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1-[4-(1*H*-Benzimidazol-1-yl)butyl]-1*H*-benzimidazol-3-ium nitrate

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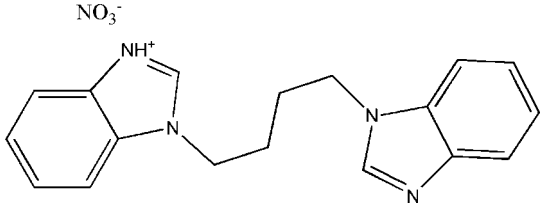
Key indicators: single-crystal X-ray study; T = 153 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.053; wR factor = 0.192; data-to-parameter ratio = 15.2.

In the proton-transfer title compound, $C_{18}H_{19}N_4^+\cdot NO_3^-$, the 1-[4-(1*H*-benzimidazol-1-yl)butyl]-1*H*-benzimidazolium cation is entrosymmetric with disorder of the N-bound H atoms, and the nitrate anion lies on a twofold rotation axis. The bond lengths and angles are within normal ranges. The crystal packing is stabilized by intermolecular N—H···N and C—H···O hydrogen bonds, which link the molecules into infinite ribbons running along the *a* axis, and by weak π–π stacking interactions [centroid–centroid distance 3.638 (2) Å].

Related literature

For a related crystal structure, see: Niu *et al.* (2005). For applications of benzimidazole derivatives, see: Gudmundsson *et al.* (2000); Payra *et al.* (2001).



Experimental

Crystal data

$C_{18}H_{19}N_4^+\cdot NO_3^-$
M_r = 353.38
Monoclinic, $C2/c$
a = 21.176 (4) Å
b = 7.317 (2) Å
c = 13.330 (3) Å
β = 122.20 (3)°
V = 1747.7 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 153 (2) K
0.27 × 0.20 × 0.15 mm

Data collection

Rigaku R-AXIS RAPID IP area-detector diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.975, T_{max} = 0.986
8300 measured reflections
2001 independent reflections
1413 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.192
S = 1.19
2001 reflections
132 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···N2^{i}	0.90 (3)	1.78 (3)	2.674 (3)	173 (4)
C7—H7A···O1^{ii}	0.95	2.31	3.221 (3)	162

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

Data collection: *RAPID-AUTO* (Rigaku, 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2334).

References

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supplementary materials

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1-[4-(1*H*-Benzimidazol-1-yl)butyl]-1*H*-benzimidazol-3-ium nitrate

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Comment

Recently, benzimidazole derivatives have been the focus of increasing attention due to their applications, such as antivirals (Gudmundsson *et al.*, 2000) and ligands (Payra *et al.*, 2001). In this paper, we report the crystal structure of the title compound, (I).

In (I) (Fig. 1), all bond lengths and angles are normal and in a good agreement with those reported previously (Niu *et al.*, 2005). The relatively short distance of 3.638 (2)Å between the centroids of benzene ring C1–C6 and imidazole ring N1/N2/C1/C6/C7 [at (1/2-*X*,-1/2+Y,1/2-*Z*)] indicates the presence of weak π-π interactions, which contribute to the stability of the crystal packing. Intermolecular N—H···N and C—H···O hydrogen bonds, which link the molecules into infinite ribbons running along the *a* axis.

Experimental

A mixture of benzimidazole 2.36 g (0.02 mol), butane-1,4-diol 1.8 g (0.02 mol) and potassium carbonate 1.38 g (0.01 mol) was stirred in refluxing acetone (10 ml) for 5 h at 327 K to afford the compound 1-(4-(1*H*-benzimidazol-1-yl)butyl)-1*H*-benzimidazole 4.99 g (yield 86%). Single crystals suitable for X-ray measurements were obtained by recrystallization from diluted nitric acid (10%) at room temperature.

Refinement

All H atoms were found on difference maps. C-bound H atoms were placed in calculated positions (C—H 0.95 or 0.99 Å) and refined as riding, with U_{iso}(H)= 1.2U_{eq}(C). Atoms H2A was refined over two positions, giving an N—H bond distance of 0.90 (3) Å. (occupancies 0.500 for the primed and 0.500 for the unprimed atoms).

