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The crystal structure of 2-bromoisonicotinic acid, $C_6H_4BrNO_2$

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Abstract

$C_6H_4BrNO_2$, monoclinic, $P2_1/c$ (no. 14), $a = 11.9715(14)$ Å, $b = 7.5753(7)$ Å, $c = 7.4498(8)$ Å, $\beta = 102.302(4)^\circ$, $V = 660.09(12)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0290$, $wR_{ref}(F^2) = 0.0635$, $T = 150(2)$ 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.

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Table 1: Data collection and handling.

Crystal:	Colourless
Size:	$0.25 \times 0.15 \times 0.10$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	6.15 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} :	8662, 1354, 0.058
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1106
$N(\text{param})_{\text{refined}}$:	92
Programs:	Bruker [1], SHELX [2], Olex2 [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>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.45682(3)	0.20988(4)	0.56170(5)	0.02182(12)
C1	0.1845(3)	0.7529(4)	0.6430(4)	0.0192(7)
C2	0.2045(3)	0.5562(4)	0.6335(4)	0.0166(7)
C3	0.1205(3)	0.4401(4)	0.6635(4)	0.0195(7)
H3	0.0509(3)	0.4828(4)	0.6892(4)	0.0234(8)*
C4	0.1405(3)	0.2614(4)	0.6551(5)	0.0207(7)
H4	0.0824(3)	0.1818(4)	0.6725(5)	0.0248(8)*
C5	0.3180(3)	0.3086(4)	0.5988(4)	0.0160(6)
C6	0.3057(3)	0.4902(4)	0.5985(4)	0.0166(7)
H6	0.3641(3)	0.5664(4)	0.5753(4)	0.0200(8)*
N1	0.2385(2)	0.1946(3)	0.6234(4)	0.0185(6)
O1	0.2684(2)	0.8449(3)	0.5986(4)	0.0254(6)
H1	0.2552(17)	0.9532(3)	0.606(5)	0.0380(8)*
O2	0.1016(2)	0.8137(3)	0.6875(4)	0.0293(6)

Source of material

The title compound, 2-bromoisonicotinic acid (2.01 g, 1 mmol), was added to 10 mL THF and stirred for 10 min under room temperature for 10 min. Then the solution was filtered and let evaporate in air. Many colorless crystals were obtained after 2 days, yield 83.5% (based on 2-bromoisonicotinic acid).

Experimental details

The structure was solved by direct methods with the SHELXS program. All H-atoms were positioned with idealized geometry and refined isotropically ($U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ and $1.5U_{\text{eq}}(\text{O})$, for all H atoms) using a riding model with C–H = 0.950–0.951 Å and O–H = 0.840 Å).

Comment

Up to now, the crystal structures of halogen substituted isonicotinic acid or their complexes have been reported rarely. To the best of our knowledge, there are only one example of 3-chloroisonicotinic acid [5] and two examples of 3-chloroisonicotinic acid based Cu(I) or Ag(I) complexes [6, 7] and a related Sn(II) complex [8]. There are also some examples of di-halogen substituted isonicotinic acids such as 3,5-dichloroisonicotinic acid, 2,6-dibromoisonicotinic acid and 3,5-dibromoisonicotinic acid [9]. Indeed, it has not been reported any crystal structure of mono-bromoisonicotinic acid or its complexes.

The title compound, 2-aminoisophthalic acid, with the molecule formula of C₆H₄BrNO₂, crystallizes in monoclinic, *P*2₁/*c* space group. All of the atoms are nearly co-planar, including the hydrogen atoms. The bond length of C—Br is 1.8956(36) Å, the bond lengths of C—C in the ring range from 1.384(4) to 1.389(4) Å. The two C—N are 1.326(4) and 1.344(4) Å. The single bond of C—C is 1.513(4) Å. The two C—O bond lengths from carboxyl group are 1.203(4) and 1.321(4), indicating that the shorter is C=O bond, and the longer is the C—O bond. There are only O—H···N hydrogen bonds, which link adjacent molecules to a one-dimensional supramolecular structure by a head-to-tail mode. All bond lengths and angles of 2-bromoisonicotinic acid are normal and comparable with its analogues.

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