



Crystal structures of the two salts 2-methyl-1*H*-imidazol-3-ium nitrate–2-methyl-1*H*-imidazole (1/1) and 2-methyl-1*H*-imidazol-3-ium nitrate

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Keywords: crystal structure; 2-methyl-1*H*-imidazol-3-ium cations; bifurcated hydrogen bonds; chain structure.**CCDC references:** 1458009; 1458008**Supporting information:** this article has supporting information at journals.iucr.org/e

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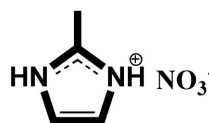
The title salts, $C_4H_7N_2^+ \cdot NO_3^- \cdot C_4H_6N_2$, (I), and $C_4H_7N_2^+ \cdot NO_3^-$, (II), were obtained from solutions containing 2-methylimidazole and nitric acid in different concentrations. In the crystal structure of salt (I), one of the –NH H atoms of the imidazole ring shows half-occupancy, hence only every second molecule is in its cationic form. The nitrate anion in this structure lies on a twofold rotation axis. The neutral 2-methylimidazole molecule and the 2-methyl-1*H*-imidazol-3-ium cation interact through $N-H \cdots N$ hydrogen bonds to form $[(C_4H_6N_2) \cdots (C_4H_7N_2)^+]$ pairs. These pairs are linked with two nitrate anions on both sides through bifurcated $N-H \cdots (O,O)$ hydrogen bonds into chains running parallel to [001]. In the crystal structure of salt (II), the $C_4H_7N_2^+$ cation and the NO_3^- anion are both located on a mirror plane, leading to a statistical disorder of the methyl H atoms. The cations and anions again interact through bifurcated $N-H \cdots (O,O)$ hydrogen bonds, giving rise to the formation of chains consisting of alternating anions and cations parallel to [100].

1. Chemical context

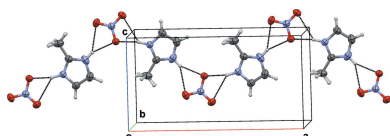
While targeting the synthesis of new Sn^{IV} complexes, crystals of the salt $C_4H_7N_2^+ \cdot NO_3^-$, (II), were obtained serendipitously by mixing trimethyltin acetate with 2-methylimidazole in the presence of nitric acid. In the dynamic of seeking new ammonium salts soluble in organic solvents that can be used for further metallorganic syntheses, we have initiated the targeted preparation of this salt. However, by variation of the ratio between nitric acid and 2-methylimidazole we also obtained crystals of compound (I), $C_4H_6N_2 \cdot C_4H_7N_2^+ \cdot NO_3^-$, and report the two structures in this communication.



(I)



(II)



2. Structural commentary

The asymmetric unit of salt (I) consists of a 2-methylimidazole moiety in a general position and part of a nitrate anion. The

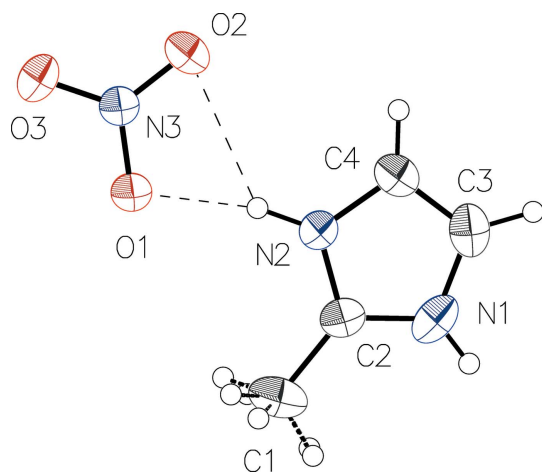


Figure 2
The molecular components of salt (II), showing the atom labelling and displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radius and hydrogen bonds are shown as dashed lines.

anion is completed by application of twofold rotation symmetry. The hydrogen atom H1 attached to N1 of the imidazole ring has a statistical occupancy of 0.5, thus leading to a 1:1 mixture of a 2-methyl-1*H*-imidazol-3-ium cation and a neutral 2-methylimidazole molecule in the crystal applying symmetry operation (i) $1 - x, y, \frac{1}{2} - z$ (Fig. 1). In the nitrate anion, the N—O bond lengths [1.2433 (11)–1.2774 (19) Å], are in a typical range (see, for example, Diop *et al.*, 2013) and indicate some π delocalization over the two oxygen atoms O1 and O1ⁱ. The longer N—O distance is observed for atom O2 involved in the stronger of the two observed N—H \cdots O hydrogen bonds (Table 1). The imidazole ring is planar with a maximum deviation of 0.005 (1) Å. The asymmetric unit of salt (II) consists of an ordered 2-methyl-1*H*-imidazol-3-ium cation and a nitrate anion (Fig. 2), both lying on a mirror plane.

