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4-(3-Carboxyphenyl)pyridinium nitrate

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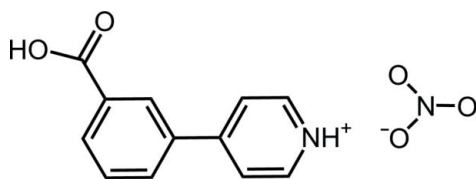
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Key indicators: single-crystal X-ray study; $T = 293\text{ K}$; mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$; R factor = 0.050; wR factor = 0.127; data-to-parameter ratio = 13.0.

In the title salt, $\text{C}_{12}\text{H}_{10}\text{NO}_2^+\cdot\text{NO}_3^-$, the dihedral angle between the pyridine ring and the benzene ring of the 4-(3-carboxyphenyl)pyridinium cation is $30.14(2)^\circ$. Inversion-related pairs of cations are linked into dimers by pairs of $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds. Pairs of dimers are linked by $\text{N}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds involving nitrate anions as acceptors, generating supramolecular chains along the diagonal of the bc plane.

Related literature

For structures of similar compounds, see: Jin *et al.* (2003); Bei *et al.* (2004); Rademeyer (2005); Wang (2006); Yu *et al.* (2006).



Experimental

Crystal data

 $\text{C}_{12}\text{H}_{10}\text{NO}_2^+\cdot\text{NO}_3^-$ $M_r = 262.22$ Triclinic, $P\bar{1}$ $a = 5.2545(11)\text{ \AA}$ $b = 7.0617(14)\text{ \AA}$ $c = 16.469(3)\text{ \AA}$ $\alpha = 97.39(3)^\circ$ $\beta = 92.96(5)^\circ$ $\gamma = 106.05(3)^\circ$ $V = 580.0(2)\text{ \AA}^3$ $Z = 2$ Mo $K\alpha$ radiation $\mu = 0.12\text{ mm}^{-1}$ $T = 293\text{ K}$ $0.15 \times 0.13 \times 0.10\text{ mm}$

Data collection

Bruker SMART diffractometer

Absorption correction: multi-scan

(SADABS; Sheldrick, 1996)

 $T_{\min} = 0.982$, $T_{\max} = 0.988$

4634 measured reflections

2255 independent reflections

1599 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.042$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.050$ $wR(F^2) = 0.127$ $S = 1.00$

2255 reflections

174 parameters

H-atom parameters constrained

 $\Delta\rho_{\max} = 0.20\text{ e \AA}^{-3}$ $\Delta\rho_{\min} = -0.22\text{ e \AA}^{-3}$

Table 1

Hydrogen-bond geometry ($\text{\AA}$, $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C10}-\text{H10}\cdots\text{O4}^{\text{i}}$	0.93	2.42	3.225 (3)	145
$\text{C11}-\text{H11}\cdots\text{O5}^{\text{ii}}$	0.93	2.49	3.122 (3)	125
$\text{O1}-\text{H1}\cdots\text{O2}^{\text{iii}}$	0.82	1.82	2.624 (2)	167
$\text{N2}-\text{H2}\cdots\text{O4}^{\text{ii}}$	0.86	1.89	2.748 (3)	174

Symmetry codes: (i) $x+1, y+1, z$; (ii) $-x+1, -y+1, -z$; (iii) $-x, -y+3, -z+1$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2401).

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supporting information

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4-(3-Carboxyphenyl)pyridinium nitrate

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S1. Comment

The title compound consists of a 3-(pyridin-4-yl)benzoic acid cation and a nitrate anion (Fig. 1). The nitric acid is deprotonated and the pyridine ring accepts the proton to produce the protonated organic cation, namely 3-(pyridin-4-yl)benzoic acid nitrate. The dihedral angle between pyridine ring and the benzene ring of the 3-(pyridin-4-yl)benzoic acid is 30.14 (2)°. The two components are linked by O—H $\cdots$ O [O1—H1 $\cdots$ O2, 2.624 (2) Å] hydrogen bonds to form the cation dimers (Fig. 2), and further through N—H $\cdots$ O and C—H $\cdots$ O [N2—H2 $\cdots$ O4, 2.748 (3) Å; C10—H10 $\cdots$ O4, 3.225 (3) Å; C11—H11 $\cdots$ O5, 3.122 (3) Å] hydrogen bonds. The cation dimers are thus connected by the nitrate anions into extended one-dimensional supramolecular chains (Fig. 2).

S2. Experimental

The title compound was prepared by a hydrothermal method. An aqueous solution (20 mL) containing 3-(pyridin-4-yl)benzoic acid (0.10 mmol) and samarium nitrate hexahydrate (0.10 mmol) was placed in a Parr Teflon-lined stainless steel vessel (25 mL) under autogenous pressure, and then heated to 433 K for 72 h and subsequently cooled to room temperature at a rate of 5 K an hour. The targeted Sm³⁺ complex was not synthesized. Unintentionally, colorless single crystals of the title compound suitable for X-ray analysis were obtained from the reaction mixture.

