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The crystal structure of 6-amino-2-carboxypyridin-1-ium bromide, $C_6H_7BrN_2O_2$

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Abstract

$C_6H_7BrN_2O_2$, monoclinic, $C2/c$ (no. 15), $a = 23.7470(19)$ Å, $b = 5.4355(4)$ Å, $c = 15.3336(11)$ Å, $\beta = 127.291(2)^\circ$, $V = 1574.6(2)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0292$, $wR_{\text{ref}}(F^2) = 0.0756$, $T = 150(2)$ K.

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The molecular 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.

Source of material

All reagents are purchased from TCI Chemicals and used as received. An amount of 1.38 g of 6-aminopicolinic acid (10 mmol) was added to 10 mL concentrated hydrobromic

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.20 × 0.15 × 0.10 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	5.17 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max} , completeness:	26.4°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	10,308, 1605, 0.065
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1263
$N(\text{param})_{\text{refined}}$:	104
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

acid (40%) and stirred for 10 min at room temperature, then filtered, left quietly, and evaporated naturally. Several days later, colorless crystals of 6-amino-2-carboxypyridin-1-ium bromide, $C_6H_7BrN_2O_2$ (ACPB) were obtained, yield 58% (based on 6-aminopicolinic acid).

Experimental details

The structure was solved by direct methods with the SHELXS-2018 program. All H-atoms from C and N atoms were positioned with idealized geometry and refined

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.35629 (2)	0.70045 (7)	0.18186 (3)	0.02907 (13)
C1	0.35421 (19)	0.2212 (7)	0.3494 (3)	0.0250 (8)
C2	0.38342 (19)	0.3998 (7)	0.4415 (3)	0.0242 (8)
C3	0.45042 (19)	0.4028 (7)	0.5351 (3)	0.0263 (8)
H3	0.483723	0.281395	0.548937	0.032*
C4	0.46953 (19)	0.5918 (7)	0.6117 (3)	0.0266 (8)
H4	0.516490	0.598262	0.677762	0.032*
C5	0.4222 (2)	0.7632 (7)	0.5925 (3)	0.0262 (8)
H5	0.436086	0.890552	0.644345	0.031*
C6	0.3517 (2)	0.7534 (6)	0.4950 (3)	0.0245 (8)
H1	0.391 (3)	−0.027 (9)	0.325 (4)	0.049(17)*
N1	0.30312 (17)	0.9169 (6)	0.4716 (2)	0.0311 (7)
H1A	0.260270	0.907483	0.409111	0.037*
H1B	0.313628	1.035041	0.518458	0.037*
N2	0.33528 (15)	0.5722 (5)	0.4226 (2)	0.0231 (7)
H2	0.291958	0.564967	0.361020	0.028*
O1	0.40101 (15)	0.0577 (5)	0.3690 (2)	0.0333 (7)
O2	0.29337 (13)	0.2345 (5)	0.26787 (19)	0.0273 (6)

isotropically ($U_{iso}(H) = 1.2U_{eq}(C)$ and $U_{iso}(H) = 1.2U_{eq}(N)$) using a riding model with $C-H = 0.95 \text{ \AA}$ and $N-H = 0.88 \text{ \AA}$. The hydrogen atom at O1 was seen in the Fourier difference map and refined with the distance of $O2-H2 = 0.84 \text{ \AA}$ ($U_{iso}(H) = 1.5U_{eq}(O)$).

Comment

Known as a multi-functional ligand, 6-aminopicolinic acid was used to construct metal complexes or metal-organic frameworks [5–9]. We here report the crystal structure of the bromide salt of the 6-aminopicolinic acid, ACPB. ACPB crystallizes in monoclinic system, $C2/c$ group (no. 15). The asymmetric unit is composed of one protonated 6-aminopicolinic acid (2-amino-6-carboxypyridin-1-ium cation) and one bromide anion. The 2-amino-6-carboxypyridin-1-ium cations are linked to each other by $N-H \cdots Br$; $O-H \cdots Br$ and $N-H \cdots O$ hydrogen bonds, respectively. All of the bond lengths of the ACPB are similar to the reported results [5–10].

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