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2-(1*H*-Benzimidazol-2-ylsulfanyl)butanoic acid

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.002 Å
R factor = 0.043
wR factor = 0.122
Data-to-parameter ratio = 26.8

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

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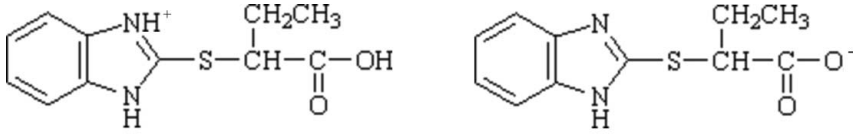
The title compound, $C_{11}H_{12}N_2O_2S$, forms ionic crystals consisting of protonated benzimidazole cations and carboxylic acid anions, $C_{11}H_{13}N_2O_2^+ \cdot C_{11}H_{11}N_2O_2^-$. In both ions, the bicyclic 1*H*-benzimidazole core is essentially planar. The plane of the 2-alkylsulfanyl substituent is almost coplanar with the benzimidazole plane. The three-dimensional packing is stabilized by strong intermolecular N—H···O and O—H···N interactions and weak C—H···O intermolecular hydrogen bonds.

Comment

It is known that benzimidazoles exhibit a broad spectrum of biological activity of both human and veterinary importance (antiparasitic, antiviral, antimicrobial, fungicidal, herbicidal and insecticidal activity). A wide variety of benzimidazole derivatives have been described for their chemotherapeutic effects (Boruah & Skibo, 1994; Huang *et al.*, 2000; El-masry *et al.*, 2000). Owing to the presence of benzimidazole systems in a large number of common therapeutic agents (*e.g.* the spasmolytic and hypotensive drug Bendazol), such systems are widely used in organic and medicinal chemistry (Boiani & Gonzalez, 2005). 2-Alkylsulfanyl derivatives having a carboxyl functionality at the C atom adjacent to the S atom are, moreover, useful intermediates for cyclization to the corresponding 2-alkylbenzo[*d*]imidazo[2,1-*b*]thiazolidin-2-ones (Kooš, 1994; Jain & Pujari, 1981; Gupta *et al.*, 1979). In this context, we have synthesized several starting 2-(benzimidazol-2-ylsulfanyl)alkanoic acids, among them (I), and we report here its crystal structure.



(I)

The structure of (I) is illustrated in Fig. 1. Two independent species in the asymmetric unit are actually a cation *A*, in which the second imidazole N atom is protonated, and an anion *B*, in which the carboxylic acid group is deprotonated. The two adjacent ions are bound into a neutral unit by hydrogen bonds. The three-dimensional crystal packing is stabilized by strong intermolecular hydrogen bonding of the O—H···N and N—H···O types involving atoms O1*A*, N1*A* and N3*A* as donors, and atoms N3*B* and O1*B* as acceptors. Atoms C2*B* act as donors and atoms O2*A* as acceptors for weak intermolecular C—H···O interactions. There are also weak intramolecular

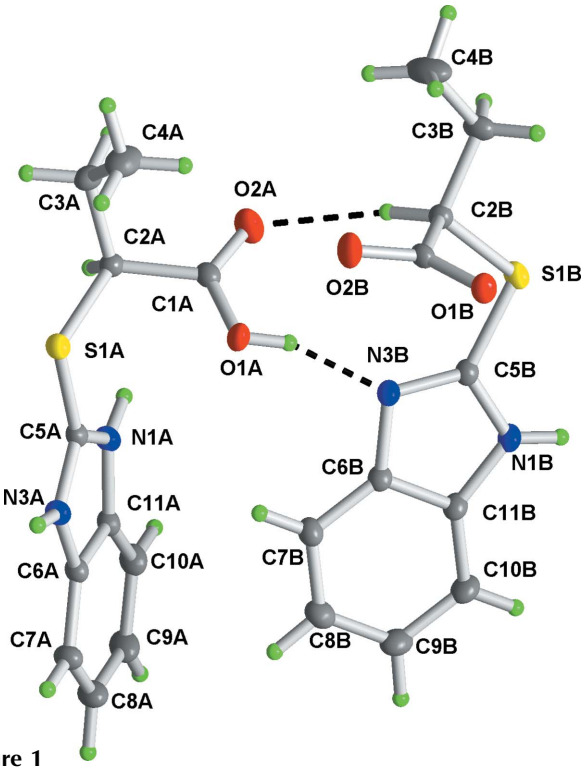


Figure 1
A perspective drawing of (I), showing the atom-numbering scheme. Atomic displacement ellipsoids are shown at the 30% probability level. Hydrogen bonds binding the two adjacent ions into a neutral unit are shown as dashed lines.