S3. Refinement

All H atoms were positioned geometrically (C—H = 0.93 Å, O—H = 0.82 Å and N—H = 0.86 Å) and allowed to ride on their parent atoms, with $U_{\text{iso}}(\text{H})$ values equal to 1.2 $U_{\text{eq}}(\text{C}, \text{N})$ or 1.5 $U_{\text{eq}}(\text{O})$.

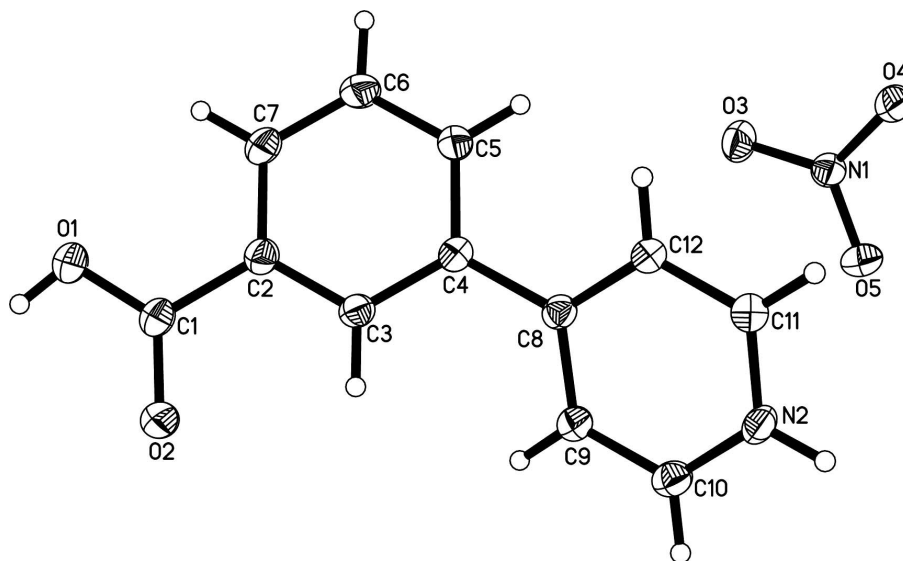


Figure 1

Thermal ellipsoid plot of the title compound (30% probability level), hydrogen atoms are drawn as spheres of arbitrary radius.

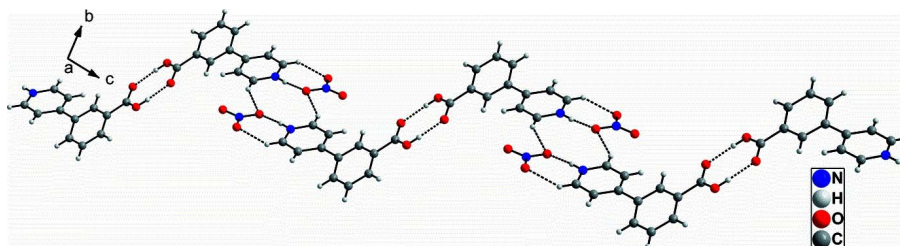


Figure 2

The one-dimensional hydrogen-bonded supramolecular chains in the crystal structure. Dashed lines denote hydrogen bonds.

4-(3-Carboxyphenyl)pyridinium nitrate

Crystal data

$C_{12}H_{10}NO_2^+ \cdot NO_3^-$

$M_r = 262.22$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 5.2545 (11) \text{ \AA}$

$b = 7.0617 (14) \text{ \AA}$

$c = 16.469 (3) \text{ \AA}$

$\alpha = 97.39 (3)^\circ$

$\beta = 92.96 (5)^\circ$

$\gamma = 106.05 (3)^\circ$

$V = 580.0 (2) \text{ \AA}^3$

$Z = 2$

$F(000) = 272$

$D_x = 1.502 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 2373 reflections

$\theta = 3.0\text{--}27.5^\circ$

$\mu = 0.12 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Block, colourless

$0.15 \times 0.13 \times 0.10 \text{ mm}$

Data collection

Bruker SMART
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.982$, $T_{\max} = 0.988$

4634 measured reflections
 2255 independent reflections
 1599 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.042$
 $\theta_{\max} = 26.0^\circ$, $\theta_{\min} = 3.0^\circ$
 $h = -4 \rightarrow 6$
 $k = -8 \rightarrow 8$
 $l = -20 \rightarrow 17$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.127$
 $S = 1.00$
 2255 reflections
 174 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods
 Secondary atom site location: difference Fourier
 map

Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.063P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.20 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.22 \text{ e } \text{\AA}^{-3}$
 Extinction correction: SHELXL97 (Sheldrick,
 2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.039 (7)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\sigma(F^2)$ 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 ($\text{\AA}^2$)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N2	0.5337 (3)	0.8423 (2)	0.07114 (11)	0.0357 (4)
H2	0.6254	0.8387	0.0295	0.043*
O2	0.1639 (3)	1.3909 (2)	0.43108 (10)	0.0541 (5)
O1	-0.2183 (3)	1.2586 (2)	0.48279 (11)	0.0555 (5)
H1	-0.1765	1.3685	0.5113	0.083*
C4	0.0785 (4)	0.8582 (3)	0.27490 (12)	0.0316 (5)
C8	0.2414 (4)	0.8503 (3)	0.20449 (13)	0.0306 (5)
C12	0.1417 (4)	0.7172 (3)	0.13261 (13)	0.0352 (5)
H12	-0.0275	0.6284	0.1292	0.042*
C10	0.6380 (4)	0.9746 (3)	0.13859 (13)	0.0370 (5)
H10	0.8073	1.0623	0.1400	0.044*
C9	0.4953 (4)	0.9804 (3)	0.20526 (13)	0.0341 (5)
H9	0.5685	1.0726	0.2519	0.041*
C3	0.1074 (4)	1.0373 (3)	0.32576 (12)	0.0332 (5)
H3	0.2408	1.1501	0.3185	0.040*

C1	−0.0351 (4)	1.2460 (3)	0.43713 (14)	0.0394 (5)
C5	−0.1169 (4)	0.6893 (3)	0.28921 (14)	0.0384 (5)
H5	−0.1356	0.5674	0.2568	0.046*
C11	0.2897 (4)	0.7157 (3)	0.06729 (14)	0.0384 (5)
H11	0.2208	0.6260	0.0196	0.046*
C7	−0.2581 (4)	0.8815 (3)	0.39976 (14)	0.0410 (5)
H7	−0.3726	0.8898	0.4404	0.049*
C2	−0.0603 (4)	1.0497 (3)	0.38714 (13)	0.0349 (5)
C6	−0.2820 (5)	0.7011 (3)	0.35072 (14)	0.0454 (6)
H6	−0.4108	0.5872	0.3595	0.054*
O3	0.2280 (3)	0.3289 (2)	0.18749 (10)	0.0466 (4)
O5	0.5525 (3)	0.3893 (2)	0.10947 (10)	0.0513 (5)
N1	0.3185 (4)	0.3064 (2)	0.12032 (11)	0.0355 (4)
O4	0.1696 (3)	0.1940 (2)	0.05948 (10)	0.0479 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N2	0.0348 (10)	0.0396 (9)	0.0369 (11)	0.0138 (7)	0.0154 (8)	0.0091 (8)
O2	0.0551 (11)	0.0408 (9)	0.0583 (11)	0.0013 (7)	0.0264 (9)	−0.0027 (7)
O1	0.0538 (11)	0.0479 (10)	0.0593 (12)	0.0076 (8)	0.0287 (9)	−0.0062 (8)
C4	0.0293 (11)	0.0351 (11)	0.0310 (12)	0.0097 (8)	0.0049 (9)	0.0057 (8)
C8	0.0289 (11)	0.0306 (10)	0.0345 (12)	0.0104 (8)	0.0059 (9)	0.0075 (8)
C12	0.0317 (11)	0.0327 (10)	0.0390 (13)	0.0057 (8)	0.0102 (10)	0.0017 (9)
C10	0.0311 (12)	0.0372 (11)	0.0440 (14)	0.0093 (9)	0.0066 (10)	0.0102 (10)
C9	0.0304 (11)	0.0353 (10)	0.0354 (12)	0.0077 (8)	0.0055 (9)	0.0038 (9)
C3	0.0312 (11)	0.0341 (10)	0.0318 (12)	0.0051 (8)	0.0053 (9)	0.0047 (9)
C1	0.0409 (13)	0.0431 (12)	0.0346 (12)	0.0113 (10)	0.0125 (10)	0.0052 (9)
C5	0.0415 (13)	0.0315 (11)	0.0394 (13)	0.0054 (9)	0.0124 (10)	0.0032 (9)
C11	0.0383 (13)	0.0349 (11)	0.0398 (13)	0.0077 (9)	0.0090 (10)	0.0015 (9)
C7	0.0411 (13)	0.0458 (12)	0.0350 (13)	0.0081 (10)	0.0160 (10)	0.0068 (10)
C2	0.0353 (12)	0.0375 (11)	0.0316 (12)	0.0097 (9)	0.0064 (10)	0.0041 (9)
C6	0.0453 (14)	0.0393 (12)	0.0457 (14)	−0.0001 (10)	0.0159 (11)	0.0071 (10)
O3	0.0551 (11)	0.0481 (9)	0.0378 (10)	0.0147 (7)	0.0171 (8)	0.0054 (7)
O5	0.0345 (10)	0.0517 (9)	0.0570 (11)	−0.0034 (7)	0.0119 (8)	0.0011 (8)
N1	0.0376 (11)	0.0296 (9)	0.0397 (11)	0.0085 (7)	0.0093 (9)	0.0061 (8)
O4	0.0348 (9)	0.0587 (10)	0.0393 (9)	0.0008 (7)	0.0076 (7)	−0.0064 (7)

