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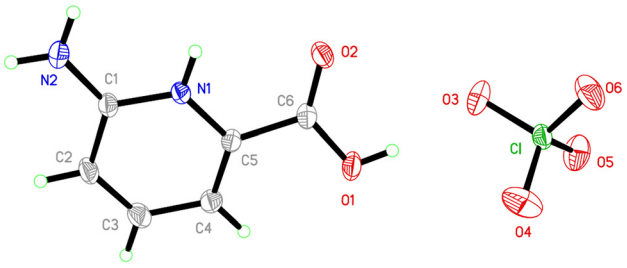
The crystal structure of 6-amino-2-carboxypyridin-1-ium perchlorate, $C_6H_7ClN_2O_6$



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Abstract

$C_6H_7ClN_2O_6$, monoclinic, $P2_1/c$ (no. 14), a = 9.2699(5) Å, b = 12.5329(6) Å, c = 7.9536(5) Å, β = 99.472(6)°, V = 911.44(9) Å^3, Z = 4, $R_{gt}(F)$ = 0.0442, wR_{ref} (F^2) = 0.1120, T = 292.98(10) K.

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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:	Colourless block
Size:	0.13 × 0.12 × 0.09 mm
Wavelength:	MoKα radiation (0.71073 Å)
μ:	0.43 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	27.0°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	4014, 1941, 0.023
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 1544
$N(param)_{refined}$:	137
Programs:	CrysAlis^{Pro} [1], Olex2 [2], Shelx [3, 4]

1 Source of materials

An amount of 0.138 g 6-aminopyridine-2-carboxylic acid (1.0 mmol) was added to 10 mL 70 % perchloric acid, stirred for 10 min, and filtered. The clear solution evaporated slowly at room temperature. After several days, many colorless crystals were obtained, washed with deionized water and anhydrous ethanol, and dried in air, yield 63 % (based on 6-aminopyridine-2-carboxylic acid).

2 Experimental details

The structure was solved by Direct Methods with the Shelxs-2018 program. All H-atoms from C atoms were positioned with idealized geometry and refined isotropically (U_{iso}(H) = 1.2 U_{eq}(C) or U_{eq}(C)) using a riding model with C–H = 0.930 Å and N–H = 0.860 Å. The H-atom from O atom positioned with Q peak refined isotropically with the distance of O–H = 0.82 Å (U_{iso}(H) = 1.5U_{eq}(O)).

3 Comment

The perchlorate salts of substituted 2-aminopyridines, such as 2-amino-3-carboxypyridinium perchlorate [5, 6], 2-amino-3-nitropyridinium perchlorate [7], 2-amino-3-benzyloxy pyridinium perchlorate [8], 2,6-diaminopyridinium perchlorate [9, 10], 6-aminopyridine-2-carboxylic acid, have been reported many times. Besides, some similar results, including 3-amino-5-carboxypyridin-1-ium perchlorate monohydrate [11], 6-amino-2-carboxypyridin-1-ium pentaiodide monohydrate [12], and 6-amino-2-carboxypyridin-1-ium bromide [13], have also been published elsewhere. However, the crystal structure of 6-amino-2-carboxypyridin-1-ium perchlorate has not been found. Thus, we report the single crystal structure of the title compound.

The asymmetrical unit consists of one 6-amino-2-carboxypyridin-1-ium cation and one perchlorate anion. The two adjacent 6-amino-2-carboxypyridin-1-ium cations

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.6821 (3)	0.27478 (19)	0.4286 (3)	0.0297 (5)
C2	0.8148 (3)	0.2386 (2)	0.3828 (3)	0.0370 (6)
H2	0.836323	0.166064	0.385570	0.044*
C3	0.9098 (3)	0.3090 (2)	0.3352 (4)	0.0407 (6)
H3	0.996756	0.284535	0.305031	0.049*
C4	0.8800 (3)	0.4193 (2)	0.3302 (3)	0.0355 (6)
H4	0.945835	0.467759	0.296905	0.043*
C5	0.7532 (3)	0.45253 (19)	0.3752 (3)	0.0285 (5)
C6	0.7030 (3)	0.56540 (19)	0.3762 (3)	0.0319 (6)
Cl	0.74513 (7)	0.91787 (5)	0.37484 (8)	0.0339 (2)
N1	0.6574 (2)	0.38101(15)	0.4227 (2)	0.0288 (5)
H1	0.577299	0.404478	0.450221	0.035*
N2	0.5846 (2)	0.21052 (17)	0.4788 (3)	0.0437 (6)
H2A	0.506200	0.236415	0.507367	0.052*
H2B	0.599539	0.142779	0.482959	0.052*
O1	0.8055 (2)	0.63344 (15)	0.3542 (3)	0.0476 (5)
H1A	0.773367	0.694372	0.354978	0.071*
O2	0.5806 (2)	0.58856 (14)	0.3952 (2)	0.0407 (5)
O3	0.6639 (3)	0.82812 (17)	0.2954 (3)	0.0689 (7)
O4	0.8767 (2)	0.8804 (2)	0.4724 (3)	0.0744 (8)
O5	0.7792 (3)	0.98764 (17)	0.2455 (3)	0.0594 (6)
O6	0.6586 (2)	0.97236 (17)	0.4796 (3)	0.0607 (6)

are connected by the hydrogen bond N1–H1···O2 to form a dimer. The dimers are linked by the hydrogen bonds O1–H1A···O3, N2–H2B···O6, and C2–H2···O5 to generate a 1D structure, which is further bridged by the hydrogen bond C4–H4···O5 to construct a 2D sheet. All the bonds are comparable with the similar results [5–13].

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Competing interest: The authors declare no conflicts of interest regarding this article.

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Data availability: The datasets used or analysed during the current study are available from the corresponding author on reasonable request.

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