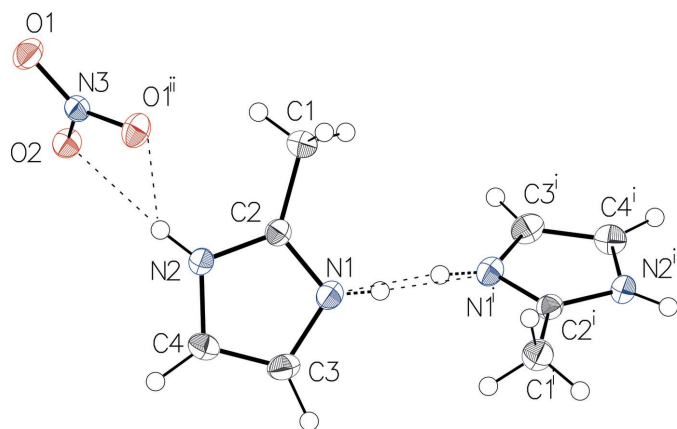


Figure 1
The molecular components of salt (I), showing the atom labelling and displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radius and hydrogen bonds are shown as dashed lines. [Symmetry codes: (i) $1 - x, y, \frac{1}{2} - z$, (ii): $1 - x, y, \frac{3}{2} - z$.]

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

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N2—H2 \cdots O1 ⁱ	0.845 (19)	2.594 (19)	3.1837 (14)	127.9 (15)
N2—H2 \cdots O2	0.845 (19)	2.06 (2)	2.9031 (10)	172.5 (18)
N1—H1 \cdots N1 ⁱⁱ	0.83 (3)	1.86 (3)	2.678 (2)	173 (4)

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

Table 2
Hydrogen-bond geometry (Å, °) for (II).

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1 \cdots O1 ⁱ	0.82 (4)	2.12 (4)	2.894 (2)	157 (4)
N1—H1 \cdots O3 ⁱ	0.82 (4)	2.41 (4)	3.125 (3)	147 (4)
N2—H2 \cdots O1	0.94 (3)	1.83 (3)	2.760 (2)	167 (3)
N2—H2 \cdots O2	0.94 (3)	2.50 (3)	3.231 (2)	135 (2)

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

In the two structures, the O—N—O angles have normal values close to 120° and their sum (360°) reflect a perfect trigonal-planar geometry for each of the nitrate anions. For the 2-methyl-1*H*-imidazol-3-ium cations and for the neutral 2-methylimidazole molecule, the N—C distances involving C2, the C atom that carries the methyl group, are equal within 0.01 Å, and their values are consistent with double-bond character, as previously observed (Diop *et al.*, 2015).

3. Supramolecular features

In the crystal structure of salt (I), the neutral 2-methylimidazole molecule is connected to the 2-methyl-1*H*-imidazol-3-ium cation through N—H \cdots N hydrogen bonds, forming a [(C₄H₆N₂) \cdots (C₄H₇N₂)⁺] pair (Fig. 1). Such pairs are then linked to two nitrate anions through bifurcated N—H \cdots (O,O) hydrogen bonds (Table 1), leading to chains extending along [001] (Fig. 3).

In the crystal structure of (II), the 2-methyl-1*H*-imidazol-3-ium cations and the nitrate anions are alternately linked by bifurcated N—H \cdots (O,O) hydrogen bonds (Table 2), leading to the formation of hydrogen-bonded chains parallel to [100] (Fig. 4).