Geometric parameters (Å, °)

N2—C11	1.338 (3)	C9—H9	0.9300
N2—C10	1.339 (3)	C3—C2	1.385 (3)
N2—H2	0.8600	C3—H3	0.9300
O2—C1	1.263 (3)	C1—C2	1.487 (3)
O1—C1	1.267 (3)	C5—C6	1.377 (3)
O1—H1	0.8200	C5—H5	0.9300
C4—C3	1.391 (3)	C11—H11	0.9300
C4—C5	1.398 (3)	C7—C6	1.388 (3)

C4—C8	1.481 (3)	C7—C2	1.393 (3)
C8—C12	1.392 (3)	C7—H7	0.9300
C8—C9	1.393 (3)	C6—H6	0.9300
C12—C11	1.360 (3)	O3—N1	1.235 (2)
C12—H12	0.9300	O5—N1	1.240 (2)
C10—C9	1.364 (3)	N1—O4	1.272 (2)
C10—H10	0.9300		
C11—N2—C10	121.3 (2)	O2—C1—O1	123.68 (19)
C11—N2—H2	119.4	O2—C1—C2	118.7 (2)
C10—N2—H2	119.4	O1—C1—C2	117.61 (19)
C1—O1—H1	109.5	C6—C5—C4	120.80 (19)
C3—C4—C5	118.4 (2)	C6—C5—H5	119.6
C3—C4—C8	120.40 (18)	C4—C5—H5	119.6
C5—C4—C8	121.07 (17)	N2—C11—C12	120.49 (19)
C12—C8—C9	116.8 (2)	N2—C11—H11	119.8
C12—C8—C4	121.07 (18)	C12—C11—H11	119.8
C9—C8—C4	122.08 (18)	C6—C7—C2	119.0 (2)
C11—C12—C8	120.60 (19)	C6—C7—H7	120.5
C11—C12—H12	119.7	C2—C7—H7	120.5
C8—C12—H12	119.7	C3—C2—C7	120.33 (18)
N2—C10—C9	119.82 (19)	C3—C2—C1	119.46 (18)
N2—C10—H10	120.1	C7—C2—C1	120.2 (2)
C9—C10—H10	120.1	C5—C6—C7	120.6 (2)
C10—C9—C8	121.00 (19)	C5—C6—H6	119.7
C10—C9—H9	119.5	C7—C6—H6	119.7
C8—C9—H9	119.5	O3—N1—O5	122.40 (18)
C2—C3—C4	120.76 (18)	O3—N1—O4	119.71 (18)
C2—C3—H3	119.6	O5—N1—O4	117.89 (19)
C4—C3—H3	119.6		
C3—C4—C8—C12	−147.6 (2)	C8—C4—C5—C6	−174.7 (2)
C5—C4—C8—C12	28.9 (3)	C10—N2—C11—C12	−0.6 (3)
C3—C4—C8—C9	29.6 (3)	C8—C12—C11—N2	0.0 (3)
C5—C4—C8—C9	−154.0 (2)	C4—C3—C2—C7	1.1 (3)
C9—C8—C12—C11	0.5 (3)	C4—C3—C2—C1	−176.26 (19)
C4—C8—C12—C11	177.82 (19)	C6—C7—C2—C3	0.9 (3)
C11—N2—C10—C9	0.6 (3)	C6—C7—C2—C1	178.2 (2)
N2—C10—C9—C8	0.0 (3)	O2—C1—C2—C3	−10.7 (3)
C12—C8—C9—C10	−0.6 (3)	O1—C1—C2—C3	168.3 (2)
C4—C8—C9—C10	−177.83 (18)	O2—C1—C2—C7	172.0 (2)
C5—C4—C3—C2	−2.4 (3)	O1—C1—C2—C7	−9.0 (3)
C8—C4—C3—C2	174.15 (18)	C4—C5—C6—C7	0.1 (4)
C3—C4—C5—C6	1.8 (3)	C2—C7—C6—C5	−1.5 (4)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
C10—H10 $\cdots$ O4 ⁱ	0.93	2.42	3.225 (3)	145
C11—H11 $\cdots$ O5 ⁱⁱ	0.93	2.49	3.122 (3)	125
O1—H1 $\cdots$ O2 ⁱⁱⁱ	0.82	1.82	2.624 (2)	167
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