

2-Methylanilinium nitrate

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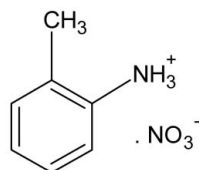
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å;
 R factor = 0.050; wR factor = 0.135; data-to-parameter ratio = 18.7.

The crystal structure of the title compound, $\text{C}_7\text{H}_{10}\text{N}^+\cdot\text{NO}_3^-$, consists of anion–cation layers parallel to the (100) plane, generated by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds. 16 of the 22 atoms (the exceptions being two O and four H atoms) lie on a mirror plane.

Related literature

For related structures see: Benali-Cherif, Direm *et al.* (2007); Benali-Cherif, Allouche *et al.* (2007); Bendeif *et al.* (2007).



Experimental

Crystal data

$\text{C}_7\text{H}_{10}\text{N}^+\cdot\text{NO}_3^-$
 $M_r = 170.17$
Orthorhombic, $Pnma$
 $a = 16.5632$ (2) Å
 $b = 6.7242$ (2) Å
 $c = 7.6849$ (3) Å
 $V = 855.90$ (4) Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 293$ (2) K
 $0.15 \times 0.1 \times 0.05$ mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
9040 measured reflections
1331 independent reflections
895 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.075$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.135$
 $S = 1.05$
1331 reflections
71 parameters
4 restraints
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.14$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.16$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H11}\cdots\text{N2}$	0.85	2.61	3.441 (2)	168
$\text{N1}-\text{H11}\cdots\text{O3}$	0.85	2.23	2.9974 (17)	150
$\text{N1}-\text{H11}\cdots\text{O3}^{\text{i}}$	0.85	2.23	2.9974 (17)	150
$\text{N1}-\text{H12}\cdots\text{O3}^{\text{ii}}$	0.88	1.97	2.8466 (14)	176

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

Data collection: *KappaCCD Server Software* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2199).

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supplementary materials

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2-Methylanilinium nitrate

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Comment

O-toluidine is used in the manufacture of rubber vulcanization accelerator, hypnotic and anesthetic pharmaceuticals, and pesticides. 2-Methylaniline is highly toxic to humans when absorbed through the skin, inhaled as vapor or swallowed, hemoglobin is changed to methemoglobin and caused damage to the cells of the central nervous system.

The crystal structure of *o*-methylanilinium nitrate, (I), was determined as part of our investigations on the structural characteristics of organic-inorganic layered compounds and an ongoing study on D—H...A hydrogen-bonding in systems of hybrid materials including anilinium derivatives such as 4-Carboxyanilinium hydrogensulfate (Benali-Cherif, Direm *et al.*, 2007), 2-carboxyanilinium dihydrogenphosphate (Benali-Cherif, Allouche *et al.*, 2007) and guaninium phosphite and guaninium phosphate salts (Bendeif *et al.* 2007).

The asymmetric unit of (I) contains a monoprotonated *o*-methylanilinium cation and nitrate anion (Figure 1). Intra atomic bond distances and angles in the title compound shows the monoprotonation of the organic entity and confirms the presence of the nitrate (NO₃⁻) anion. All atoms in the asymmetric unit except three are positioned on a mirror plane (*x*, 1/4, *z*).

The structure of (C₇H₁₀N⁺. NO₃⁻) is composed of cationic (C₇H₁₀N⁺), and anionic (NO₃⁻) entities which are linked by N—H...O hydrogen bonds to build up layers developing parallel to the (1 0 0) plane (Table 1, Fig. 2).

Experimental

Single crystals of the title compound are prepared by slow evaporation at room temperature of an aqueous solution of *o*-methylaniline (C₇H₉N) and nitrate acid (HNO₃) in the stoichiometric ratio 1:1.

Refinement

Aromatic H atoms were located in difference Fourier syntheses and were allowed to ride on their parent C atoms with C—H = 0.93 Å and *U*_{iso} = 1.2*U*_{eq}(C). The methylene and ammonium H-atoms of the cation entity were located in difference Fourier syntheses but were not refined.

