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1-(Carboxymethyl)-1,3-benzimidazol-3-ium-3-acetate

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The title compound, $C_{11}H_{10}N_2O_4$, was synthesized from benzimidazole and chloroacetic acid. In the crystal structure, O—H···O hydrogen bonds lead to the formation of extended one-dimensional chains along the [101] direction.

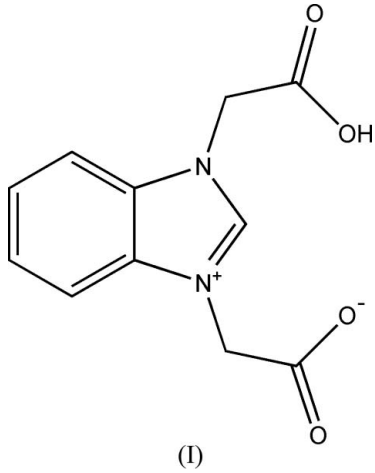
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.038
wR factor = 0.104
Data-to-parameter ratio = 12.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Benzimidazole carboxylic acids are important pharmaceutical intermediates, and they are widely used in the design of therapeutic agents, such as antifilarial and antineoplastic (Ram *et al.*, 1992), anthelmintic (Dubey *et al.*, 1985) and antiviral compounds (Garuti *et al.*, 2000) as well as 5-HT4 receptor antagonists (Maria *et al.*, 1999). These types of acids are good ligands which are capable of forming supramolecular metal complexes, as an N atom in the benzimidazole ring and the O atoms in the carboxylate group can act as donor sites. Some polymer complexes derived from benzimidazole-2(5)-carboxylic acid have been reported in the literature (Rettig *et al.*, 1999; Liu *et al.*, 2005; Deng *et al.*, 2006). In an extension of this research, we have synthesized the title compound (I), and determined its crystal structure.



(I)

The molecular structure of (I) is shown in Fig. 1 and selected bond lengths and angles are given in Table 1. In the imidazolium ring, the bond lengths range from 1.3322 (16) to 1.3947 (16) Å, in good agreement with the presence of conjugated double bonds (Dik-Edixhoven *et al.*, 1973); these bond lengths remain essentially unchanged in a zwitterionic structure (Fei *et al.*, 2004). The benzimidazolium unit (C1/N1/C2–C7/N2) is essentially planar, with a mean deviation of 0.011 Å. The C7—N2—C8—C9 and C1—N1—C10—C11 torsion angles are −123.91 (13) and 100.56 (15)°, respectively.

In the crystal structure, a hydrogen bond involving the hydroxy O atom (O4) leads to the formation of extended one-

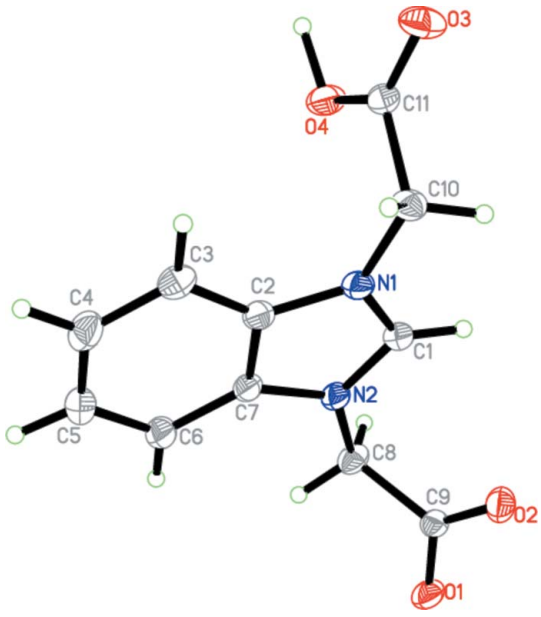


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

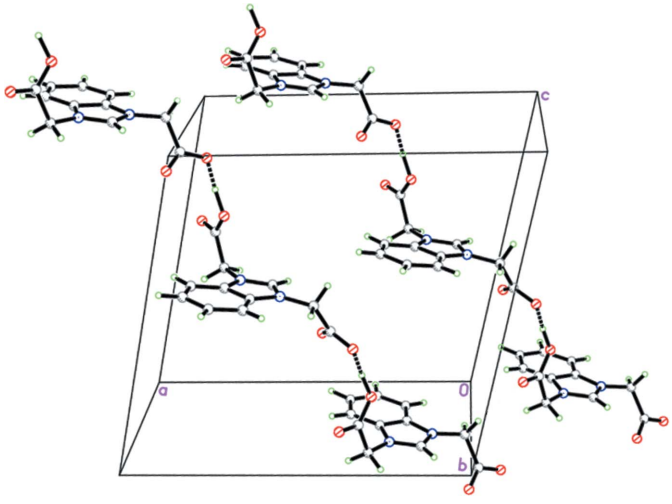


Figure 2
Part of the crystal structure of (I). Dashed lines indicate hydrogen bonds.

dimensional chains along the [101] direction (Table 2 and Fig. 2).

Experimental

Benzimidazole (8.90 g, 75.4 mmol) and chloroacetic acid (14.2 g, 150.0 mmol) were dissolved in water (150 ml). The pH of the solution was adjusted to 8–10 by the addition of NaOH. The solution was refluxed and the pH adjusted as above at 15 min intervals. After approximately 4 h, the pH no longer changed. 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 88%; m.p. 573.4–573.5 K).

Crystal data

$C_{11}H_{10}N_2O_4$
M_r = 234.21
Monoclinic, $C2/c$
a = 14.9168 (17) Å
b = 9.8014 (11) Å
c = 14.3344 (16) Å
β = 102.283 (1)°
V = 2047.8 (4) Å^3
Z = 8
D_x = 1.519 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 293 (2) K
Block, colorless
0.39 × 0.28 × 0.15 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.961, T_{max} = 0.982
8976 measured reflections
2512 independent reflections
1991 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 28.3°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.104
S = 1.05
2512 reflections
194 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.051P)^2 + 0.7851P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C9	1.2853 (15)	N1—C10	1.4632 (16)
O2—C9	1.2256 (15)	N2—C1	1.3322 (16)
O3—C11	1.2200 (15)	N2—C7	1.3990 (15)
O4—C11	1.2816 (16)	N2—C8	1.4667 (16)
N1—C1	1.3358 (17)	C2—C7	1.3920 (16)
N1—C2	1.3947 (16)		
C1—N1—C2	108.38 (10)	C7—C2—N1	106.58 (11)
C1—N2—C7	108.31 (10)	C2—C7—N2	106.52 (11)
N2—C1—N1	110.19 (11)	O3—C11—O4	126.84 (12)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O4—H11···O1^{i}	1.13 (2)	1.32 (2)	2.4521 (13)	176 (2)
O4—H11···O2^{i}	1.13 (2)	2.56 (2)	3.2968 (14)	121.2 (15)

Symmetry code: (i) $x-\frac{1}{2}, -y+\frac{3}{2}, z-\frac{1}{2}$.

All H atoms were located in a difference Fourier map and refined isotropically, giving C—H distances in the range 0.962 (16)–0.998 (17) Å and O—H = 1.13 (2) Å.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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