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3-Isopropyl-1-[2-(pyrrolidinium-1-yl)-ethyl]benzimidazolium diodide

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.005 Å
R factor = 0.030
wR factor = 0.066
Data-to-parameter ratio = 24.2

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

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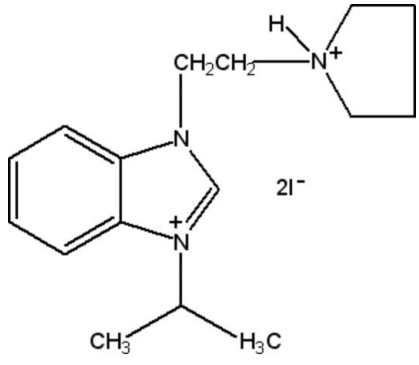
In the molecule of the title compound, $C_{16}H_{25}N_3^{2+}\cdot 2I^-$, the pyrrolidine ring has an envelope conformation. The crystal structure has been determined at room temperature and exhibits intermolecular N—H···I and C—H···I interactions.

Comment

There is a considerable interest in the pharmacology of heterocyclic compounds and their derivatives (Mishra & Yadaw, 2000). In particular, benzimidazole ring systems are an important pharmacophore in modern drug discovery (Tebbe *et al.*, 1997). Benzimidazole derivatives generally exhibit versatile pharmacological activity, such as antitumour, diuretic, fungicidal, bactericidal, anthelmintic, anti-allergic, vasodilator, antihistaminic, anti-ulcer and local analgesic properties. We have also reported the syntheses and antimicrobial activities of many benzimidazole derivatives (Küçükbay *et al.*, 2003, 2004; Küçükbay & Durmaz, 1997). The objective of this study was to synthesize and elucidate the crystal structure of a new benzimidazole compound having a pyrrolidine ring.



(I)

The molecular structure of (I) is shown in Fig. 1. The geometric parameters of (I) are in good agreement with the results obtained in our previous studies of related benzimidazole derivatives (Akkurt, Yıldırım *et al.*., 2005; Yıldırım *et al.*, 2005; Akkurt, Türktekin *et al.*, 2005). The nine-membered benzimidazole ring system (N1/N2/C1–C7) of (I) is essentially planar, the maximum deviation from planarity being 0.038 (2) Å for atom C1. The conformation of the five-membered pyrrolidine ring (N3/C13–C16) is close to an envelope, with N3 as flap atom; the puckering parameters are $Q(2) = 0.310$ (4) Å and $\varphi(2) = 6.2$ (9)° (Cremer & Pople, 1975). The details of the C—H···I and N—H···I hydrogen-bonding interactions are given in Table 1 (Fig. 2).

Experimental

To a solution of 1-(2-pyrrolidinium-1-yl)ethylbenzimidazole iodide (2.86 g, 8.33 mmol) in dimethylformamide (5 ml) was added isopropyl iodide (1.42 g, 8.33 mmol) and the mixture was heated under reflux for 8 h. The mixture was then cooled and the solvent was removed *in vacuo*. The residue was crystallized from EtOH (15 ml) (yield 3.50 g, 82%; m.p. 494–495 K). ^{1}H NMR (DMSO-d_6): δ 1.67 (*d*, CH_3, 6H), 1.87 (*m*, ring methylene, 2H), 2.08 (*t*, ring methylene, 2H), 3.24 (*m*, ring methylene, 2H), 3.70 (*t*, ring methylene, 2H), 3.92 (*t*, CH_2CH_2-pyrrolidine, 2H), 4.91 (*t*, CH_2CH_2-pyrrolidine, 2H), 7.74 (*m*, Ar—H, 2H), 8.19 (*m*, Ar—H, 2H), 9.58 (*s*, 2-CH, 1H), 10.01 (*s*, pyrrolidine-1H, 1H). ^{13}C NMR (DMSO-d_6): δ 22.07, 23.16, 43.53, 51.35, 52.08, 54.31, 114.49, 114.64, 127.14, 127.23, 131.03, 131.73, 132.04. Analysis calculated for $C_{16}H_{25}N_3I_2$: C 37.42, H 4.87, N 8.18%; found: C 37.71, H 4.87, N 8.24%.

