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5,6-Dimethyl-1*H*-benzimidazol-3-ium nitrate

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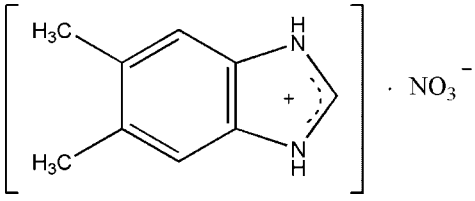
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.045; wR factor = 0.136; data-to-parameter ratio = 13.6.

The title salt, $C_9H_{11}N_2^+\cdot NO_3^-$, features a planar cation (r.m.s. for 11 non-H atoms = 0.016 Å). In the crystal, N—H···O hydrogen bonds link nitrate and benzimidazole ions into a three-dimensional network.

Related literature

For background to benzimidazole, see: Roderick *et al.* (1972). For related crystal structures, see: Lee & Scheidt (1986), Liu (2012), Cui *et al.* (2009).



Experimental

Crystal data

$C_9H_{11}N_2^+\cdot NO_3^-$
M_r = 209.21
Monoclinic, $P2_1/c$
a = 6.938 (4) Å
b = 14.694 (8) Å
c = 10.379 (6) Å
β = 108.598 (9)°
V = 1002.8 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 296 K
0.29 × 0.27 × 0.22 mm

Data collection

Rigaku R-AXIS Spider diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi 1995)
T_{min} = 0.970, T_{max} = 0.977
5401 measured reflections
1973 independent reflections
1617 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.136
S = 1.05
1973 reflections
145 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1^{i}	0.96 (3)	2.31 (3)	3.043 (3)	133.0 (15)
N2—H2···O3^{i}	0.96 (3)	1.86 (3)	2.797 (3)	165 (2)
N1—H1···O1^{ii}	0.90 (3)	2.60 (2)	3.191 (3)	123.8 (17)
N1—H1···O2^{ii}	0.90 (3)	1.89 (2)	2.797 (3)	178 (2)

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

Data collection: *RAPID-AUTO* (Rigaku, 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG5349).

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supporting information

Acta Cryst. (2013). E**69**, o1645 [doi:10.1107/S1600536813027578]

5,6-Dimethyl-1*H*-benzimidazol-3-ium nitrate

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S1. Comment

Benzimidazole and its derivatives have attracted increased interest, not only because of their biological activity, but their abilities to bind to different metal ions (Roderick *et al.*, 1972). In this paper, we describe the synthesis and structure of the title compound $C_9H_{11}N_3O_3$. In the title compound the molecules are linked by N—H···O hydrogen bonds between nitrate and benzimidazole ions into a three-dimensional network structure. Some 5,6-dimethylbenzimidazole derivatives with similar structures have been reported, which include 5.6-Dimethylbenzimidazole (Lee & Scheidt, 1986), 5,6-dimethyl-lH-benzo[*d*]imidazol-3-ium 2-(4-chlorophenoxy)acetate (Liu, 2012),and Bis(5,6-dicarboxybenzimidazolium) sulfate monohydrate (Cui *et al.*, 2009).

S2. Experimental

A mixture of 5,6-Dimethylbenzimidazole (2.86 mg, 0.02 mmol) and $Co(NO_3)_2.6H_2O$ (5.82 mg, 0.02 mmol) was added to H_2O (20 ml). The mixture was refluxed for half an hour then filtered. The resulting solution was allowed to stand at room temperature to give yellow block crystals suitable for structural determination after 3 weeks. Analysis, calculated for $C_9H_{11}N_3O_3$: C 51.67, H 5.30, N 20.09%; Found: C 51.61, H 5.25, N 20.19%.

S3. Refinement

H atoms on N1 and N2 atoms were positioned geometrically and allowed to ride on their parent atoms with N—H = 0.90 or 0.96 Å. H atoms of the methyl groups were positioned geometrically (C—H = 0.96 Å) and allowed to ride on their parent atoms with U_{iso}(H) = 1.5 times U_{eq}(C). All the other H atoms were positioned geometrically(C—H = 0.93 Å) and allowed to ride on their parent atoms with U_{iso}(H) = 1.2 times U_{eq}(C).

