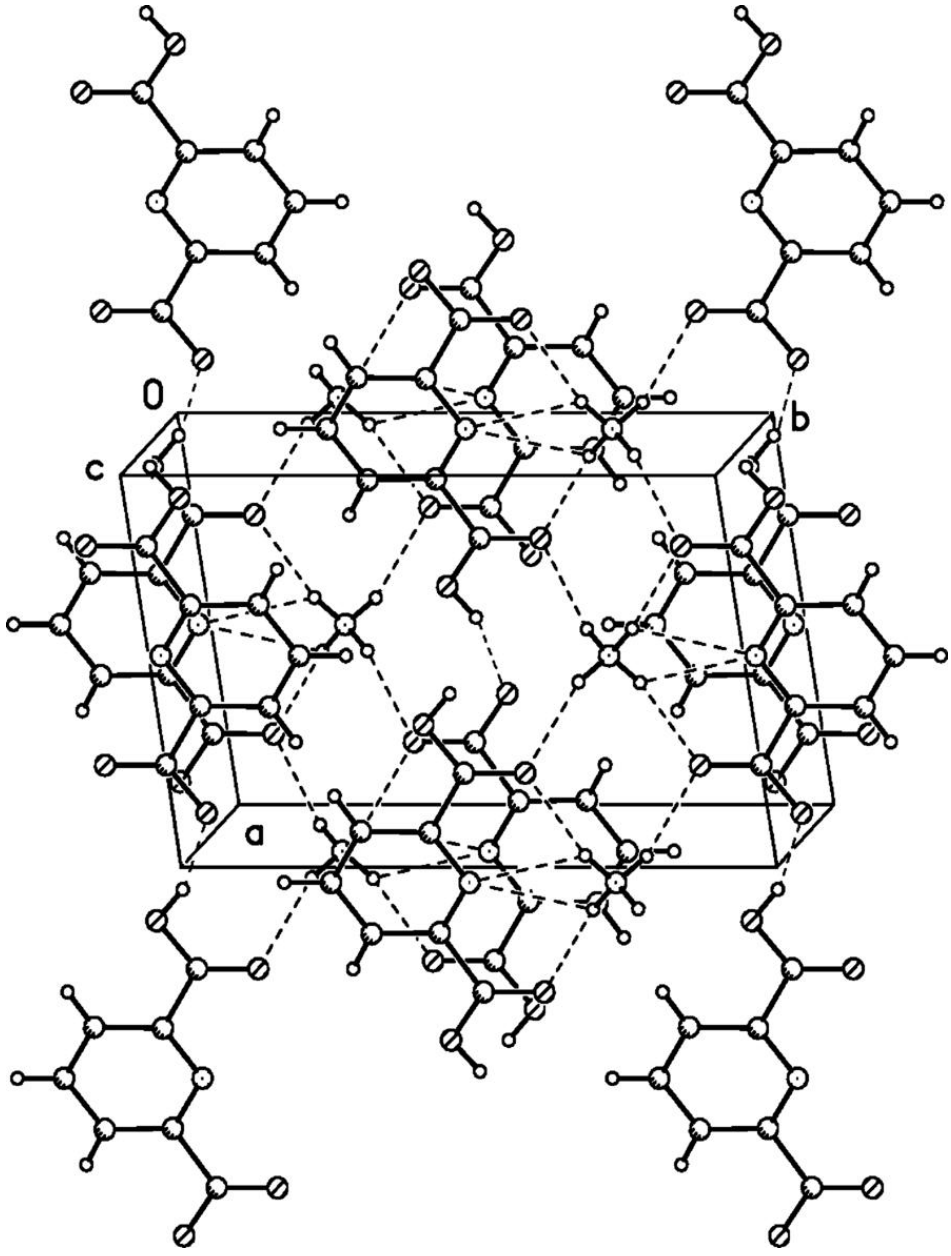
Fig. 2