Crystal structure of 2-acetamido-5-bromobenzoic acid, $C_9H_8BrNO_3$

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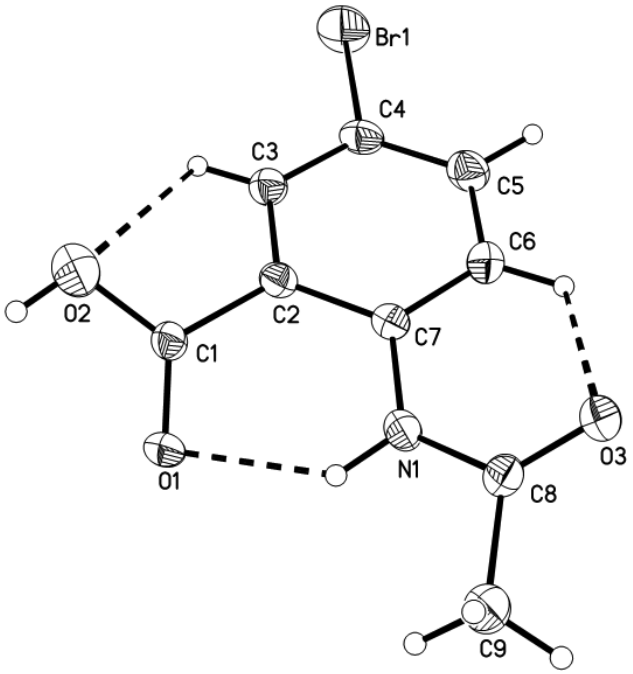

Abstract

$C_9H_8BrNO_3$, monoclinic, *C*12/*c*1 (no. 15), a = 14.919(4) Å, b = 9.333(2) Å, c = 15.508(4) Å, β = 113.13(1)°, V = 1985.8 Å^3, Z = 8, $R_{gt}(F)$ = 0.038, $wR_{ref}(F^2)$ = 0.100, T = 296 K.

Source of material

To a solution of lappaconitine 1 mmol (0.584 g) in acetic acid (40 mL), *N*-bromosuccinimide (NBS, 8 mmol, 1.42 g) was added, and the solution was heated for 8 h at 303 K. After basifying to pH = 10 with NH_4OH, the solution was extracted with $CHCl_3$ (20 mL). Drying (Na_2SO_4) and removal of solvent in vacuum afforded the mixed product. Adding $CHCl_3$ to the mixed product, a white precipitate was obtained. Colorless single crystals suitable for analysis were obtained by slowly evaporating the anhydrous methanol solution at room temperature for a week.

Experimental details

The hydrogen atoms H1, H3, H5 and H6 are found in the difference Fourier maps and refined freely. The other hydrgen atoms are positioned geometrically with d(C—H) = 0.96 Å, d(O—H) = 0.82 Å and U_{iso}(H) = 1.5 U_{eq}(C,O)

Discussion

Lappaconitine, an important alkaloid of the aconite family [1], exhibited a wide range of biological activities, for example, acesodyne and antiarrhythmic activities [2,3]. The title compound was obtained, when we tried to synthesize *N*-deethyllappaconitine by lappaconitine refering to the literature [4].

