Dan-Xia Li*, Hai-Jun Wang, Zhen-Li Yan and Chao-Jun Du

The crystal structure of 6-amino-5-carboxypyridin-1-ium pentaiodide monohydrate $C_6H_9I_5N_2O_3$

Table 1: Data collection and handling.

Crystal:	Red block
Size:	0.20 × 0.15 × 0.10 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	9.39 mm ⁻¹
Diffractometer, scan mode:	BRUKER APEX-II, φ and ω
$\theta_{\max}$, completeness:	26.4°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	17,263, 3386, 0.045
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 2864
$N(\text{param})_{\text{refined}}$:	149
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

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Abstract

$C_6H_9I_5N_2O_3$, triclinic, $P\bar{1}$ (no. 2), $a = 7.4851(5)$ Å, $b = 9.6056(6)$ Å, $c = 12.4084(7)$ Å, $\alpha = 109.119(3)^\circ$, $\beta = 99.341(3)^\circ$, $\gamma = 92.334(3)^\circ$, $V = 827.53(9)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0234$, $wR_{\text{ref}}(F^2) = 0.0449$, $T = 150.0$ 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

An amount of 1.38 g of 6-aminopyridine-2-carboxylic acid (10 mmol) was dissolved in 10 mL 45% hydroiodic acid, then filtered. Many red crystals were harvested after about 10 days, yield 74% (based on 6-aminopyridine-2-carboxylic acid).

Experimental details

The structure was solved by direct methods with the SHELXS program. All H-atoms from C and N atoms were positioned with idealized geometry ($U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ and (N)) using a riding model with C–H = 0.95 Å and N–H = 0.88 Å. The H-atoms for O and O1W were refined with the distance of O1–H1A = 0.84, O1–H1WA = O1–H1WB = 0.87 Å with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$, respectively.

Comment

Many pyridinium-based polyiodides have been reported, for example, 2-amino-pyridin-1-ium triiodide [5, 6] and bis((4-aminopyridin-1-ium) iodide triiodide. But only a few pyridinium-based pentaiodides have been published, such as aminopyridinium pentaiodide [7], isoquinolinium pentaiodide [8], nevirapinium pentaiodide [9], *N*-(6-methyl-2-pyridyl)mesitylenesulfonamide pentaiodide [10], and

*Corresponding author: Dan-Xia Li, Zhang Zhongjing College of Chinese Medicine, Nanyang Institute of Technology, Nanyang, Henan, 473000, P. R. China, E-mail: 781488117@qq.com

Hai-Jun Wang, Agriculture Engineering Department, Nanyang Vocational College of Agriculture, Nanyang, Henan, 473000, P. R. China

Zhen-Li Yan, State key Laboratory of Motor Vehicle Biofuel Technology, Nanyang, Henan, 473000, P. R. China

Chao-Jun Du, Nanyang Research Institute of Zhengzhou University, Nanyang Institute of Technology, Nanyang, Henan, 473000, P. R. China. <https://orcid.org/0000-0002-4767-7057>

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.9540 (6)	0.2526 (5)	0.6136 (4)	0.0204 (10)
C2	0.8744 (6)	0.2678 (5)	0.5012 (4)	0.0209 (10)
C3	0.8110 (6)	0.3904 (5)	0.4862 (4)	0.0257 (11)
H3	0.816407	0.478628	0.550662	0.031*
C4	0.7357 (6)	0.3856 (6)	0.3719 (4)	0.0279 (11)
H4	0.687965	0.470450	0.359699	0.033*
C5	0.7322 (6)	0.2592 (6)	0.2800 (4)	0.0269 (11)
H5	0.682366	0.256349	0.203720	0.032*
C6	0.8028 (6)	0.1314 (5)	0.2977 (4)	0.0208 (10)
I1	0.62729 (5)	0.31666 (4)	0.96864 (3)	0.02959 (9)
I2	0.48900 (4)	0.25468 (3)	0.69938 (2)	0.02325 (8)
I3	0.36535 (4)	0.18894 (4)	0.46452 (3)	0.03150 (9)
I4	0.80776 (5)	−0.30113 (4)	0.81772 (3)	0.03111 (9)
I5	0.72338 (4)	−0.00810 (3)	0.89310 (2)	0.02435 (8)
N1	0.8668 (5)	0.1409 (4)	0.4078 (3)	0.0189 (8)
H1	0.905890	0.061554	0.420587	0.023*
N2	0.8079 (5)	0.0076 (4)	0.2119 (3)	0.0277 (9)
H2A	0.853126	−0.068723	0.226765	0.033*
H2B	0.765963	0.001213	0.139807	0.033*
O1	0.9811 (5)	0.3782 (3)	0.7009 (3)	0.0275 (8)
H1A	1.020862	0.362390	0.762739	0.041*
O2	0.9927 (4)	0.1336 (3)	0.6218 (2)	0.0223 (7)
O1W	0.0920 (6)	0.3149 (4)	0.8878 (3)	0.0399 (9)
H1WA	0.199111	0.349720	0.930224	0.060*
H1WB	0.017030	0.342507	0.935769	0.060*

3-[5-(pyridin-3-yl)-1,2,4-thiadiazol-3-yl]pyridinium pentaio-
dide [11].

The title compound, with the formula of $\text{C}_6\text{H}_9\text{I}_5\text{N}_2\text{O}_3$, crystallizes in triclinic, *P*-1 (no. 2). The asymmetric unit is made of one 6-amino-5-carboxypyridin-1-ium cation, one pentaiodide anion and one water molecule. In the 6-amino-5-carboxypyridin-1-ium cation, the N atom from of the pyridine ring is protonated and both the amino and carboxyl groups are neutral. The distances of I4–I5 and I2–I3 are 2.794 and 2.760 $\text{\AA}$, respectively, rather shorter than the distances of I1–I5 (3.101 $\text{\AA}$) and I1–I2 (3.181 $\text{\AA}$). The structure features an organic moiety consisting of two water molecules and two cations to form the dimers, which are generated by the hydrogen bonds N1–H1...O2, N2–H2A...O2, and O1–H1A...O1W. The anions and the cations are connected by van der Waals interactions to form layers in the *bc* plane.

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