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# 2-Methylbenzimidazolium nitrate

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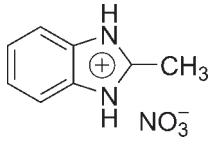
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.045; $wR$ factor = 0.135; data-to-parameter ratio = 13.2.

In the title compound, $C_8H_9N_2^+\cdot NO_3^-$, intermolecular N—H···O hydrogen bonds join the molecules into a chain extending along the $b$ axis.

## Related literature

For the applications of related benzimidazole compounds, see: Wright (1951); El-masry *et al.* (2000); Gümüş *et al.* (2003).



## Experimental

### Crystal data

$C_8H_9N_2^+\cdot NO_3^-$
$M_r$ = 195.18
Monoclinic, $P2_1/c$
$a$ = 7.711 (4) Å
$b$ = 15.127 (7) Å
$c$ = 8.270 (4) Å
$\beta$ = 99.398 (7)°
$V$ = 951.7 (8) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.11 mm$^{-1}$
$T$ = 298 K
0.18 × 0.16 × 0.12 mm

### Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min}$ = 0.981, $T_{max}$ = 0.987
4774 measured reflections
1685 independent reflections
1319 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.027

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.135
$S$ = 1.07
1685 reflections
128 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.21 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H2···O1$^{i}$ | 0.86 | 2.03 | 2.855 (3) | 162 |
| N3—H3···O2$^{ii}$ | 0.86 | 1.93 | 2.775 (2) | 166 |

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

Data collection: *SMART* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; 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: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2255).

## References

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# supporting information

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## 2-Methylbenzimidazolium nitrate

**Qingshuang Ma, Wenzeng Duan, Yudao Ma, Xiao Liu and Bo Qu**

### S1. Comment

Benzimidazole and its derivatives have found practical applications in a number of fields (Wright, 1951). This ring system is present in numerous antiparasitic, antihelmintic and anti-inflammatory drugs (El-masry *et al.*, 2000). The complexes of transition metals with benzimidazole and related ligands have been extensively studied as models of some important biological molecules (Gümüş *et al.*, 2003). During our search to find new benzimidazole-metal complexes 2-methylbenzimidazole nitrate was unintentionally obtained.

Herein, we report the structure of the title compound, $C_8H_9N_3O_3$ (Fig 1). The crystal structure showed that intermolecular N—H···O hydrogen bonds link the molecules into a 1D polymeric structure (Fig. 2).

### S2. Experimental

A mixture of *o*-phenylenediamine(1.08 g, 10 mmol) and anhydrous sodium acetate (2.46 g, 30 mmol) were dissolved in 100 mL 5% hydrochloric acid. After stirring for 2 h under reflux, the solution was cooled to room temperature. Then the solution was treated with ammonia solution to pH 9-10 and an orange precipitate was formed. The precipitate was filtred and washed with water. 2-methylbenzimidazolium chloride was gained in 27.32% yield. The compound 2-methyl-benzimidazole nitrate was obtained in 35% yield when the 2-methylbenzimidazolium chloride (0.46 g, 2.73 mmol) was reacted with $Cr(NO_3)_3.9H_2O$ (1.01 g, 2.54 mmol) in ethanol under reflux. The crystals suitable for X-ray diffraction analysis were obtained by recrystallization from ethanol.

### S3. Refinement

All H atoms were located in difference maps. H atoms bonded to C atoms were then treated as riding atoms in geometrically idealized positions, with C—H distances of 0.93 (aromatic), 0.96 (CH3—H) and 0.86 (N—H) Å, and with $U_{iso}(H) = kU_{eq}(C)$, where k is 1.5 for the methyl group and 1.2 for all the other H atoms.