Figures

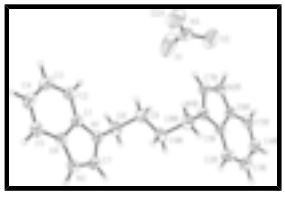

Fig. 1. The molecular structure of (I), with atom labels and 40% probability displacement ellipsoids for non-H atoms. The symmetry code for the 'B' atoms in the molecule is −*x*,1 − *y*,-*z*.

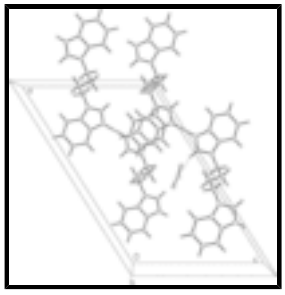

Fig. 2. A packing diagram for the title compound (I), viewed along the *b* axis of the cell.

1-[4-(1*H*-Benzimidazol-1-yl)butyl]-1*H*-benzimidazol-3-ium nitrate

Crystal data

$C_{18}H_{19}N_4^+ \cdot NO_3^-$	F_{000} = 744
M_r = 353.38	D_x = 1.343 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 4407 reflections
a = 21.176 (4) Å	θ = 2.9–26.4º
b = 7.317 (2) Å	μ = 0.10 mm^{-1}
c = 13.330 (3) Å	*T* = 153 (2) K
β = 122.20 (3)º	Block, colorless
V = 1747.7 (9) Å^3	0.27 × 0.20 × 0.15 mm
Z = 4	

Data collection

Rigaku R-AXIS RAPID IP area-detector diffractometer	2001 independent reflections
Radiation source: Rotating Anode	1413 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.025
T = 153(2) K	θ_{max} = 27.5º
ω Oscillation scans	θ_{min} = 3.0º
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	*h* = −27→27
T_{min} = 0.975, T_{max} = 0.986	*k* = −9→9
8300 measured reflections	*l* = −17→17

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.053	$w = 1/[\sigma^2(F_o^2) + (0.1007P)^2 + 0.5324P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.192	$(\Delta/\sigma)_{max} < 0.001$

S = 1.19	$\Delta\rho_{max}$ = 0.47 e Å^{-3}
2001 reflections	$\Delta\rho_{min}$ = −0.38 e Å^{-3}
132 parameters	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.021 (3)
Secondary atom site location: difference Fourier map	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.0000	0.8204 (5)	0.2500	0.1393 (18)	
O2	−0.05586 (11)	1.0720 (3)	0.17563 (19)	0.0984 (8)	
N1	0.12401 (8)	0.2918 (2)	0.08925 (14)	0.0443 (5)	
N2	0.21483 (9)	0.2962 (3)	0.05499 (15)	0.0473 (5)	
H2A	0.239 (2)	0.274 (6)	0.018 (4)	0.039 (11)*	0.50
N3	0.0000	0.9904 (4)	0.2500	0.0552 (7)	
C1	0.18964 (10)	0.3164 (3)	0.19839 (17)	0.0412 (5)	
C2	0.20375 (13)	0.3321 (3)	0.3119 (2)	0.0519 (5)	
H2B	0.1647	0.3315	0.3270	0.062*	
C3	0.27768 (13)	0.3485 (3)	0.4022 (2)	0.0589 (6)	
H3A	0.2899	0.3580	0.4817	0.071*	
C4	0.33497 (12)	0.3515 (3)	0.3795 (2)	0.0576 (6)	
H4A	0.3851	0.3644	0.4441	0.069*	
C5	0.32102 (11)	0.3363 (3)	0.2672 (2)	0.0512 (5)	
H5A	0.3603	0.3375	0.2525	0.061*	
C6	0.24670 (10)	0.3189 (3)	0.17511 (17)	0.0415 (5)	
C7	0.14243 (11)	0.2789 (3)	0.00758 (18)	0.0474 (5)	
H7A	0.1075	0.2597	−0.0745	0.057*	
C8	0.04942 (11)	0.2681 (3)	0.0685 (2)	0.0541 (6)	
H8A	0.0534	0.1987	0.1354	0.065*	
H8B	0.0188	0.1952	−0.0045	0.065*	
C9	0.01042 (11)	0.4484 (3)	0.0559 (2)	0.0510 (6)	
H9B	0.0440 (13)	0.526 (3)	0.129 (2)	0.061 (7)*	
H9A	−0.0352 (14)	0.414 (3)	0.056 (2)	0.059 (6)*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.108 (3)	0.091 (2)	0.098 (3)	0.000	−0.026 (2)	0.000
O2	0.0685 (12)	0.1104 (19)	0.0745 (14)	0.0185 (11)	0.0101 (10)	0.0123 (12)
N1	0.0304 (8)	0.0549 (10)	0.0445 (9)	0.0041 (6)	0.0179 (7)	0.0027 (7)
N2	0.0388 (8)	0.0616 (11)	0.0429 (9)	0.0025 (7)	0.0227 (7)	−0.0018 (7)
N3	0.0416 (12)	0.0805 (19)	0.0372 (12)	0.000	0.0167 (10)	0.000
C1	0.0355 (9)	0.0464 (10)	0.0394 (10)	0.0030 (7)	0.0184 (8)	0.0028 (7)
C2	0.0549 (12)	0.0575 (12)	0.0472 (12)	0.0022 (9)	0.0297 (10)	0.0041 (9)
C3	0.0668 (14)	0.0602 (13)	0.0393 (11)	−0.0004 (10)	0.0212 (10)	0.0017 (9)
C4	0.0445 (11)	0.0567 (12)	0.0472 (12)	−0.0012 (9)	0.0080 (9)	0.0012 (9)
C5	0.0347 (9)	0.0557 (12)	0.0547 (13)	0.0006 (8)	0.0181 (9)	−0.0007 (9)
C6	0.0361 (9)	0.0440 (10)	0.0405 (10)	0.0016 (7)	0.0179 (8)	0.0000 (7)
C7	0.0379 (9)	0.0606 (12)	0.0391 (10)	0.0024 (8)	0.0175 (8)	−0.0005 (8)
C8	0.0352 (10)	0.0609 (13)	0.0647 (13)	−0.0009 (8)	0.0257 (9)	0.0048 (10)
C9	0.0369 (9)	0.0633 (13)	0.0547 (12)	0.0027 (9)	0.0256 (9)	0.0002 (10)