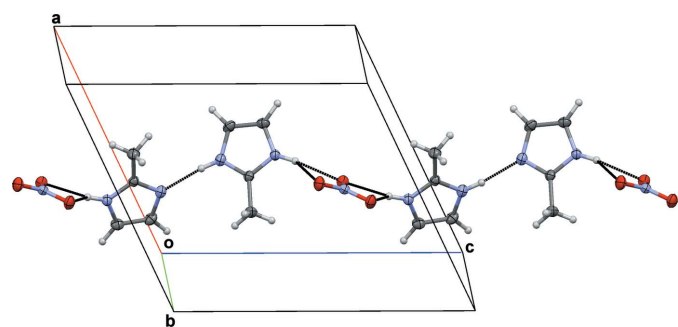


Figure 3
Partial view of the packing in the crystal structure of (I), showing a chain of hydrogen-bonded molecules. Only one of the statistically disordered H-atom positions between the imidazole rings is shown.

Table 3
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	$C_4H_6N_2^+ \cdot NO_3^- \cdot C_4H_7N_2$	$C_4H_7N_2^+ \cdot NO_3^-$
M_r	227.23	145.13
Crystal system, space group	Monoclinic, $C2/c$	Orthorhombic, $Pnma$
Temperature (K)	100	110
a, b, c (Å)	10.1879 (4), 10.0912 (4), 11.9055 (5)	14.1402 (11), 6.2297 (5), 7.4571 (6)
α, β, γ (°)	90, 115.188 (2), 90	90, 90, 90
V (Å ³)	1107.60 (8)	656.89 (9)
Z	4	4
Radiation type	Ga $K\alpha$, $\lambda = 1.34139$ Å	Ga $K\alpha$, $\lambda = 1.34139$ Å
μ (mm ⁻¹)	0.58	0.70
Crystal size (mm)	0.25 × 0.19 × 0.19	0.09 × 0.04 × 0.03
Data collection		
Diffractometer	Bruker Venture Metaljet	Bruker Venture Metaljet
Absorption correction	Multi-scan (<i>SADABS</i> ; Krause <i>et al.</i> , 2015)	Multi-scan (<i>SADABS</i> ; Krause <i>et al.</i> , 2015)
T_{min} , T_{max}	0.682, 0.752	0.471, 0.752
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8771, 1286, 1210	13253, 817, 761
R_{int}	0.033	0.060
$(\sin \theta/\lambda)_{max}$ (Å ⁻¹)	0.650	0.651
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.034, 0.100, 1.04	0.049, 0.146, 1.04
No. of reflections	1286	817
No. of parameters	102	68
H-atom treatment	All H-atom parameters refined	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å ⁻³)	0.25, -0.20	0.22, -0.28

Computer programs: *APEX2* and, *SAINT* (Bruker, 2014), *SHELXT* (Sheldrick, 2015a), *SHELXL2014* (Sheldrick, 2015b), *OLEX2* (Dolomanov *et al.*, 2009), *Mercury* (Macrae *et al.*, 2008), and *pubCIF* (Westrip, 2010).

In the two structures, the stability between the chains is dominated by electrostatic interactions.

4. Database survey

A search of the Cambridge Structural Database (Version 5.37 with one update, Groom & Allen, 2014) for structures containing imidazole or imidazolium rings with nitrate anions returned 21 hits. Molecular chains with bifurcated hydrogen bonds between imidazol-3-ium cations and nitrate anions as found in (II) have been reported for 2-(1-naphthylidiazonyl)-1*H*-imidazol-3-ium nitrate (Pramanik *et al.*, 2010), 2-azidoimidazolium nitrate (Tang *et al.*, 2012) and 2-phenylimidazolium nitrate hemihydrate (Zhang *et al.*, 2007). Molecular chains similar to those observed in (I) with pairs of

imidazole and imidazolium rings linked through bifurcated hydrogen bonds to nitrate anions are also found in the structure of 2-(1*H*-imidazol-2-yl)-1*H*-imidazol-3-ium nitrate (Jin *et al.*, 2011).

5. Synthesis and crystallization

All chemicals were purchased from Aldrich (Germany) and were used as received. Single crystals suitable for X-ray studies of (II) were first obtained by serendipity when a mixture of 2-methylimidazole and concentrated nitric acid was added to trimethyltin acetate in methanol. Colourless single crystals of (I) were obtained after slow evaporation at room temperature of an aqueous solution consisting of 2-methylimidazole and concentrated nitric acid in a 2:1 ratio. Compound (II) can also be prepared in a similar way by changing the ratio between 2-methylimidazole and nitric acid to 1:1.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. For (I), all H atoms were clearly discernible from difference Fourier maps and were freely refined. Half-occupancy of H1 is required for structural reasons and was indicated by the values of the residual density peaks found in the difference Fourier map (0.83 vs 0.47 e Å⁻³

