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Dimethylammonium nitrate

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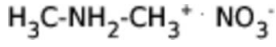
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(O–N) = 0.003 Å; R factor = 0.036; wR factor = 0.087; data-to-parameter ratio = 8.6.

The title compound, $C_2H_8N^+\cdot NO_3^-$, is composed of discrete cations and anions which are connected by classical N—H···O hydrogen bonds. Geometric parameters are in the usual ranges.

Related literature

For related literature, see: Bieller *et al.* (2005); Trofimenko (1993); Vitze *et al.* (2006); Zhang *et al.* (2004).



Experimental

Crystal data

$C_2H_8N^+\cdot NO_3^-$
M_r = 108.10
Orthorhombic, $Pna2_1$
a = 9.9552 (18) Å
b = 9.7684 (12) Å
c = 5.7191 (7) Å
V = 556.16 (14) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 173 (2) K
0.37 × 0.17 × 0.08 mm

Data collection

STOE IPDS II two-circle-diffractometer
Absorption correction: none
4065 measured reflections
557 independent reflections
492 reflections with $I > 2\sigma(I)$
R_{int} = 0.081

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.087
S = 1.07
557 reflections
65 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O3	0.92	2.38	3.018 (3)	126
N2—H2B···O1^{i}	0.92	1.98	2.892 (3)	169
N2—H2B···O3^{i}	0.92	2.43	3.035 (3)	123
N2—H2A···O1^{ii}	0.92	2.37	2.994 (3)	125
N2—H2A···O2^{ii}	0.92	2.25	3.000 (3)	139

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

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2464).

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supplementary materials

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Dimethylammonium nitrate

H. Vitze, H.-W. Lerner and M. Bolte

Comment

Poly(pyrazol-1-yl)borates ("*scorpionates*") are among the most important ligands in transition metal chemistry and were invented by Trofimenko more than 30 years ago (Trofimenko, 1993). Recently, we have extended our studies to ditopic scorpionates with *meta*- and *para*-phenylene backbones. Our studies have shown that the synthesis of scorpionates can conveniently be achieved by reaction of bis(dimethylamino)arylboranes and pyrazole or pyrazole derivatives in the presence of bases (Bieller *et al.*, 2005; Zhang *et al.*, 2004). In attempting to synthesize a zinc complex with the $[C_6F_5Bpz_3]$ ligand (pz = pyrazolyl) from $C_6F_5B(NMe_2)_2$ (Me = CH_3) and $[Znpz_4(NO_3)_2]$ (Vitze *et al.*, 2006) we obtained the title compound, $H_2NMe_2NO_3$, as a by-product. Single crystals of $H_2NMe_2NO_3$ were obtained from a mixture of tetrahydrofuran/hexane at ambient temperature.

The title compound is composed of discrete cations and anions (Fig. 1) which are connected by classical N—H···O hydrogen bonds. Two of the nitrate O atoms accept two hydrogen bonds, whereas the third one just accepts one (Fig. 2). Geometric parameters are in the usual ranges.

Experimental

A mixture of $C_6F_5B(NMe_2)_2$ (0.266 g) and $[Znpz_4(NO_3)_2]$ (Vitze *et al.*, 2006) in 6 ml tetrahydrofuran and 6 ml NEt_3 was refluxed for 1 h. After removal of the solvent *in vacuo* the residue was extracted with tetrahydrofuran. Layering with hexane led to the deposition of crystals of $H_2NMe_2NO_3$ at ambient temperature.

Refinement

All H atoms could be located by difference Fourier synthesis. They were refined with fixed individual displacement parameters [U_{iso}(H) = 1.2 U_{eq}(N) or U_{iso}(H) = 1.5 U_{eq}(C)] using a riding model with N—H = 0.92Å or C—H = 0.98 Å, respectively. In the absence of anomalous scatterers, the Flack (1983) parameter is meaningless and therefore, Friedel pairs had been merged prior to refinement.

Figures

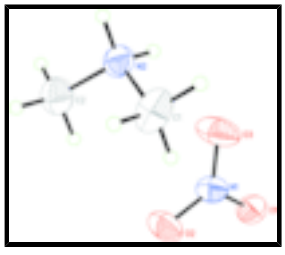

Fig. 1. Perspective view of the title compound with the atom numbering; displacement ellipsoids are at the 50% probability level.

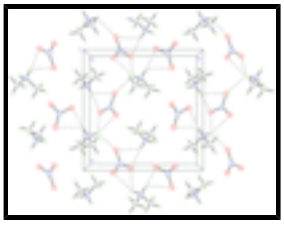

Fig. 2. Packing diagram of the title compound with view onto the *ab* plane. Hydrogen bonds are drawn as dashed lines.