Geometric parameters (Å, °)

O1—N3	1.244 (4)	C3—C4	1.398 (3)
O2—N3	1.220 (2)	C3—H3A	0.9500
N1—C7	1.341 (3)	C4—C5	1.368 (3)
N1—C1	1.388 (2)	C4—H4A	0.9500
N1—C8	1.462 (2)	C5—C6	1.393 (3)
N2—C7	1.317 (3)	C5—H5A	0.9500
N2—C6	1.377 (3)	C7—H7A	0.9500
N2—H2A	0.90 (3)	C8—C9	1.517 (3)
N3—O2^{i}	1.220 (2)	C8—H8A	0.9900
C1—C2	1.382 (3)	C8—H8B	0.9900
C1—C6	1.399 (2)	C9—C9^{ii}	1.511 (4)
C2—C3	1.380 (3)	C9—H9B	1.02 (3)
C2—H2B	0.9500	C9—H9A	1.00 (2)
C7—N1—C1	107.35 (16)	C4—C5—C6	117.08 (19)
C7—N1—C8	126.21 (18)	C4—C5—H5A	121.5
C1—N1—C8	126.24 (17)	C6—C5—H5A	121.5
C7—N2—C6	107.08 (16)	N2—C6—C5	131.23 (18)
C7—N2—H2A	126 (3)	N2—C6—C1	108.08 (16)
C6—N2—H2A	126 (3)	C5—C6—C1	120.66 (19)
O2^{i}—N3—O2	121.4 (3)	N2—C7—N1	111.81 (18)
O2^{i}—N3—O1	119.30 (17)	N2—C7—H7A	124.1
O2—N3—O1	119.30 (17)	N1—C7—H7A	124.1
C2—C1—N1	132.07 (18)	N1—C8—C9	112.78 (17)
C2—C1—C6	122.24 (18)	N1—C8—H8A	109.0
N1—C1—C6	105.67 (16)	C9—C8—H8A	109.0
C3—C2—C1	116.4 (2)	N1—C8—H8B	109.0
C3—C2—H2B	121.8	C9—C8—H8B	109.0

