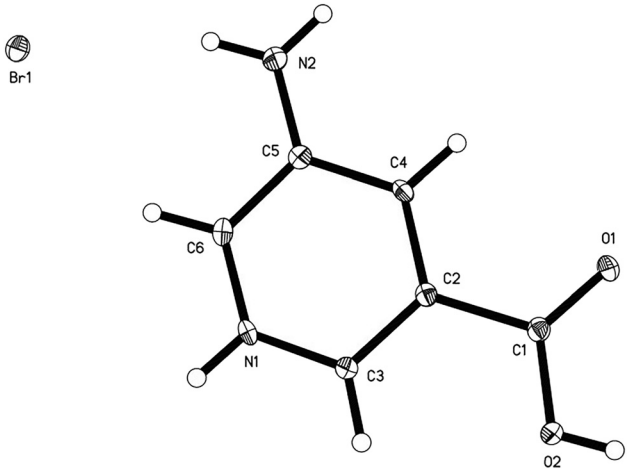
Chao-Jun Du*, De-Long Niu, Zhen-Li Yan, Shi-Li Zheng and Yan Zeng

The crystal structure of 3-amino-5-carboxypyridin-1-ium bromide, $C_6H_7BrN_2O_2$



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Abstract

$C_6H_7BrN_2O_2$, triclinic, $P\bar{1}$ (no. 2), a = 7.3026(4) Å, b = 7.4618(4) Å, c = 7.8746(4) Å, α = 81.542(3)°, β = 89.459(3)°, γ = 61.718(3)°, V = 372.86(4) Å^3, Z = 2, $R_{gt}(F)$ = 0.0209, $wR_{ref}(F^2)$ = 0.0473, T = 150(2) K.

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The asymmetric unit of the title salt structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colorless needle
Size:	0.20 × 0.15 × 0.10 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	5.46 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	26.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7980, 1537, 0.042
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 1398
$N(param)_{refined}$:	108
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

Source of material

All starting materials were purchased and used as received. Under stirring, 1.38 g of 5-aminopyridine-3-carboxylic acid (10 mmol) was mixed with 10 mL 6 M hydrobromic acid to form a solution. After 15 min, the solution was filtered. Colorless crystals were deposited after about 50 h, yield 65% (based on 5-aminopyridine-3-carboxylic acid).

Experimental details

The structure was solved by direct methods with the SHELXS-2018 program. All H-atoms were positioned with idealized geometry and refined isotropic (U_{iso}(H) = 1.2U_{eq}(C)) and (N) using a riding model with C–H = 0.95 Å and N–H = 0.88 Å of amino group (If the two H atoms of amino group were refined freely, the acceptor of H2A was not found). The H-atoms from O atom and N of pyridine ring were positioned with Q peaks and refined freely with the distance of O2–H2 = 0.795 and N1–H1 = 0.867 Å, respectively.

Comment

Well known as an excellent organic linker, 5-aminopyridine-3-carboxylic acid, that has three different potential coordination groups at the same time, has been exploited to construct metal complexes [5–13]. Recently, the crystal structure of the corresponding perchlorate salt has been

***Corresponding author: Chao-Jun Du**, Nanyang Research Institute of Zhengzhou University, Nanyang Institute of Technology, Nanyang, Henan 473000, P. R. China, E-mail: hyperchem@126.com. https://orcid.org/0000-0002-4767-7057
De-Long Niu and Zhen-Li Yan, State Key Laboratory of Motor Vehicle Biofuel Technology, Nanyang, Henan 473000, P. R. China
Shi-Li Zheng, Innovation Academy for Green Manufacture, Chinese Academy of Sciences, Beijing 100190, P. R. China
Yan Zeng, College of Chemistry and Chemical Engineering, Xinxiang University, Xinxiang, Henan 453003, P. R. China

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.73011 (4)	0.11619 (3)	0.12488 (3)	0.02096 (8)
C1	0.7768 (3)	0.8168 (3)	0.7494 (3)	0.0161 (4)
C2	0.7724 (3)	0.6531 (3)	0.6628 (3)	0.0137 (4)
C3	0.8191 (3)	0.4625 (3)	0.7575 (3)	0.0168 (4)
H3	0.853904	0.432947	0.877970	0.020*
C4	0.7662 (3)	0.3524 (3)	0.5041 (3)	0.0174 (5)
H4	0.766441	0.245151	0.452135	0.021*
C5	0.7164 (3)	0.5424 (3)	0.4037 (3)	0.0149 (4)
C6	0.7223 (3)	0.6935 (3)	0.4880 (3)	0.0146 (4)
H6	0.691664	0.823974	0.424192	0.018*
H1	0.839 (5)	0.204 (5)	0.732 (4)	0.041 (9)*
H2	0.810 (5)	0.854 (5)	0.961 (4)	0.043 (9)*
N1	0.8135 (3)	0.3216 (3)	0.6730 (2)	0.0190 (4)
N2	0.6669 (3)	0.5749 (3)	0.2329 (2)	0.0238 (4)
H2A	0.665895	0.476291	0.184485	0.029*
H2B	0.635612	0.694739	0.170076	0.029*
O1	0.7418 (3)	0.9827 (2)	0.6701 (2)	0.0240 (4)
O2	0.8235 (3)	0.7595 (3)	0.9161 (2)	0.0206 (4)

reported [14]. But the crystal structure of its hydrobromide has not been published.

There are two parts in the asymmetric unit: one is the 3-amino-5-carboxypyridin-1-ium cation, and the other is the bromide anion. The nitrogen atom of the pyridine ring is protonated (see the Figure). The distances of C1–O2 and C1–O1 are 1.209 and 1.314 Å, respectively, indicating that the latter is single-bonded. The 3-amino-5-carboxypyridin-1-ium cations are linked by hydrogen bonds N1–H1···O1′ and N2–H2B···O2″ to form one-dimensional chains, which further are bridged by N2–H2A···Br1 and O2–H2···Br1‴ to generate a two-dimensional supramolecular structure. All of the C–C, C–N and C–O bond lengths are similar to the reported 3-amino-5-carboxypyridin-1-ium perchlorate monohydrate [14].

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