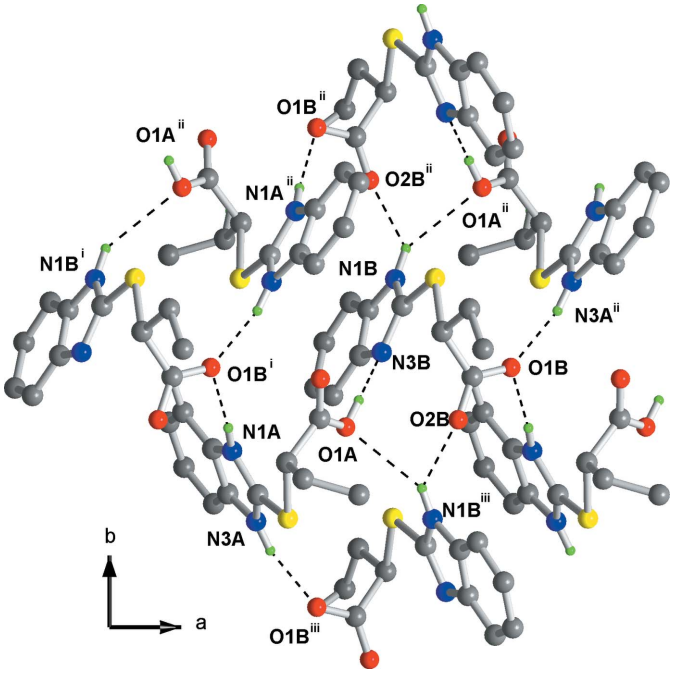


Figure 2
Hydrogen bonding in (I). Hydrogen bonds are shown as dashed lines. H atoms not involved in the strong hydrogen bonds have been omitted for clarity. For symmetry codes see Table 2.

C—H···O and C—H···S interactions with atoms C7*A* and C4*A* as donors and atoms O2*A* and S1*A* atom as acceptors.

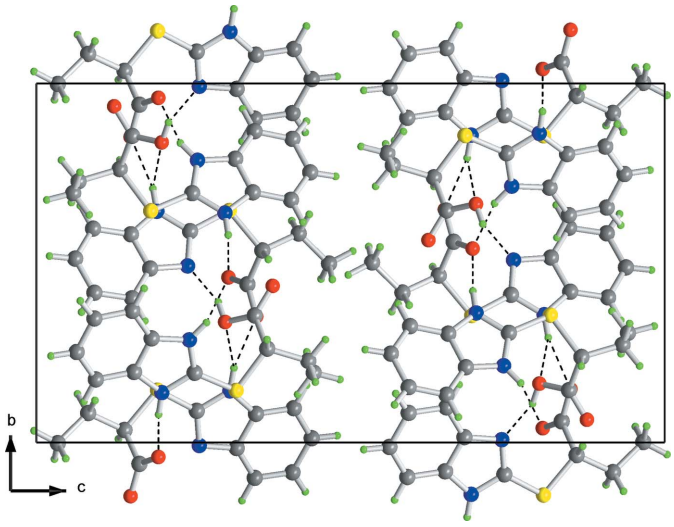


Figure 3
A projection of the unit cell contents of (I) along the *a* axis. Hydrogen bonds are shown as dashed lines.

This situation is illustrated for strong hydrogen bonds in Fig. 2, and more details of the hydrogen bonding are given in Table 1. Interionic hydrogen bonds link the residues in the structure of (I) into infinite layers extending parallel to the *ab* plane of the unit cell (Fig. 3).

Experimental

Compound (I) was prepared from the sodium salt of 2-mercaptobenzimidazole and 2-bromobutanoic acid according to the previously described procedure (Koóš, 1994). Colourless single crystals of adequate quality for diffraction analysis were obtained by slow crystallization from ethanol by cooling in a refrigerator.

Crystal data

$C_{11}H_{12}N_2O_2S$
M_r = 236.29
Monoclinic, $P2_1/c$
a = 7.8750 (1) Å
b = 12.5588 (1) Å
c = 22.0210 (1) Å
β = 93.645 (1)°
V = 2173.48 (3) Å^3
Z = 8
D_x = 1.444 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.28 mm^{-1}
T = 295 (2) K
Plate, colorless
0.80 × 0.34 × 0.16 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.805, T_{max} = 0.956
33031 measured reflections
7839 independent reflections
5889 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 33.1°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.122
S = 1.02
7839 reflections
292 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0601P)^2 + 0.6348P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1A—H1A1···O1B^{i}	0.86	1.83	2.6574 (14)	160
O1A—H1A···N3B	0.82	1.79	2.5757 (14)	160
N1B—H1B···O1A^{ii}	0.86	2.57	3.3137 (17)	145
N1B—H1B···O2B^{ii}	0.86	2.16	2.8252 (14)	134
N3A—H3A···O1B^{iii}	0.86	1.93	2.7658 (14)	165
C2A—H2A···O2B^{i}	0.98	2.58	3.5550 (18)	172
C2B—H2B···O2A	0.98	2.50	3.4676 (18)	170
C4A—H4A1···S1A	0.96	2.86	3.2565 (18)	106
C7A—H7A···O2A^{iv}	0.93	2.52	3.0843 (19)	119

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$; (iii) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$; (iv) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$.

The H atoms were constrained to an ideal geometry using an appropriate riding model. For the methyl groups, the C—H distances (0.96 Å) and C—C—H angles (109.5°) were kept fixed, while the torsion angles were allowed to refine with the starting position based on the threefold averaged circular Fourier synthesis. For other H atoms, C—H = 0.93–0.98 Å, N—H = 0.86 Å and O—H = 0.82 Å; U_{iso}(H) = 1.2U_{eq}(C,N) or 1.5U_{eq}(C_{methyl},O).

Data collection: *SMART* (Siemens, 1995); cell refinement: *SAINT* (Siemens, 1995); data reduction: *SAINT* and *SADABS* (Sheldrick, 2002); program(s) used to solve structure: *SHELXTL* (Bruker, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *SHELXTL*.

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