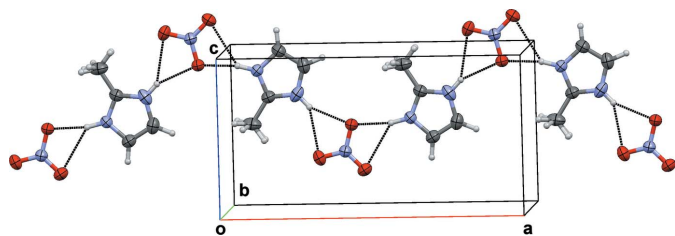


Figure 4
Partial view of the packing in the crystal structure of (II), showing a chain made up of hydrogen-bonded nitrate anions and 2-methyl-1*H*-imidazol-3-ium cations.

for an occupancy factor of 1 and 0.5, respectively). For (II), the H atoms bound to C were placed in calculated positions and then refined using a riding model with C–H = 0.95 Å (aromatic) and 0.98 Å (methyl) and $U_{\text{iso}}(\text{H}) = 1.2$ and $1.5U_{\text{eq}}(\text{C})$, respectively. As a result of the mirror symmetry of the 2-methyl-1*H*-imidazol-3-ium cation, the methyl H atoms are statistically disordered over two positions. H atoms bound to N atoms were located from a difference Fourier map and were freely refined.

Acknowledgements

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supporting information

Acta Cryst. (2016). E72, 482-485 [https://doi.org/10.1107/S2056989016003789]

Crystal structures of the two salts 2-methyl-1*H*-imidazol-3-ium nitrate–2-methyl-1*H*-imidazole (1/1) and 2-methyl-1*H*-imidazol-3-ium nitrate

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Computing details

For both compounds, data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINTE* (Bruker, 2014); data reduction: *SAINTE* (Bruker, 2014); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009) and *publCIF* (Westrip, 2010).

(I) 2-Methyl-1*H*-imidazol-3-ium nitrate–2-methyl-1*H*-imidazole (1/1)

Crystal data

$C_4H_6N_2^+ \cdot NO_3^- \cdot C_4H_7N_2$

$M_r = 227.23$

Monoclinic, *C2/c*

$a = 10.1879$ (4) Å

$b = 10.0912$ (4) Å

$c = 11.9055$ (5) Å

$\beta = 115.188$ (2)°

$V = 1107.60$ (8) Å³

$Z = 4$

$F(000) = 480$

$D_x = 1.363$ Mg m⁻³

Ga $K\alpha$ radiation, $\lambda = 1.34139$ Å

Cell parameters from 6125 reflections

$\theta = 5.7$ – 60.7 °

$\mu = 0.58$ mm⁻¹

$T = 100$ K

Block, clear light colourless

$0.25 \times 0.19 \times 0.19$ mm

Data collection

Bruker Venture Metaljet
diffractometer

Radiation source: Metal Jet, Gallium Liquid
Metal Jet Source

Helios MX Mirror Optics monochromator

Detector resolution: 10.24 pixels mm⁻¹

ω and φ scans

Absorption correction: multi-scan
(SADABS; Krause *et al.*, 2015)

$T_{\min} = 0.682$, $T_{\max} = 0.752$

8771 measured reflections

1286 independent reflections

1210 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.033$

$\theta_{\max} = 60.7$ °, $\theta_{\min} = 5.7$ °

$h = -12 \rightarrow 13$

$k = -13 \rightarrow 13$

$l = -15 \rightarrow 13$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.034$

$wR(F^2) = 0.100$

$S = 1.04$

1286 reflections

102 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Hydrogen site location: difference Fourier map

All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.053P)^2 + 0.953P]$

where $P = (F_o^2 + 2F_c^2)/3$

$$(\Delta/\sigma)_{\max} < 0.001$$

$$\Delta\rho_{\max} = 0.25 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.20 \text{ e } \text{\AA}^{-3}$$

Special details

Experimental. X-ray crystallographic data for I were collected from a single crystal sample, which was mounted on a loop fiber. Data were collected using a Bruker Venture diffractometer equipped with a Photon 100 CMOS Detector, a Helios MX optics and a Kappa goniometer. The crystal-to-detector distance was 4.0 cm, and the data collection was carried out in 1024 x 1024 pixel mode.