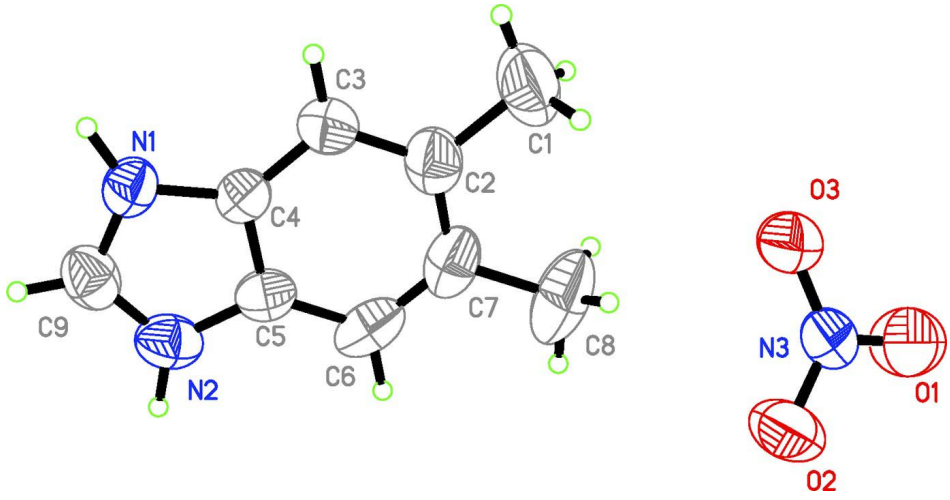

Figure 1

The structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme.

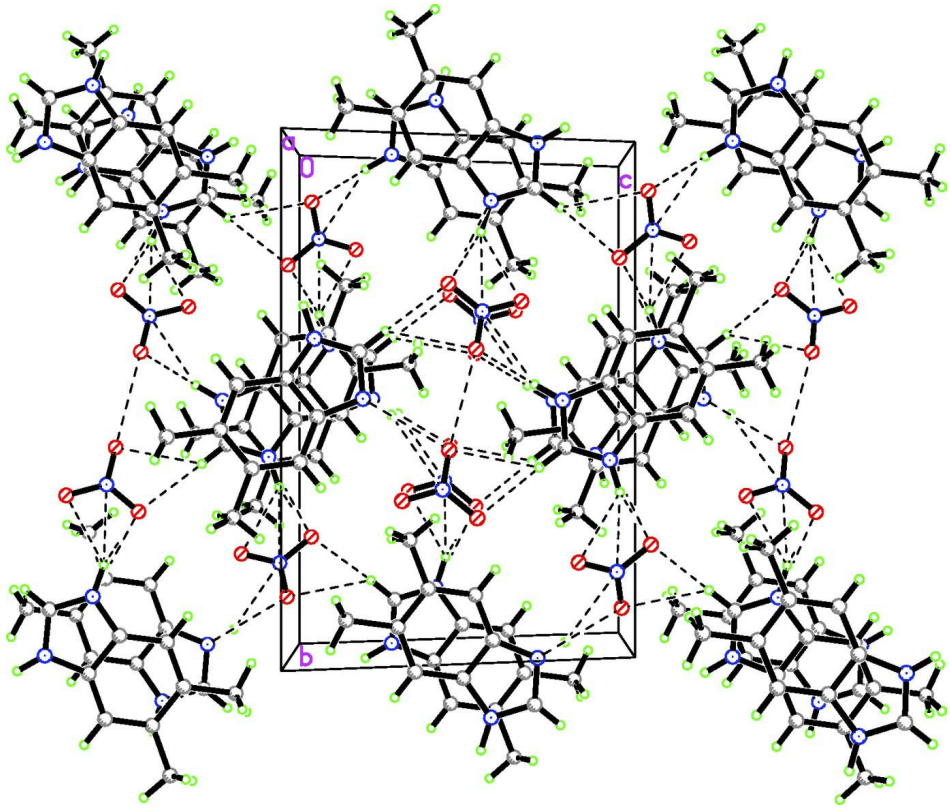

Figure 2

The crystal packing diagram viewed down the *a* axis.

