Crystal structure of 1-(carboxymethyl)-2-methyl-1,3-benzimidazol-3-ium-3-acetate hydrate, $[C_{10}H_9N_2O_2][CH_3CO_2] \cdot H_2O$

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Abstract

$C_{12}H_{14}N_2O_5$, triclinic, $P\bar{1}$ (no. 2), $a = 8.117(1)$ Å, $b = 8.848(2)$ Å, $c = 9.675(2)$ Å, $\alpha = 89.020(2)^\circ$, $\beta = 72.824(2)^\circ$, $\gamma = 65.766(2)^\circ$, $V = 601.0$ Å³, $Z = 2$, $R_g(F) = 0.038$, $wR_{ref}(F^2) = 0.098$, $T = 173$ K.

Source of material

2-Methyl-1*H*-benzimidazole (3.96 g, 0.03 mol) and chloroacetic acid (7.2 g, 0.075 mol) were dissolved in water (70 ml). The pH of the solution was kept to 8 by the addition of NaOH for 4 h. The solution was cooled to room temperature, then the pH was adjusted to 2–3 by addition of hydrochloric acid and a white precipitate began to form. The precipitate was filtered off and recrystallized from water (yield 74 %).

Experimental details

H atoms attached to O atoms were located in a difference Fourier map, giving O—H distances in the range 0.89–0.95 Å. The other H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of $d(C_{sp}-H) = 0.95$ Å with $U_{iso}(H) = 1.2 U_{eq}(\text{parent atom})$, $d(C_{sp^2}-H) = 0.95$ Å with $U_{iso}(H) = 1.2 U_{eq}(\text{parent atom})$, $d(C_{sp^3}-H) = 0.99$ Å for $-CH_2-$ and 0.98 Å for $-CH_3$ with $U_{iso}(H) = 1.5 U_{eq}(\text{parent atom})$.

Discussion

Imidazole and benzimidazole carboxylic acids have gained great interest for their functional properties of antifilarial, antineoplastic, anthelmintic and antiviral activities, and lead to their widely use in the design of therapeutic agents, such as antifilarial and antineoplastic, anthelmintic, antiviral compounds and 5-HT₄ receptor antagonists [1–4]. In addition, these special amino acids are known as good ligands in the supramolecular chemistry, as an N atom in the imidazole ring and the O atoms in the carboxylate groups can be donor sites [5–7]. For example, deprotonated monoanion of bis(1,3-carboxymethyl)imidazole is an interesting aminoacetate anion having two $-CH_2CO_2-$ arms which can each ligate to a metal [8] and thus easy to form frameworks.

The bond lengths within the imidazolium ring, ranging from 1.336(2) to 1.400(2) Å, are similar to the presence of conjugated double bonds of the imidazole ring in 2-methylbenzimidazole [9]. The fact indicates these bond lengths remain essentially unchanged even in zwitterionic structure [10]. The benzimidazolium moiety (C1/N2/C3–C7/C2/N1) is still planar, with mean deviation of 0.005 Å. The two $-CH_2CO_2-$ arms are located on both sides of the plane, respectively (figure, top). In the crystal structure, a strong hydrogen bond involving the hydroxy O atom (O3) leads to formation of infinite one-dimensional chains along the [010] direction. In addition, the water molecules act as hydrogen-bond donors to carboxylate O atoms and link adjacent chain to form a two-dimensional layers parallel to (100), while non-classical C—H...O hydrogen bond creates a three-dimensional framework (figure, bottom).

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Table 1. Data collection and handling.

Crystal:	colorless block, size 0.20 × 0.30 × 0.50 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.16 cm ⁻¹
Diffractometer, scan mode:	Bruker AXS SMART APEX II CCD, φ/ω
$2\theta_{\max}$:	51.98°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	4716, 2317
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1715
$N(\text{param})_{\text{refined}}$:	173
Programs:	SHELXTL [11]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(4)	2i	-0.0772	0.8104	0.5366	0.030
H(5)	2i	0.0483	0.6831	0.2952	0.034
H(6)	2i	0.3152	0.4291	0.2227	0.034
H(7)	2i	0.4710	0.2897	0.3881	0.031
H(8A)	2i	0.3187	0.5900	0.9533	0.049
H(8B)	2i	0.2887	0.4219	0.9671	0.049
H(8C)	2i	0.1085	0.5995	1.0158	0.049
H(9A)	2i	0.5520	0.2826	0.7633	0.029
H(9B)	2i	0.6016	0.2360	0.5926	0.029
H(11A)	2i	-0.0942	0.8013	0.9408	0.029
H(11B)	2i	-0.1686	0.8557	0.8041	0.029
H(3)	2i	0.2123	1.0064	0.7576	0.037
H(1A)	2i	0.6399	0.1491	-0.0151	0.050
H(1B)	2i	0.5003	0.1273	0.1134	0.050