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.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
N1	0.56964 (11)	0.62468 (10)	0.37404 (10)	0.0219 (3)	
H1	0.533 (4)	0.626 (3)	0.297 (3)	0.034 (9)*	0.5
N2	0.59828 (11)	0.65353 (10)	0.56421 (9)	0.0198 (2)	
C1	0.37127 (14)	0.74799 (14)	0.39888 (12)	0.0265 (3)	
H1A	0.386 (2)	0.837 (2)	0.3796 (19)	0.053 (6)*	
H1B	0.303 (2)	0.713 (2)	0.3230 (19)	0.049 (5)*	
H1C	0.333 (2)	0.741 (2)	0.454 (2)	0.058 (6)*	
C2	0.51030 (12)	0.67449 (11)	0.44456 (10)	0.0189 (3)	
H2	0.578 (2)	0.6791 (19)	0.6223 (17)	0.036 (4)*	
C3	0.70215 (14)	0.57154 (12)	0.45264 (12)	0.0251 (3)	
H3	0.766 (2)	0.5300 (18)	0.4208 (15)	0.037 (4)*	
C4	0.72067 (13)	0.58907 (12)	0.57091 (12)	0.0238 (3)	
H4	0.802 (2)	0.5656 (17)	0.6485 (16)	0.034 (4)*	
O1	0.45994 (10)	0.92001 (9)	0.82112 (8)	0.0274 (2)	
O2	0.5000	0.73329 (12)	0.7500	0.0229 (3)	
N3	0.5000	0.85988 (14)	0.7500	0.0194 (3)	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0253 (5)	0.0217 (5)	0.0215 (5)	0.0005 (4)	0.0127 (4)	0.0000 (4)
N2	0.0216 (5)	0.0213 (5)	0.0177 (5)	0.0016 (4)	0.0094 (4)	0.0007 (4)
C1	0.0218 (6)	0.0317 (7)	0.0260 (6)	0.0055 (5)	0.0100 (5)	0.0035 (5)
C2	0.0198 (5)	0.0183 (5)	0.0196 (5)	-0.0012 (4)	0.0094 (4)	0.0005 (4)
C3	0.0253 (6)	0.0228 (6)	0.0315 (6)	0.0045 (5)	0.0164 (5)	0.0010 (5)
C4	0.0216 (6)	0.0215 (6)	0.0269 (6)	0.0036 (4)	0.0089 (5)	0.0029 (4)
O1	0.0335 (5)	0.0278 (5)	0.0261 (5)	0.0028 (4)	0.0176 (4)	-0.0026 (3)
O2	0.0258 (6)	0.0211 (6)	0.0238 (6)	0.000	0.0123 (5)	0.000
N3	0.0164 (6)	0.0234 (7)	0.0170 (6)	0.000	0.0056 (5)	0.000

Geometric parameters (\AA , $^\circ$)

N1—H1	0.83 (3)	C1—H1C	0.90 (2)
N1—C2	1.3247 (15)	C1—C2	1.4821 (16)

N1—C3	1.3822 (17)	C3—H3	0.974 (19)
N2—C2	1.3381 (15)	C3—C4	1.3504 (18)
N2—H2	0.845 (19)	C4—H4	0.971 (18)
N2—C4	1.3783 (16)	O1—N3	1.2433 (11)
C1—H1A	0.95 (2)	O2—N3	1.2774 (19)
C1—H1B	0.94 (2)	N3—O1 ⁱ	1.2433 (11)
C2—N1—H1	125 (3)	N1—C2—N2	109.58 (10)
C2—N1—C3	107.21 (10)	N1—C2—C1	125.45 (11)
C3—N1—H1	128 (3)	N2—C2—C1	124.91 (11)
C2—N2—H2	122.4 (13)	N1—C3—H3	121.6 (10)
C2—N2—C4	108.41 (10)	C4—C3—N1	108.53 (11)
C4—N2—H2	129.1 (13)	C4—C3—H3	129.9 (10)
H1A—C1—H1B	104.5 (17)	N2—C4—H4	123.6 (10)
H1A—C1—H1C	114.3 (18)	C3—C4—N2	106.26 (11)
H1B—C1—H1C	107.4 (19)	C3—C4—H4	130.1 (10)
C2—C1—H1A	109.7 (13)	O1—N3—O1 ⁱ	121.58 (14)
C2—C1—H1B	111.1 (12)	O1 ⁱ —N3—O2	119.21 (7)
C2—C1—H1C	109.7 (14)	O1—N3—O2	119.21 (7)
N1—C3—C4—N2	-0.02 (14)	C3—N1—C2—C1	176.33 (12)
C2—N1—C3—C4	0.57 (14)	C4—N2—C2—N1	0.92 (13)
C2—N2—C4—C3	-0.53 (14)	C4—N2—C2—C1	-176.35 (11)
C3—N1—C2—N2	-0.91 (13)		

Symmetry code: (i) $-x+1, y, -z+3/2$.