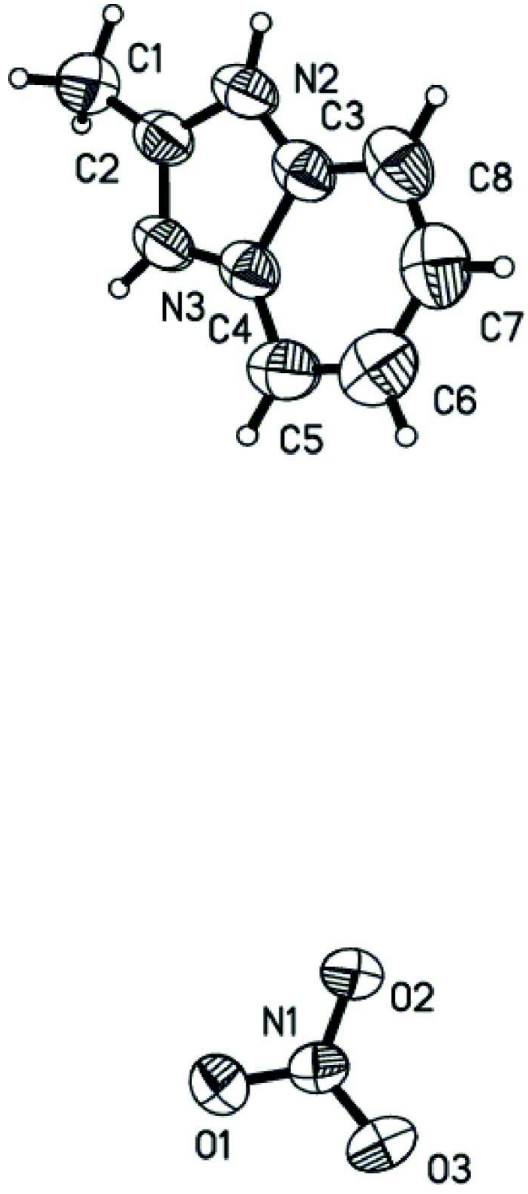


**Figure 1**

The structure of the title compound showing 50% probability displacement.

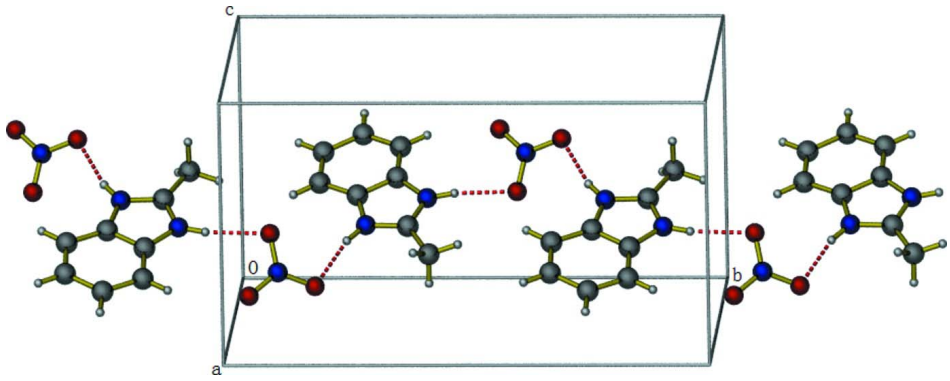


**Figure 2**

The supramolecular chain of the title compound formed via N—H···O hydrogen bonds.

### 2-methylbenzimidazolium nitrate

*Crystal data*

$C_8H_9N_2^+·NO_3^-$
$M_r$ = 195.18
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a$ = 7.711 (4) Å
$b$ = 15.127 (7) Å
$c$ = 8.270 (4) Å
$\beta$ = 99.398 (7)°
$V$ = 951.7 (8) Å$^3$
$Z$ = 4
$F(000)$ = 408
$D_x$ = 1.362 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 2174 reflections
$\theta$ = 2.5–25.5°
$\mu$ = 0.11 mm$^{-1}$
$T$ = 298 K
Block, colorless
0.18 × 0.16 × 0.12 mm

*Data collection*

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min}$ = 0.981, $T_{max}$ = 0.987
4774 measured reflections
1685 independent reflections
1319 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.027
$\theta_{max}$ = 25.1°, $\theta_{min}$ = 2.7°
$h$ = −9→9
$k$ = −11→18
$l$ = −9→9

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.135
$S$ = 1.07
1685 reflections
128 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0681P)^2 + 0.208P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.21 e Å$^{-3}$