Crystal data

$C_{16}H_{25}N_3^{2+}\cdot 2I^-$
M_r = 513.19
Monoclinic, $P2_1/c$
a = 11.639 (5) Å
b = 12.945 (5) Å
c = 13.192 (5) Å
β = 94.349 (5)°
V = 1981.9 (14) Å^3
Z = 4
D_x = 1.720 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 47374 reflections
θ = 2.2–28.0°
μ = 3.17 mm^{-1}
T = 296 K
Prism, yellow
0.70 × 0.47 × 0.32 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.181, T_{max} = 0.362
32700 measured reflections
4649 independent reflections
3867 reflections with $I > 2\sigma(I)$
R_{int} = 0.115
θ_{max} = 27.9°
$h = -15 \rightarrow 15$
$k = -16 \rightarrow 16$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.066
S = 1.03
4649 reflections
192 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0183P)^2 + 1.4496P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···I1	0.91	2.52	3.410 (3)	166
C7—H7···I2	0.93	2.98	3.715 (3)	136
C8—H8···I2^{i}	0.98	2.89	3.856 (3)	168
C12—H12A···I1^{ii}	0.97	2.98	3.921 (3)	164

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

All H atoms were placed geometrically in ideal positions and refined with a riding model, with C—H = 0.93–0.98 Å, N—H = 0.91 Å and U_{iso}(H) constrained to be 1.2U_{eq} (1.5U_{eq} for methyl groups) of the parent atom.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

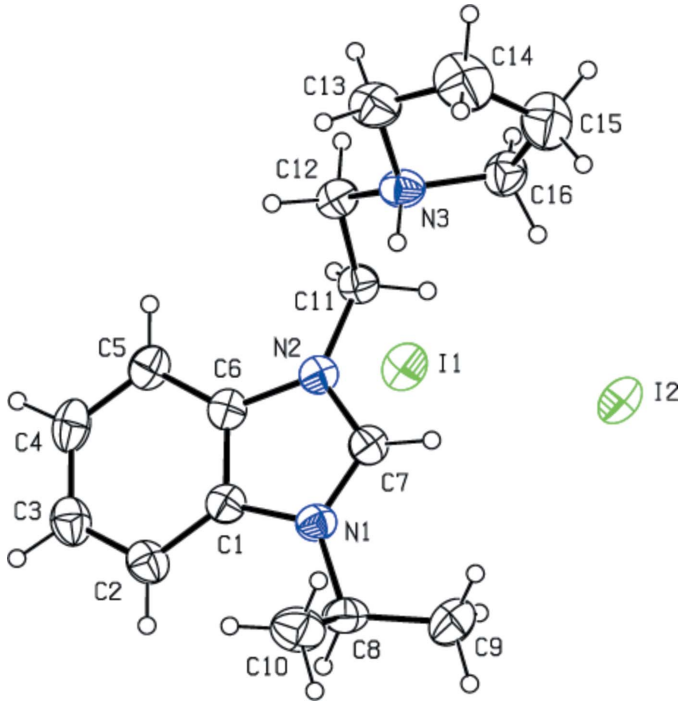


Figure 1
A plot of (I), with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level.

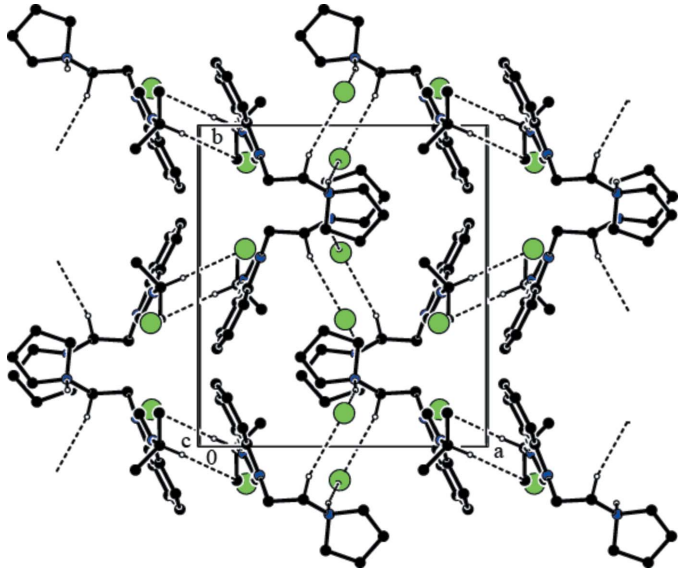


Figure 2
The packing and hydrogen-bonding interactions (dashed lines) of (I), viewed down the *c* axis. H atoms not involved in hydrogen bonding have been omitted.

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