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The crystal structure of 3-bromoisonicotinic acid, $C_6H_4BrNO_2$

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	$0.25 \times 0.15 \times 0.10$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	6.32 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max} , completeness:	26.0° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	7419, 1263, 0.061
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1119
$N(\text{param})_{\text{refined}}$:	95
Programs:	Bruker [1], SHELXT [2], Olex2 [3, 4]

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.31443(7)	0.07999(6)	0.77220(6)	0.0268(2)
C1	0.2434(6)	0.4894(6)	0.9021(6)	0.0156(8)
C2	0.2406(6)	0.5263(6)	0.6891(6)	0.0148(8)
C3	0.2684(6)	0.3667(6)	0.6160(6)	0.0141(8)
C4	0.2666(6)	0.4153(6)	0.4193(6)	0.0179(8)
H4	0.2866(6)	0.3051(7)	0.3733(7)	0.021*
C5	0.2105(6)	0.7657(6)	0.3534(6)	0.0193(8)
H5	0.1896(4)	0.9051(8)	0.2618(5)	0.023*
C6	0.2116(6)	0.7284(6)	0.5505(6)	0.0172(8)
H6	0.1922(9)	0.8418(2)	0.5913(5)	0.021*
H1	0.274(10)	0.633(10)	1.046(10)	0.056(18)*
N1	0.2381(6)	0.6109(5)	0.2878(5)	0.0188(7)
O1	0.2623(5)	0.6511(5)	0.9162(5)	0.0257(7)
O2	0.2278(5)	0.3314(4)	1.0405(5)	0.0235(7)

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Abstract

$C_6H_4BrNO_2$, triclinic, $P\bar{1}$ (no. 2), $a = 7.2512(6)$ Å, $b = 7.3916(6)$ Å, $c = 7.4920(7)$ Å, $\alpha = 65.065(4)^\circ$, $\beta = 68.858(4)^\circ$, $\gamma = 64.919(4)^\circ$, $V = 321.60(5)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0369$, $wR_{\text{ref}}(F^2) = 0.1017$, $T = 150(2)$ 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 of the starting materials were used as received. 4.02 g 3-bromoisonicotinic acid (2 mmol) was added to 50 mL THF

at room temperature. After stirring for 5 min, the solution was filtered and sealed with perforated parafilm. Colorless crystals suitable for single XRD were harvested; yield 78.2% (based on 3-bromoisonicotinic acid).

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Experimental details

The structure was solved by direct methods with the program SHELXT [2] and refined with Olex2 [3, 4]. H-atoms attached to C were positioned with idealized geometry and refined using a riding model with $C-H = 0.950$ Å and $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(C)$. The acidic H-atom was freely refined.

Comment

Several single crystal structures of mono-halogen substituted isonicotinic acid have been reported in the literature. There are several single crystal structures of transition metal or lanthanide complexes based on 2-fluoroisonicotinic acid [5, 6] and 3-fluoroisonicotinic acid [7–10]. The crystal structures of three polymorphs of 3-chloroisonicotinic acid [11] and two of its Cu(I) and Ag(I) complexes have also been reported [12, 13]. Some examples of uranyl complexes of di-halogen substituted isonicotinic acid such as 3,5-dichloroisonicotinic acid, 2,6-dibromoisonicotinic acid and 3,5-dibromoisonicotinic acid have also been published [8]. However, to the best of our knowledge, no structures of mono-bromoisonicotinic acid or its metal coordination complexes have been reported.

The crystal structure of 3-bromoisonicotinic acid is reported herein. The title compound crystallizes in the centrosymmetric triclinic space group and is isostructural with one of the polymorphs of 3-chloroisonicotinic acid [11]. O—H...N and C—H...O hydrogen bonds, arranged in a R₂²(7) fashion according to graph-set analysis [14], link molecules into chains that run along the *c*-axis. These chains are further stabilized by C—H...O contacts.

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