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
C(1)	2i	0.2223(2)	0.5403(2)	0.7962(2)	0.0226(8)	0.0153(8)	0.029(1)	-0.0108(7)	-0.0080(7)	0.0039(7)
C(2)	2i	0.2872(2)	0.4712(2)	0.5596(2)	0.0211(8)	0.0171(8)	0.027(1)	-0.0107(7)	-0.0073(7)	0.0037(7)
C(3)	2i	0.1278(2)	0.6241(2)	0.6031(2)	0.0197(8)	0.0147(8)	0.0263(9)	-0.0088(7)	-0.0052(7)	0.0017(7)
C(4)	2i	0.0327(2)	0.7071(2)	0.5067(2)	0.0226(8)	0.0199(9)	0.032(1)	-0.0095(7)	-0.0085(7)	0.0054(7)
C(5)	2i	0.1077(2)	0.6304(2)	0.3650(2)	0.032(1)	0.030(1)	0.032(1)	-0.0183(8)	-0.0148(8)	0.0107(8)
C(6)	2i	0.2686(2)	0.4773(2)	0.3213(2)	0.032(1)	0.030(1)	0.026(1)	-0.0182(8)	-0.0073(8)	0.0014(8)
C(7)	2i	0.3621(2)	0.3938(2)	0.4176(2)	0.0225(8)	0.0195(8)	0.033(1)	-0.0102(7)	-0.0046(7)	-0.0015(7)
C(8)	2i	0.2357(3)	0.5377(2)	0.9458(2)	0.043(1)	0.025(1)	0.033(1)	-0.0131(9)	-0.0162(9)	0.0048(8)
C(9)	2i	0.4994(2)	0.2662(2)	0.6876(2)	0.0193(8)	0.0159(8)	0.037(1)	-0.0057(7)	-0.0104(7)	0.0046(7)
C(10)	2i	0.4429(2)	0.1211(2)	0.7191(2)	0.0244(9)	0.0145(9)	0.031(1)	-0.0065(7)	-0.0108(7)	0.0014(7)
C(11)	2i	-0.0553(2)	0.8200(2)	0.8373(2)	0.0207(8)	0.0159(8)	0.029(1)	-0.0058(7)	-0.0032(7)	0.0000(7)
C(12)	2i	0.0097(2)	0.9602(2)	0.8263(2)	0.0229(8)	0.0170(8)	0.0242(9)	-0.0065(7)	-0.0068(7)	0.0023(7)
N(1)	2i	0.3421(2)	0.4237(2)	0.6836(1)	0.0204(7)	0.0122(7)	0.0294(8)	-0.0064(6)	-0.0092(6)	0.0031(6)
N(2)	2i	0.0905(2)	0.6621(2)	0.7516(1)	0.0208(7)	0.0137(7)	0.0255(8)	-0.0069(6)	-0.0055(6)	0.0015(6)
O(1)	2i	0.5719(2)	-0.0190(1)	0.7046(1)	0.0277(7)	0.0149(6)	0.0509(8)	-0.0045(5)	-0.0126(6)	0.0039(6)
O(2)	2i	0.2641(2)	0.1598(1)	0.7619(1)	0.0228(6)	0.0177(6)	0.0570(9)	-0.0097(5)	-0.0116(6)	0.0046(6)
O(3)	2i	0.1849(2)	0.9196(1)	0.7542(1)	0.0245(6)	0.0171(6)	0.0474(8)	-0.0111(5)	-0.0029(6)	-0.0017(5)
O(4)	2i	-0.1088(2)	1.0995(1)	0.8873(1)	0.0296(7)	0.0167(6)	0.0375(8)	-0.0069(5)	-0.0016(5)	-0.0021(5)
O(1W)	2i	0.5081(2)	0.1858(2)	0.0329(1)	0.0314(7)	0.0385(8)	0.0433(8)	-0.0066(6)	-0.0088(6)	0.0141(6)

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