C1—C2—H2B	121.8	H8A—C8—H8B	107.8
C2—C3—C4	121.7 (2)	C9^{ii}—C9—C8	113.6 (2)
C2—C3—H3A	119.2	C9^{ii}—C9—H9B	110.8 (14)
C4—C3—H3A	119.2	C8—C9—H9B	108.4 (13)
C5—C4—C3	122.0 (2)	C9^{ii}—C9—H9A	110.7 (14)
C5—C4—H4A	119.0	C8—C9—H9A	104.7 (13)
C3—C4—H4A	119.0	H9B—C9—H9A	108 (2)
C7—N1—C1—C2	−177.8 (2)	C4—C5—C6—C1	−0.3 (3)
C8—N1—C1—C2	−2.6 (3)	C2—C1—C6—N2	178.54 (18)
C7—N1—C1—C6	0.6 (2)	N1—C1—C6—N2	0.0 (2)
C8—N1—C1—C6	175.69 (17)	C2—C1—C6—C5	0.5 (3)
N1—C1—C2—C3	177.4 (2)	N1—C1—C6—C5	−178.08 (17)
C6—C1—C2—C3	−0.7 (3)	C6—N2—C7—N1	1.0 (2)
C1—C2—C3—C4	0.8 (3)	C1—N1—C7—N2	−1.0 (2)
C2—C3—C4—C5	−0.7 (3)	C8—N1—C7—N2	−176.11 (18)
C3—C4—C5—C6	0.5 (3)	C7—N1—C8—C9	−99.1 (2)
C7—N2—C6—C5	177.2 (2)	C1—N1—C8—C9	86.6 (2)
C7—N2—C6—C1	−0.6 (2)	N1—C8—C9—C9^{ii}	68.5 (3)
C4—C5—C6—N2	−177.9 (2)		

Symmetry codes: (i) −*x*, *y*, −*z*+1/2; (ii) −*x*, −*y*+1, −*z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···N2^{iii}	0.90 (3)	1.78 (3)	2.674 (3)	173 (4)
C7—H7A···O1^{iv}	0.95	2.31	3.221 (3)	162

Symmetry codes: (iii) −*x*+1/2, −*y*+1/2, −*z*; (iv) *x*, −*y*+1, *z*−1/2.

Fig. 1

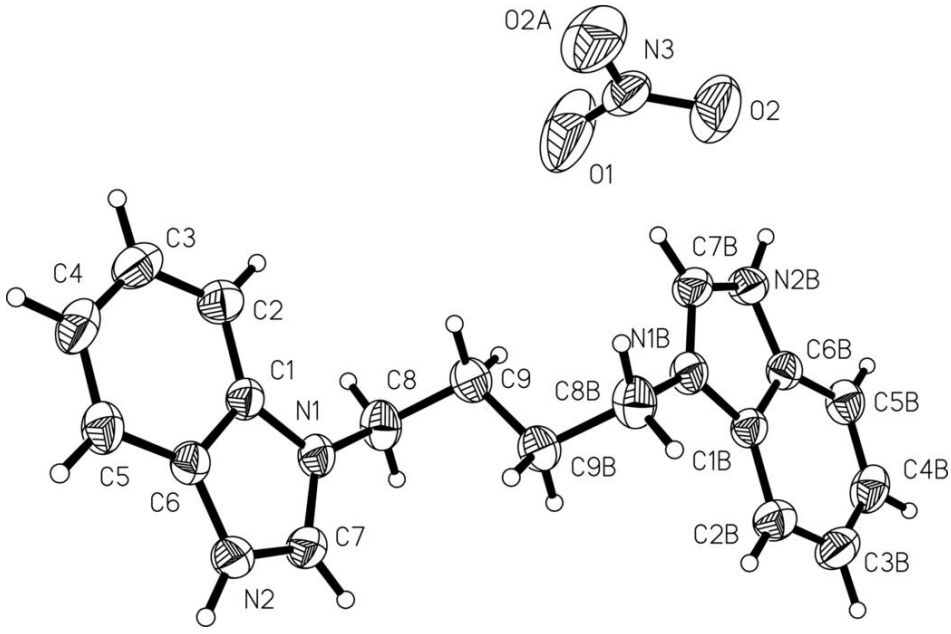
O2A
N3
O2
O1
C3
C4
C2
C1
N1
C8
C9
C8B
N1B
C7B
N2B
C6B
C5B
C4B
C1B
C9B
C5
C6
C7
N2
C2B
C3B

Fig. 2