Figures

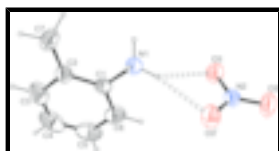


Fig. 1. Molecular view of the title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Hydrogen bonds are shown as dashed lines. H atoms are represented as small spheres of arbitrary radii. [Symmetry code: (i) *x*, 1/2 - *y*, *z*]

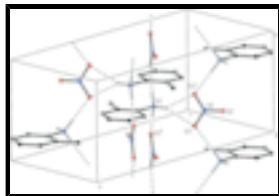


Fig. 2. Partial packing view showing the hydrogen-bonding network. H atoms not involved in H bonds have been omitted for clarity. [Symmetry code: (i) $x, 1/2 - y, z$; (ii) $1/2 - x, -y, 1/2 + z$.]

2-Methylanilinium nitrate

Crystal data

$C_7H_{10}N^+ \cdot NO_3^-$

$M_r = 170.17$

Orthorhombic, $Pnma$

Hall symbol: $-P\ 2ac\ 2n$

$a = 16.5632\ (2)\ \text{\AA}$

$b = 6.7242\ (2)\ \text{\AA}$

$c = 7.6849\ (3)\ \text{\AA}$

$V = 855.90\ (4)\ \text{\AA}^3$

$Z = 4$

$F_{000} = 360$

$D_x = 1.321\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 1467 reflections

$\theta = 2.9\text{--}30.0^\circ$

$\mu = 0.10\ \text{mm}^{-1}$

$T = 293\ (2)\ \text{K}$

Prism, brown

$0.15 \times 0.1 \times 0.05\ \text{mm}$

Data collection

Nonius KappaCCD diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293\ (2)\ \text{K}$

ω - θ scans

Absorption correction: none

9040 measured reflections

1331 independent reflections

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

$R_{\text{int}} = 0.075$

$\theta_{\text{max}} = 30.0^\circ$

$\theta_{\text{min}} = 2.9^\circ$

$h = 0 \rightarrow 23$

$k = 0 \rightarrow 9$

$l = 0 \rightarrow 10$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.050$

$wR(F^2) = 0.135$

$S = 1.05$

1331 reflections

71 parameters

4 restraints

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0468P)^2 + 0.1577P]$$

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

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

$\Delta\rho_{\text{max}} = 0.14\ \text{e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.16\ \text{e \AA}^{-3}$

Extinction correction: SHELXL97,

$$F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$$

Primary atom site location: structure-invariant direct methods Extinction coefficient: 0.23 (3)

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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.40546 (11)	0.2500	0.7455 (2)	0.0478 (5)
C2	0.44414 (12)	0.2500	0.9045 (2)	0.0566 (5)
C3	0.52755 (15)	0.2500	0.8999 (4)	0.0880 (8)
H3	0.5563	0.2500	1.0039	0.106*
C4	0.56892 (16)	0.2500	0.7448 (5)	0.1032 (11)
H4	0.6251	0.2500	0.7456	0.124*
C5	0.52909 (18)	0.2500	0.5908 (4)	0.0943 (9)
H5	0.5577	0.2500	0.4866	0.113*
C6	0.44633 (15)	0.2500	0.5899 (3)	0.0722 (6)
H6	0.4182	0.2500	0.4852	0.087*
C7	0.39797 (17)	0.2500	1.0725 (3)	0.0797 (7)
H71	0.4314	0.2500	1.1725	0.120*
H72	0.3651	0.1307	1.0813	0.120*
N1	0.31729 (9)	0.2500	0.74140 (19)	0.0532 (4)
H11	0.3021	0.2500	0.6356	0.080*
H12	0.2989	0.1443	0.7967	0.080*
N2	0.22458 (9)	0.2500	0.3407 (2)	0.0527 (4)
O2	0.18148 (12)	0.2500	0.2138 (2)	0.0919 (6)
O3	0.24779 (7)	0.09186 (15)	0.40892 (14)	0.0761 (4)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0494 (10)	0.0416 (8)	0.0525 (9)	0.000	0.0035 (7)	0.000
C2	0.0555 (11)	0.0532 (10)	0.0611 (11)	0.000	-0.0069 (8)	0.000
C3	0.0553 (14)	0.0946 (19)	0.114 (2)	0.000	-0.0189 (14)	0.000
C4	0.0474 (13)	0.0858 (19)	0.176 (4)	0.000	0.0188 (18)	0.000
C5	0.085 (2)	0.0830 (18)	0.115 (2)	0.000	0.0503 (18)	0.000
C6	0.0780 (16)	0.0786 (15)	0.0599 (12)	0.000	0.0207 (10)	0.000
C7	0.0927 (17)	0.0981 (18)	0.0484 (11)	0.000	-0.0069 (11)	0.000
N1	0.0518 (9)	0.0583 (9)	0.0497 (8)	0.000	-0.0026 (7)	0.000