*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 $F^2 > \sigma(F^2)$ 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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.3641 (2) | 0.06287 (10) | 0.78462 (18) | 0.0735 (5) |
| O2 | 0.33057 (19) | 0.15524 (9) | 0.97555 (17) | 0.0643 (4) |
| O3 | 0.2248 (3) | 0.02349 (11) | 0.9773 (2) | 0.0915 (6) |
| N1 | 0.3063 (2) | 0.07987 (10) | 0.9136 (2) | 0.0538 (4) |
| N2 | 0.6679 (2) | 0.12445 (11) | 0.25067 (19) | 0.0600 (5) |
| H2 | 0.6828 | 0.0687 | 0.2374 | 0.072* |
| N3 | 0.5577 (2) | 0.24731 (11) | 0.31726 (19) | 0.0576 (5) |
| H3 | 0.4889 | 0.2842 | 0.3542 | 0.069* |
| C1 | 0.3941 (3) | 0.11166 (15) | 0.3743 (3) | 0.0727 (6) |
| H1A | 0.3921 | 0.0516 | 0.3368 | 0.109* |
| H1B | 0.2839 | 0.1395 | 0.3331 | 0.109* |
| H1C | 0.4132 | 0.1126 | 0.4920 | 0.109* |
| C2 | 0.5376 (3) | 0.15990 (13) | 0.3145 (2) | 0.0569 (5) |
| C3 | 0.7769 (3) | 0.19049 (13) | 0.2083 (2) | 0.0561 (5) |
| C4 | 0.7057 (3) | 0.26993 (13) | 0.2519 (2) | 0.0542 (5) |
| C5 | 0.7834 (3) | 0.35057 (14) | 0.2303 (3) | 0.0677 (6) |
| H5 | 0.7356 | 0.4035 | 0.2595 | 0.081* |
| C6 | 0.9355 (3) | 0.34816 (19) | 0.1630 (3) | 0.0805 (7) |
| H6 | 0.9920 | 0.4009 | 0.1463 | 0.097* |
| C7 | 1.0070 (3) | 0.2684 (2) | 0.1191 (3) | 0.0802 (7) |
| H7 | 1.1095 | 0.2695 | 0.0734 | 0.096* |
| C8 | 0.9302 (3) | 0.18829 (18) | 0.1416 (3) | 0.0715 (7) |
| H8 | 0.9787 | 0.1353 | 0.1134 | 0.086* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.1006 (12) | 0.0560 (9) | 0.0718 (10) | 0.0061 (8) | 0.0370 (9) | −0.0048 (7) |
| O2 | 0.0865 (10) | 0.0434 (8) | 0.0651 (9) | −0.0085 (7) | 0.0188 (7) | −0.0067 (6) |
| O3 | 0.1307 (16) | 0.0554 (10) | 0.0990 (13) | −0.0287 (10) | 0.0499 (12) | 0.0017 (8) |
| N1 | 0.0628 (10) | 0.0406 (9) | 0.0590 (10) | 0.0020 (7) | 0.0127 (8) | 0.0042 (7) |
| N2 | 0.0735 (11) | 0.0464 (9) | 0.0564 (10) | 0.0153 (8) | −0.0006 (8) | −0.0086 (7) |
| N3 | 0.0710 (11) | 0.0476 (10) | 0.0521 (9) | 0.0150 (8) | 0.0038 (8) | −0.0094 (7) |
| C1 | 0.0902 (16) | 0.0612 (14) | 0.0673 (13) | 0.0017 (12) | 0.0145 (12) | −0.0006 (11) |
| C2 | 0.0728 (13) | 0.0487 (12) | 0.0462 (10) | 0.0126 (10) | 0.0004 (9) | −0.0052 (8) |
| C3 | 0.0621 (12) | 0.0575 (12) | 0.0440 (10) | 0.0131 (10) | −0.0051 (8) | −0.0086 (9) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C4 | 0.0619 (11) | 0.0536 (11) | 0.0433 (10) | 0.0096 (9) | −0.0024 (8) | −0.0058 (8) |
