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The crystal structure of 2-amino-5-carboxypyridin-1-ium iodide monohydrate, $C_6H_9IN_2O_3$

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Abstract

$C_6H_9IN_2O_3$, monoclinic, $P2_1/c$ (no. 14), $a = 9.3654(2)$ Å, $b = 8.0312(2)$ Å, $c = 12.7668(3)$ Å, $\beta = 107.825(1)^\circ$, $V = 914.16(4)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0156$, $wR_{ref}(F^2) = 0.0354$, $T = 150(2)$ K.

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The asymmetric unit of the title crystal 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.

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.25 × 0.15 × 0.10 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	3.48 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} :	11,225, 1856, 0.027
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 1718
$N(param)_{refined}$:	113
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}^*/U_{eq}
C1	0.3796 (2)	0.9077 (3)	0.58637 (16)	0.0230 (4)
C2	0.4503 (2)	0.7404 (2)	0.60802 (16)	0.0205 (4)
C3	0.3672 (2)	0.5897 (3)	0.59103 (16)	0.0209 (4)
H3	0.260836	0.593148	0.563624	0.025*
C4	0.4385 (2)	0.4404 (2)	0.61360 (16)	0.0213 (4)
H4	0.381851	0.340272	0.602708	0.026*
C5	0.5972 (2)	0.4342 (2)	0.65336 (15)	0.0209 (4)
C6	0.6027 (2)	0.7309 (3)	0.64626 (16)	0.0227 (4)
H6	0.660799	0.830015	0.657726	0.027*
I1	0.04451 (2)	0.46190 (2)	0.70205 (2)	0.02843 (6)
N1	0.67348 (19)	0.2933 (2)	0.67628 (14)	0.0247 (4)
H1A	0.772105	0.295324	0.700878	0.030*
H1B	0.625638	0.197461	0.666933	0.030*
N2	0.67200 (18)	0.5812 (2)	0.66806 (14)	0.0228 (4)
H2	0.770601	0.579217	0.693029	0.027*
O1	0.45055 (17)	1.03579 (18)	0.60996 (13)	0.0311 (3)
O2	0.23347 (16)	0.90043 (19)	0.54117 (14)	0.0328 (4)
H2A	0.198206	0.997381	0.531565	0.049*
O1W	0.12017 (19)	0.1979 (2)	0.49738 (14)	0.0380 (4)
H1WA	0.083415	0.250643	0.542873	0.057*
H1WB	0.067890	0.221842	0.430133	0.057*

Source of materials

All the raw materials were used as received without further purification. An amount of 1.38 g (10 mmol) 6-aminonicotinic acid was mixed with 10 mL 40% hydroiodic acid at room temperature. Then the mixture was

filtered and stood after stirring for 30 min. One week later, colorless block crystals of the title compound suitable for single crystal X-ray diffraction measurement were obtained, yield 84% (based on 6-aminonicotinic 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 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 Å and N–H = 0.88 Å. The hydrogen atoms at oxygen atoms were refined with the distance of O2–H2A = 0.84 Å, O1W–H1WA = 0.87 Å and O1W–H1WB = 0.87 Å, respectively ($U_{iso}(H) = 1.5U_{eq}(O)$).

Comment

The 6-amino-nicotinic acid was used as a bifunctional ligand for Zn(II), Mn(II) and Co(II), Cd(II), and Ca(II) and Sr(II) to construct coordination polymers and metal-organic frameworks [5–9]. There also have been some salts of it such as its chloride, bromide, nitrate and dihydrogen phosphate [10–12] which are based on the monoprotonated cation of 6-amino-nicotinic acid. However, the crystal structure of its iodide has not been reported.

The title compound, 2-amino-5-carboxypyridin-1-ium iodide monohydrate, with the formula of $C_6H_9IN_2O_3$, crystallizes monoclinically, $P2_1/c$ (no. 14). The asymmetric unit is made of one *N*-protonated 2-amino-5-carboxypyridin-1-ium cation, one iodine anion, and one water molecule. All the bond lengths of the title compound are in the normal ranges [13]. The N atom of the pyridine is protonated to give the 2-amino-5-carboxypyridin-1-ium cation to balance the iodine anion. All the atoms of the 2-amino-5-carboxypyridin-1-ium cation are nearly co-planar. There is a one dimensional chain generated by hydrogen bond N1–H1B...O1.

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

References

1. Bruker. SAINT (v8.37A); Bruker AXS Inc: Madison, Wisconsin, USA, 2015.
2. Bourhis L. J., Dolomanov O. V., Gildea R. J., Howard J. A. K., Puschmann H. The anatomy of a comprehensive constrained, restrained refinement program for the modern computing environment—Olex2 dissected. *Acta Crystallogr.* 2015, *A71*, 59–75.
3. Sheldrick G. M. Crystal structure refinement with SHELXL. *Acta Crystallogr.* 2015, *C71*, 3–8.
4. Sheldrick G. Using phases to determine the space group. *Acta Crystallogr.* 2018, *A74*, a353.
5. Cepeda J., Sebastian E. S., Padro D., Rodríguez-Dieguez A., García J. A., Ugalde J. M., Seco J. M. A Zn based coordination polymer exhibiting long-lasting phosphorescence. *Chem. Commun.* 2016, 52, 8671–8674.
6. Wu Y.-L., Yang Y., Qian J., Yang G.-P., Jin J., Miao H.-H., Yuan J., Wang Y.-Y. Low-pressure selectivity, stepwise gas sorption behaviors, and luminescent properties (experimental findings and theoretical correlation) of three Zn(II)-based metal-organic frameworks. *Cryst. Growth Des.* 2017, *17*, 3965–3973.
7. Rodríguez-Dieguez A., Perez-Yanez S., Ruiz-Rubio L., Seco J. M., Cepeda J. From isolated to 2D coordination polymers based on 6-aminonicotinate and 3d-metal ions: towards field-induced single-ion-magnets. *CrystEngComm* 2017, *19*, 2229–2242.
8. Zhou W.-W., Zhao W., Wang F.-W., Fang W.-Y., Liu D.-F., Wei Y.-J., Xu M., Zhao X., Liang X. A 3-D metal-organic framework with a rutile topology network, right- or left-handed helical chains and tunable UV-to-visible photoluminescence. *RSC Adv.* 2015, *5*, 42616–42620.
9. Pajuelo-Corral O., Rodríguez-Dieguez A., Beobide G., Perez-Yanez S., García J. A., Sebastian E. S., Seco J. M., Cepeda J. Alkaline-earth and aminonicotinate based coordination polymers with combined fluorescence/long-lasting phosphorescence and metal ion sensing response. *J. Mater. Chem. C* 2019, *7*, 6997–7012.
10. Giannidis J., Turnbull M. M. 6-amino-nicotinic acid hydrochloride. *Acta Crystallogr.* 2000, *C56*, 334–335.
11. Dhauadi H., Marouani H., Rzaigui M., Madani A. Crystal structure and investigation of an organic phosphate. *Phosphorus, Sulfur Silicon Relat. Elem.* 2008, *182*, 2587–2603.
12. Wang J.-W., Zhang Y.-W., Wang M.-X., Luo Y.-H., Sun B.-W. Assembly of 6-aminonicotinic acid and inorganic anions into different dimensionalities: crystal structure, absorption properties and Hirshfeld surface analysis. *Polyhedron* 2017, *124*, 243–250.
13. Reiss G. J., Leske P. B. 2-Aminopyridin-1-ium triiodide. *Acta Crystallogr.* 2013, *E69*, o1060–o1061.