Hydrogen-bond geometry (\AA , $^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N2—H2 \cdots O1 ⁱ	0.845 (19)	2.594 (19)	3.1837 (14)	127.9 (15)
N2—H2 \cdots O2	0.845 (19)	2.06 (2)	2.9031 (10)	172.5 (18)
N1—H1 \cdots N1 ⁱⁱ	0.83 (3)	1.86 (3)	2.678 (2)	173 (4)

Symmetry codes: (i) $-x+1, y, -z+3/2$; (ii) $-x+1, y, -z+1/2$.

(II) 2-Methyl-1*H*-imidazol-3-ium nitrate

Crystal data

$\text{C}_4\text{H}_7\text{N}^{2+}\cdot\text{NO}_3^-$

$M_r = 145.13$

Orthorhombic, *Pnma*

$a = 14.1402$ (11) \AA

$b = 6.2297$ (5) \AA

$c = 7.4571$ (6) \AA

$V = 656.89$ (9) \AA^3

$Z = 4$

$F(000) = 304$

$D_x = 1.467$ Mg m^{-3}

Ga $K\alpha$ radiation, $\lambda = 1.34139$ \AA

Cell parameters from 9976 reflections

$\theta = 5.2$ – 60.7°

$\mu = 0.70$ mm^{-1}

$T = 110$ K

Block, clear light colorless

$0.09 \times 0.04 \times 0.03$ mm

Data collection

Bruker Venture Metaljet diffractometer	$T_{\min} = 0.471$, $T_{\max} = 0.752$
Radiation source: Metal Jet, Gallium Liquid Metal Jet Source	13253 measured reflections
Helios MX Mirror Optics monochromator	817 independent reflections
Detector resolution: 10.24 pixels mm ⁻¹	761 reflections with $I > 2\sigma(I)$
ω and φ scans	$R_{\text{int}} = 0.060$
Absorption correction: multi-scan (SADABS; Krause <i>et al.</i> , 2015)	$\theta_{\max} = 60.8^\circ$, $\theta_{\min} = 8.1^\circ$
	$h = -16 \rightarrow 18$
	$k = -8 \rightarrow 8$
	$l = -9 \rightarrow 9$

Refinement

Refinement on F^2	Hydrogen site location: mixed
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.049$	$w = 1/[\sigma^2(F_o^2) + (0.0855P)^2 + 0.2528P]$
$wR(F^2) = 0.146$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{\max} < 0.001$
817 reflections	$\Delta\rho_{\max} = 0.22 \text{ e } \text{\AA}^{-3}$
68 parameters	$\Delta\rho_{\min} = -0.28 \text{ e } \text{\AA}^{-3}$
0 restraints	

Special details

Experimental. X-ray crystallographic data for I were collected from a single crystal sample, which was mounted on a loop fiber. Data were collected using a Bruker Venture diffractometer equipped with a Photon 100 CMOS Detector, a Helios MX optics and a Kappa goniometer. The crystal-to-detector distance was 4.0 cm, and the data collection was carried out in 1024 x 1024 pixel mode.