| C5 | 0.0810 (15) | 0.0539 (13) | 0.0631 (13) | 0.0037 (11) | −0.0033 (11) | −0.0019 (10) |
| C6 | 0.0806 (16) | 0.0842 (18) | 0.0716 (15) | −0.0133 (14) | −0.0030 (12) | 0.0061 (13) |
| C7 | 0.0679 (14) | 0.105 (2) | 0.0662 (14) | 0.0061 (14) | 0.0070 (11) | −0.0032 (14) |
| C8 | 0.0685 (14) | 0.0831 (17) | 0.0593 (13) | 0.0169 (13) | −0.0007 (11) | −0.0124 (11) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—N1 | 1.248 (2) | C1—H1C | 0.9600 |
| O2—N1 | 1.252 (2) | C3—C8 | 1.384 (3) |
| O3—N1 | 1.228 (2) | C3—C4 | 1.393 (3) |
| N2—C2 | 1.322 (3) | C4—C5 | 1.383 (3) |
| N2—C3 | 1.387 (3) | C5—C6 | 1.378 (4) |
| N2—H2 | 0.8600 | C5—H5 | 0.9300 |
| N3—C2 | 1.331 (3) | C6—C7 | 1.399 (4) |
| N3—C4 | 1.383 (3) | C6—H6 | 0.9300 |
| N3—H3 | 0.8600 | C7—C8 | 1.374 (4) |
| C1—C2 | 1.476 (3) | C7—H7 | 0.9300 |
| C1—H1A | 0.9600 | C8—H8 | 0.9300 |
| C1—H1B | 0.9600 | | |
| | | | |
| O3—N1—O1 | 120.19 (17) | C8—C3—N2 | 132.5 (2) |
| O3—N1—O2 | 120.66 (17) | C8—C3—C4 | 121.5 (2) |
| O1—N1—O2 | 119.14 (16) | N2—C3—C4 | 105.96 (18) |
| C2—N2—C3 | 109.90 (17) | C5—C4—N3 | 132.13 (19) |
| C2—N2—H2 | 125.1 | C5—C4—C3 | 122.0 (2) |
| C3—N2—H2 | 125.1 | N3—C4—C3 | 105.85 (18) |
| C2—N3—C4 | 109.90 (16) | C6—C5—C4 | 116.3 (2) |
| C2—N3—H3 | 125.1 | C6—C5—H5 | 121.8 |
| C4—N3—H3 | 125.1 | C4—C5—H5 | 121.8 |
| C2—C1—H1A | 109.5 | C5—C6—C7 | 121.7 (2) |
| C2—C1—H1B | 109.5 | C5—C6—H6 | 119.2 |
| H1A—C1—H1B | 109.5 | C7—C6—H6 | 119.2 |
| C2—C1—H1C | 109.5 | C8—C7—C6 | 121.9 (2) |
| H1A—C1—H1C | 109.5 | C8—C7—H7 | 119.1 |
| H1B—C1—H1C | 109.5 | C6—C7—H7 | 119.1 |
| N2—C2—N3 | 108.40 (19) | C7—C8—C3 | 116.6 (2) |
| N2—C2—C1 | 126.37 (19) | C7—C8—H8 | 121.7 |
| N3—C2—C1 | 125.23 (19) | C3—C8—H8 | 121.7 |
| | | | |
| C3—N2—C2—N3 | −0.6 (2) | C8—C3—C4—N3 | −178.53 (17) |
| C3—N2—C2—C1 | 179.86 (19) | N2—C3—C4—N3 | −0.24 (19) |
| C4—N3—C2—N2 | 0.4 (2) | N3—C4—C5—C6 | 178.52 (19) |
| C4—N3—C2—C1 | 179.99 (19) | C3—C4—C5—C6 | 0.0 (3) |
| C2—N2—C3—C8 | 178.5 (2) | C4—C5—C6—C7 | 0.0 (3) |
| C2—N2—C3—C4 | 0.5 (2) | C5—C6—C7—C8 | −0.4 (4) |
| C2—N3—C4—C5 | −178.8 (2) | C6—C7—C8—C3 | 0.7 (3) |
| C2—N3—C4—C3 | −0.1 (2) | N2—C3—C8—C7 | −178.4 (2) |

| | | | |
|---|---|---|---|
| C8—C3—C4—C5 | 0.3 (3) | C4—C3—C8—C7 | −0.7 (3) |
| N2—C3—C4—C5 | 178.59 (17) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2···O1$^{i}$ | 0.86 | 2.03 | 2.855 (3) | 162 |
| N3—H3···O2$^{ii}$ | 0.86 | 1.93 | 2.775 (2) | 166 |

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