In the title crystal structure, there are three intramolecular hydrogen bonds. The first one is formed via H1 to N1 from the adjacent atom, O1 acts as a acceptor resulting in d(N1···O1) = 2.728 Å and ∠N1–H1···O1 of 138°. In the six-membered ring (O1, C1, C2, C7, N1, H1), all the atoms remain basically in the same plane . In C6–H6···O3 interaction, the carbonyl oxygen atom accepts protons from the adjacent atom to form hydrogen bond with d(C6···O3) = 2.919 Å and ∠C6–H6···O3 of 113°. A six-membered ring (H6, C6, C7, N1, C8, O3) is formed. All the atoms here remain in the same plane, with the mean deviation of 0.164 Å. With C3–H3···O2 interaction, a five-membered ring (H3, C3, O2, C1, C2) is formed, the carbonyl oxygen atom accepts protons from the adjacent atom to form hydrogen bond with d(C3···O2) = 2.867 Å and ∠C3–H3···O2 of 100°.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.23 × 0.27 × 0.31 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	41.20 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	49.3°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4729, 1683
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1385
$N(param)_{refined}$:	146
Programs:	SHELXS-97, SHELXL-97 [5]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(9A)	8*f*	–0.2700	0.9557	0.1267	0.082
H(9B)	8*f*	–0.2256	0.8144	0.1055	0.082
H(9C)	8*f*	–0.2858	0.9175	0.0233	0.082
H(2)	8*f*	0.0432	0.5823	–0.0350	0.099
H(1)	8*f*	–0.081(3)	0.808(5)	0.105(3)	0.05(1)
H(3)	8*f*	0.211(3)	0.805(4)	0.103(2)	0.04(1)
H(5)	8*f*	0.190(3)	1.188(5)	0.217(3)	0.05(1)
H(6)	8*f*	0.030(3)	1.140(5)	0.183(3)	0.05(1)

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Br(1)	8*f*	0.35211(3)	1.03064(6)	0.20033(4)	0.0372(3)	0.0721(4)	0.0875(4)	–0.0187(2)	0.0199(2)	–0.0122(3)
O(3)	8*f*	–0.1453(2)	1.1214(3)	0.1008(2)	0.055(2)	0.032(2)	0.065(2)	0.004(1)	0.034(1)	0.004(1)
N(1)	8*f*	–0.0741(2)	0.9045(4)	0.1067(2)	0.037(2)	0.029(2)	0.058(2)	–0.002(1)	0.021(1)	–0.005(2)
C(7)	8*f*	0.0226(2)	0.9420(4)	0.1222(2)	0.036(2)	0.030(2)	0.039(2)	–0.002(1)	0.017(2)	0.001(2)
C(5)	8*f*	0.1612(3)	1.0979(5)	0.1855(3)	0.049(2)	0.042(2)	0.043(2)	–0.013(2)	0.018(2)	–0.009(2)
C(3)	8*f*	0.1770(3)	0.8729(4)	0.1190(2)	0.035(2)	0.035(2)	0.039(2)	–0.001(2)	0.015(2)	0.000(2)
C(8)	8*f*	–0.1494(3)	0.9919(4)	0.0994(2)	0.041(2)	0.037(2)	0.040(2)	0.002(2)	0.020(2)	–0.001(2)
C(4)	8*f*	0.2169(3)	0.9981(4)	0.1640(3)	0.034(2)	0.044(2)	0.043(2)	–0.011(2)	0.015(2)	–0.001(2)
C(2)	8*f*	0.0788(2)	0.8438(4)	0.0968(2)	0.034(2)	0.029(2)	0.035(2)	–0.002(1)	0.014(1)	0.003(1)
C(6)	8*f*	0.0638(3)	1.0700(4)	0.1643(3)	0.047(2)	0.036(2)	0.048(2)	–0.002(2)	0.024(2)	–0.007(2)
C(9)	8*f*	–0.2410(3)	0.9127(5)	0.0877(3)	0.041(2)	0.045(2)	0.079(3)	0.003(2)	0.026(2)	–0.004(2)
O(1)	8*f*	–0.0204(2)	0.6376(3)	0.0739(2)	0.044(2)	0.034(1)	0.074(2)	–0.011(1)	0.036(1)	–0.007(1)
O(2)	8*f*	0.0687(2)	0.6595(3)	–0.0141(2)	0.070(2)	0.051(2)	0.083(2)	–0.007(2)	0.037(2)	–0.011(2)
C(1)	8*f*	0.0377(2)	0.7049(4)	0.0513(2)	0.031(2)	0.028(2)	0.043(2)	0.003(1)	0.016(2)	–0.000(2)

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