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N2	0.0469 (8)	0.0547 (9)	0.0566 (9)	0.000	-0.0044 (7)	0.000
O2	0.0976 (13)	0.0913 (13)	0.0868 (11)	0.000	-0.0493 (10)	0.000
O3	0.0922 (9)	0.0513 (6)	0.0848 (8)	-0.0052 (5)	-0.0271 (6)	0.0099 (5)

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

C1—C6	1.374 (3)	C5—H5	0.9300
C1—C2	1.380 (2)	C6—H6	0.9300
C1—N1	1.461 (2)	C7—H71	0.9467
C2—C3	1.382 (3)	C7—H72	0.9714
C2—C7	1.501 (3)	N1—H11	0.8508
C3—C4	1.375 (4)	N1—H12	0.8826
C3—H3	0.9300	N2—O2	1.209 (2)
C4—C5	1.355 (4)	N2—O3 ⁱ	1.2465 (12)
C4—H4	0.9300	N2—O3	1.2465 (12)
C5—C6	1.371 (4)		
C6—C1—C2	122.80 (18)	C6—C5—H5	120.3
C6—C1—N1	118.31 (17)	C5—C6—C1	119.3 (2)
C2—C1—N1	118.89 (15)	C5—C6—H6	120.4
C1—C2—C3	116.2 (2)	C1—C6—H6	120.4
C1—C2—C7	121.70 (18)	C2—C7—H71	113.6
C3—C2—C7	122.1 (2)	C2—C7—H72	110.2
C4—C3—C2	121.3 (3)	H71—C7—H72	105.7
C4—C3—H3	119.3	C1—N1—H11	108.4
C2—C3—H3	119.3	C1—N1—H12	109.6
C5—C4—C3	121.0 (2)	H11—N1—H12	111.0
C5—C4—H4	119.5	O2—N2—O3 ⁱ	121.45 (8)
C3—C4—H4	119.5	O2—N2—O3	121.45 (8)
C4—C5—C6	119.4 (2)	O3 ⁱ —N2—O3	117.10 (15)
C4—C5—H5	120.3		

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H11 \cdots N2	0.85	2.61	3.441 (2)	168
N1—H11 \cdots O3	0.85	2.23	2.9974 (17)	150
N1—H11 \cdots O3 ⁱ	0.85	2.23	2.9974 (17)	150
N1—H12 \cdots O3 ⁱⁱ	0.88	1.97	2.8466 (14)	176

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

Fig. 1

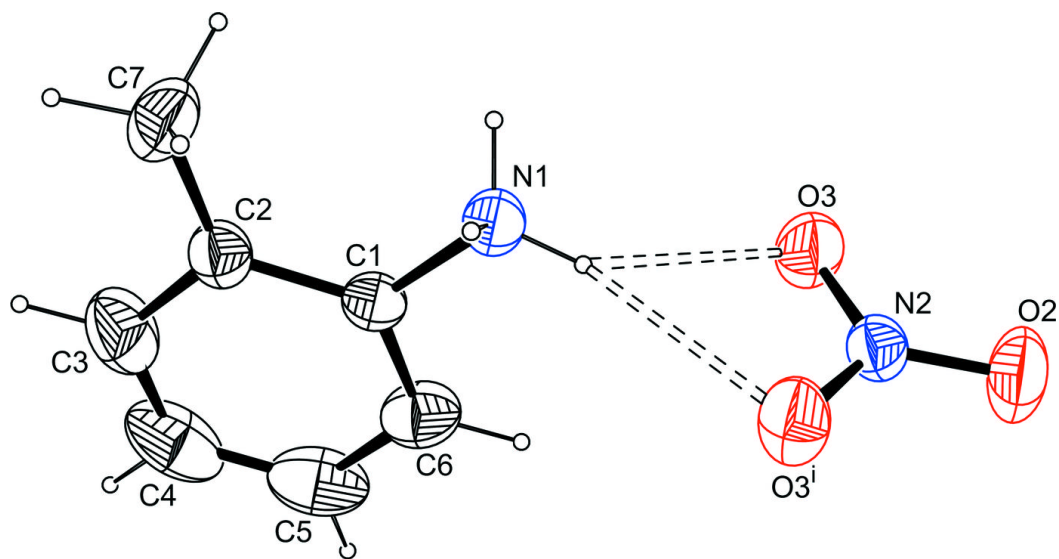


Fig. 2