5,6-Dimethyl-1*H*-benzimidazol-3-ium nitrate

Crystal data

$C_9H_{11}N_2^+\cdot NO_3^-$
M_r = 209.21

Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc

a = 6.938 (4) Å
b = 14.694 (8) Å
c = 10.379 (6) Å
β = 108.598 (9)°
V = 1002.8 (10) Å^3
Z = 4
F(000) = 440
D_x = 1.386 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3271 reflections
θ = 2.5–26.6°
μ = 0.11 mm^{-1}
T = 296 K
Block, yellow
0.29 × 0.27 × 0.22 mm

Data collection

Rigaku R-AXIS Spider diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi 1995)
T_{min} = 0.970, T_{max} = 0.977
5401 measured reflections

1973 independent reflections
1617 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 26.0°, θ_{min} = 2.5°
h = −8→8
k = −17→18
l = −8→12
13 standard reflections every 0 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.136
S = 1.05
1973 reflections
145 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0764P)^2 + 0.1519P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.102 (10)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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}
C6	0.3017 (3)	0.52774 (14)	0.13819 (18)	0.0638 (5)
H6A	0.3096	0.5767	0.1965	0.077*
C3	0.2734 (3)	0.38091 (11)	−0.03709 (17)	0.0545 (4)
H3A	0.2625	0.3319	−0.0956	0.065*
N3	0.9477 (2)	0.33163 (9)	0.55623 (14)	0.0615 (4)
C4	0.2174 (2)	0.46728 (10)	−0.08774 (14)	0.0451 (4)

C1	0.4099 (4)	0.27501 (17)	0.1557 (2)	0.0936 (8)
H1A	0.3917	0.2333	0.0816	0.140*
H1B	0.3287	0.2558	0.2102	0.140*
H1C	0.5506	0.2761	0.2104	0.140*
C2	0.3452 (3)	0.36885 (13)	0.10052 (19)	0.0613 (5)
C8	0.4308 (4)	0.4293 (2)	0.3399 (2)	0.1049 (9)
H8A	0.4287	0.4865	0.3839	0.157*
H8B	0.5670	0.4058	0.3678	0.157*
H8C	0.3432	0.3870	0.3647	0.157*
C7	0.3576 (3)	0.44249 (15)	0.18821 (18)	0.0645 (5)
C5	0.2327 (2)	0.53977 (10)	−0.00159 (17)	0.0493 (4)
N2	0.1683 (2)	0.61491 (10)	−0.08221 (17)	0.0609 (4)
C9	0.1169 (3)	0.58998 (12)	−0.20846 (19)	0.0602 (5)
H9A	0.0678	0.6289	−0.2824	0.072*
N1	0.1439 (2)	0.50169 (10)	−0.21736 (13)	0.0519 (4)
O1	0.9388 (3)	0.31136 (10)	0.66814 (13)	0.0858 (5)
O2	1.0253 (3)	0.40438 (9)	0.53833 (14)	0.0857 (5)
O3	0.8828 (3)	0.27846 (9)	0.45932 (13)	0.0829 (5)
H1	0.108 (3)	0.4713 (15)	−0.297 (2)	0.082 (7)*
H2	0.156 (4)	0.6760 (18)	−0.053 (2)	0.097 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C6	0.0503 (9)	0.0868 (13)	0.0550 (10)	−0.0109 (9)	0.0176 (7)	−0.0263 (9)
C3	0.0557 (9)	0.0498 (9)	0.0583 (9)	−0.0039 (7)	0.0187 (7)	−0.0015 (7)
N3	0.0792 (10)	0.0433 (7)	0.0533 (8)	0.0036 (7)	0.0090 (7)	0.0041 (6)
C4	0.0417 (8)	0.0517 (8)	0.0436 (7)	−0.0050 (6)	0.0161 (6)	−0.0024 (6)
C1	0.0874 (16)	0.0854 (15)	0.0949 (16)	−0.0108 (12)	0.0109 (12)	0.0373 (12)
C2	0.0511 (9)	0.0712 (11)	0.0599 (10)	−0.0090 (8)	0.0155 (7)	0.0135 (8)
C8	0.0874 (16)	0.175 (3)	0.0469 (11)	−0.0045 (17)	0.0142 (10)	0.0111 (14)
C7	0.0479 (9)	0.0968 (14)	0.0472 (9)	−0.0077 (9)	0.0128 (7)	0.0073 (9)
C5	0.0413 (8)	0.0518 (9)	0.0559 (9)	−0.0042 (6)	0.0172 (6)	−0.0079 (7)
N2	0.0529 (8)	0.0488 (8)	0.0779 (10)	0.0012 (6)	0.0163 (7)	−0.0064 (7)
C9	0.0499 (9)	0.0584 (10)	0.0699 (11)	0.0022 (7)	0.0156 (8)	0.0138 (8)
N1	0.0517 (8)	0.0601 (9)	0.0442 (7)	−0.0023 (6)	0.0157 (6)	−0.0004 (6)
O1	0.1216 (13)	0.0780 (9)	0.0593 (8)	−0.0232 (8)	0.0309 (8)	−0.0002 (7)
O2	0.1434 (14)	0.0470 (7)	0.0653 (9)	−0.0202 (7)	0.0315 (9)	0.0006 (6)
O3	0.1287 (13)	0.0498 (7)	0.0549 (7)	−0.0080 (7)	0.0078 (7)	−0.0012 (6)