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.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
N1	0.75103 (14)	0.2500	0.7473 (3)	0.0489 (5)	
H1	0.791 (3)	0.2500	0.828 (5)	0.104 (14)*	
N2	0.62239 (12)	0.2500	0.5968 (2)	0.0383 (5)	
H2	0.558 (2)	0.2500	0.566 (4)	0.065 (9)*	
C1	0.6012 (2)	0.2500	0.9290 (3)	0.0581 (7)	
H1A	0.6415	0.2943	1.0295	0.087*	0.5
H1B	0.5768	0.1053	0.9515	0.087*	0.5
H1C	0.5483	0.3504	0.9170	0.087*	0.5
C2	0.65719 (16)	0.2500	0.7623 (3)	0.0409 (5)	
C3	0.77534 (16)	0.2500	0.5686 (3)	0.0493 (6)	
H3	0.8376	0.2500	0.5208	0.059*	
C4	0.69503 (16)	0.2500	0.4755 (3)	0.0448 (6)	
H4	0.6892	0.2500	0.3486	0.054*	
O1	0.42825 (10)	0.2500	0.55858 (18)	0.0449 (5)	
O2	0.46812 (12)	0.2500	0.2772 (2)	0.0498 (5)	
O3	0.32003 (10)	0.2500	0.3546 (2)	0.0482 (5)	
N3	0.40563 (11)	0.2500	0.3932 (2)	0.0384 (5)	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0421 (10)	0.0503 (11)	0.0542 (11)	0.000	-0.0184 (8)	0.000
N2	0.0319 (8)	0.0507 (10)	0.0324 (8)	0.000	-0.0023 (6)	0.000
C1	0.0775 (18)	0.0625 (15)	0.0342 (11)	0.000	0.0076 (10)	0.000
C2	0.0430 (11)	0.0460 (11)	0.0337 (10)	0.000	-0.0045 (8)	0.000
C3	0.0357 (11)	0.0509 (13)	0.0612 (14)	0.000	0.0057 (9)	0.000
C4	0.0448 (12)	0.0522 (12)	0.0374 (10)	0.000	0.0064 (8)	0.000
O1	0.0360 (8)	0.0681 (10)	0.0308 (7)	0.000	-0.0011 (5)	0.000
O2	0.0449 (9)	0.0686 (11)	0.0359 (8)	0.000	0.0092 (6)	0.000
O3	0.0348 (7)	0.0592 (10)	0.0507 (9)	0.000	-0.0103 (6)	0.000
N3	0.0344 (8)	0.0485 (10)	0.0323 (8)	0.000	-0.0005 (6)	0.000

Geometric parameters (\AA , $^\circ$)

N1—H1	0.82 (4)	C1—H1C	0.9800
N1—C2	1.332 (3)	C1—C2	1.474 (3)
N1—C3	1.376 (3)	C3—H3	0.9500
N2—H2	0.94 (3)	C3—C4	1.331 (3)
N2—C2	1.329 (2)	C4—H4	0.9500
N2—C4	1.369 (3)	O1—N3	1.274 (2)
C1—H1A	0.9800	O2—N3	1.237 (2)
C1—H1B	0.9800	O3—N3	1.244 (2)
C2—N1—H1	128 (3)	N1—C2—C1	127.3 (2)
C2—N1—C3	109.29 (19)	N2—C2—N1	106.91 (18)
C3—N1—H1	122 (3)	N2—C2—C1	125.8 (2)
C2—N2—H2	126.1 (18)	N1—C3—H3	126.5
C2—N2—C4	109.63 (18)	C4—C3—N1	106.98 (19)
C4—N2—H2	124.3 (18)	C4—C3—H3	126.5
H1A—C1—H1B	109.5	N2—C4—H4	126.4
H1A—C1—H1C	109.5	C3—C4—N2	107.19 (19)
H1B—C1—H1C	109.5	C3—C4—H4	126.4
C2—C1—H1A	109.5	O2—N3—O1	119.85 (17)
C2—C1—H1B	109.5	O2—N3—O3	122.22 (18)
C2—C1—H1C	109.5	O3—N3—O1	117.93 (16)
N1—C3—C4—N2	0.000 (1)	C3—N1—C2—C1	180.000 (1)
C2—N1—C3—C4	0.000 (1)	C4—N2—C2—N1	0.000 (1)
C2—N2—C4—C3	0.000 (1)	C4—N2—C2—C1	180.000 (1)
C3—N1—C2—N2	0.000 (1)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D—H\cdots A$	$D—H$	$H\cdots A$	$D\cdots A$	$D—H\cdots A$
N1—H1 \cdots O1 ⁱ	0.82 (4)	2.12 (4)	2.894 (2)	157 (4)
N1—H1 \cdots O3 ⁱ	0.82 (4)	2.41 (4)	3.125 (3)	147 (4)

N2—H2···O1	0.94 (3)	1.83 (3)	2.760 (2)	167 (3)
N2—H2···O2	0.94 (3)	2.50 (3)	3.231 (2)	135 (2)

Symmetry code: (i) $x+1/2, y, -z+3/2$.