Geometric parameters (Å, º)

C6—C7	1.364 (3)	C1—H1B	0.9600
C6—C5	1.386 (3)	C1—H1C	0.9600
C6—H6A	0.9300	C2—C7	1.399 (3)
C3—C2	1.366 (3)	C8—C7	1.505 (3)
C3—C4	1.381 (2)	C8—H8A	0.9600
C3—H3A	0.9300	C8—H8B	0.9600

N3—O1	1.2197 (19)	C8—H8C	0.9600
N3—O2	1.237 (2)	C5—N2	1.371 (2)
N3—O3	1.2393 (19)	N2—C9	1.296 (2)
C4—C5	1.373 (2)	N2—H2	0.96 (3)
C4—N1	1.374 (2)	C9—N1	1.318 (2)
C1—C2	1.505 (3)	C9—H9A	0.9300
C1—H1A	0.9600	N1—H1	0.90 (2)
C7—C6—C5	118.47 (16)	C7—C8—H8A	109.5
C7—C6—H6A	120.8	C7—C8—H8B	109.5
C5—C6—H6A	120.8	H8A—C8—H8B	109.5
C2—C3—C4	118.80 (16)	C7—C8—H8C	109.5
C2—C3—H3A	120.6	H8A—C8—H8C	109.5
C4—C3—H3A	120.6	H8B—C8—H8C	109.5
O1—N3—O2	120.70 (15)	C6—C7—C2	120.78 (17)
O1—N3—O3	120.23 (15)	C6—C7—C8	118.5 (2)
O2—N3—O3	119.06 (15)	C2—C7—C8	120.7 (2)
C5—C4—N1	106.25 (15)	N2—C5—C4	106.54 (15)
C5—C4—C3	120.73 (15)	N2—C5—C6	132.67 (16)
N1—C4—C3	133.01 (14)	C4—C5—C6	120.79 (16)
C2—C1—H1A	109.5	C9—N2—C5	108.69 (15)
C2—C1—H1B	109.5	C9—N2—H2	124.2 (14)
H1A—C1—H1B	109.5	C5—N2—H2	127.1 (14)
C2—C1—H1C	109.5	N2—C9—N1	110.46 (16)
H1A—C1—H1C	109.5	N2—C9—H9A	124.8
H1B—C1—H1C	109.5	N1—C9—H9A	124.8
C3—C2—C7	120.41 (18)	C9—N1—C4	108.06 (14)
C3—C2—C1	118.79 (19)	C9—N1—H1	123.2 (14)
C7—C2—C1	120.80 (18)	C4—N1—H1	128.6 (14)
C2—C3—C4—C5	−0.2 (2)	C3—C4—C5—N2	179.37 (14)
C2—C3—C4—N1	179.02 (15)	N1—C4—C5—C6	179.55 (13)
C4—C3—C2—C7	1.3 (2)	C3—C4—C5—C6	−1.0 (2)
C4—C3—C2—C1	−178.89 (16)	C7—C6—C5—N2	−179.34 (16)
C5—C6—C7—C2	−0.1 (3)	C7—C6—C5—C4	1.2 (2)
C5—C6—C7—C8	−179.48 (16)	C4—C5—N2—C9	0.16 (18)
C3—C2—C7—C6	−1.1 (3)	C6—C5—N2—C9	−179.36 (17)
C1—C2—C7—C6	179.06 (18)	C5—N2—C9—N1	−0.22 (19)
C3—C2—C7—C8	178.23 (17)	N2—C9—N1—C4	0.19 (18)
C1—C2—C7—C8	−1.6 (3)	C5—C4—N1—C9	−0.09 (16)
N1—C4—C5—N2	−0.04 (16)	C3—C4—N1—C9	−179.40 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1^{i}	0.96 (3)	2.31 (3)	3.043 (3)	133.0 (15)
N2—H2···O3^{i}	0.96 (3)	1.86 (3)	2.797 (3)	165 (2)

N1—H1···O1^{ii}	0.90 (3)	2.60 (2)	3.191 (3)	123.8 (17)
N1—H1···O2^{ii}	0.90 (3)	1.89 (2)	2.797 (3)	178 (2)

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