Dimethylammonium nitrate

Crystal data

$C_2H_8N^+·NO_3^-$	F_{000} = 232
M_r = 108.10	D_x = 1.291 Mg m^{-3}
Orthorhombic, *Pna*2$_1$	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: P 2c -2n	Cell parameters from 3599 reflections
a = 9.9552 (18) Å	θ = 4.1–25.4º
b = 9.7684 (12) Å	μ = 0.12 mm^{-1}
c = 5.7191 (7) Å	*T* = 173 (2) K
V = 556.16 (14) Å^3	Plate, colourless
Z = 4	0.37 × 0.17 × 0.08 mm

Data collection

STOE IPDS II two-circle-diffractometer	492 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.081
Monochromator: graphite	θ_{max} = 25.3º
T = 173(2) K	θ_{min} = 4.1º
ω scans	*h* = −11→10
Absorption correction: none	*k* = −11→10
4065 measured reflections	*l* = −6→6
557 independent reflections	

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0532P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.036	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.087	$\Delta\rho_{max}$ = 0.14 e Å^{-3}
S = 1.07	$\Delta\rho_{min}$ = −0.12 e Å^{-3}
557 reflections	Extinction correction: SHELXL, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
65 parameters	Extinction coefficient: 0.042 (13)
1 restraint	

Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites

Special details

Experimental. ;

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}
N1	0.4727 (2)	0.2067 (2)	0.4304 (4)	0.0357 (5)
O1	0.52001 (19)	0.1106 (2)	0.3073 (4)	0.0427 (6)
O2	0.5451 (2)	0.2631 (2)	0.5812 (4)	0.0460 (6)
O3	0.3538 (2)	0.2435 (2)	0.3993 (5)	0.0614 (8)
C1	0.3170 (4)	0.5648 (3)	0.5141 (6)	0.0487 (8)
H1A	0.2722	0.5897	0.3676	0.073*
H1B	0.3991	0.5133	0.4793	0.073*
H1C	0.3401	0.6481	0.6007	0.073*
N2	0.2255 (2)	0.4789 (2)	0.6577 (4)	0.0348 (6)
H2A	0.2021	0.4022	0.5736	0.042*
H2B	0.1482	0.5274	0.6879	0.042*
C2	0.2866 (3)	0.4355 (3)	0.8836 (5)	0.0452 (7)
H2C	0.2221	0.3794	0.9708	0.068*
H2D	0.3099	0.5166	0.9758	0.068*
H2E	0.3679	0.3819	0.8527	0.068*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0295 (11)	0.0322 (12)	0.0452 (13)	−0.0013 (9)	−0.0008 (12)	−0.0014 (11)
O1	0.0356 (11)	0.0484 (13)	0.0440 (10)	0.0081 (9)	0.0035 (9)	−0.0096 (10)
O2	0.0388 (11)	0.0438 (12)	0.0556 (14)	−0.0041 (9)	−0.0129 (10)	−0.0129 (10)
O3	0.0350 (12)	0.0566 (15)	0.0927 (19)	0.0132 (9)	−0.0180 (13)	−0.0332 (14)
C1	0.0600 (19)	0.0417 (16)	0.0444 (17)	−0.0017 (15)	0.0152 (15)	−0.0015 (14)
N2	0.0331 (11)	0.0348 (12)	0.0364 (12)	−0.0030 (9)	0.0031 (10)	−0.0093 (10)
C2	0.0510 (17)	0.0467 (17)	0.0379 (16)	−0.0021 (14)	0.0003 (13)	0.0014 (13)

Geometric parameters (Å, °)

N1—O3	1.249 (3)	N2—C2	1.489 (4)
N1—O2	1.252 (3)	N2—H2A	0.9200
N1—O1	1.264 (3)	N2—H2B	0.9200
C1—N2	1.487 (4)	C2—H2C	0.9800
C1—H1A	0.9800	C2—H2D	0.9800
C1—H1B	0.9800	C2—H2E	0.9800
C1—H1C	0.9800		
O3—N1—O2	121.1 (3)	C2—N2—H2A	109.0
O3—N1—O1	119.2 (2)	C1—N2—H2B	109.0
O2—N1—O1	119.7 (2)	C2—N2—H2B	109.0
N2—C1—H1A	109.5	H2A—N2—H2B	107.8
N2—C1—H1B	109.5	N2—C2—H2C	109.5
H1A—C1—H1B	109.5	N2—C2—H2D	109.5
N2—C1—H1C	109.5	H2C—C2—H2D	109.5
H1A—C1—H1C	109.5	N2—C2—H2E	109.5
H1B—C1—H1C	109.5	H2C—C2—H2E	109.5
C1—N2—C2	112.9 (2)	H2D—C2—H2E	109.5
C1—N2—H2A	109.0		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···O3	0.92	2.38	3.018 (3)	126
N2—H2B···O1^{i}	0.92	1.98	2.892 (3)	169
N2—H2B···O3^{i}	0.92	2.43	3.035 (3)	123
N2—H2A···O1^{ii}	0.92	2.37	2.994 (3)	125
N2—H2A···O2^{ii}	0.92	2.25	3.000 (3)	139

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

Fig. 1

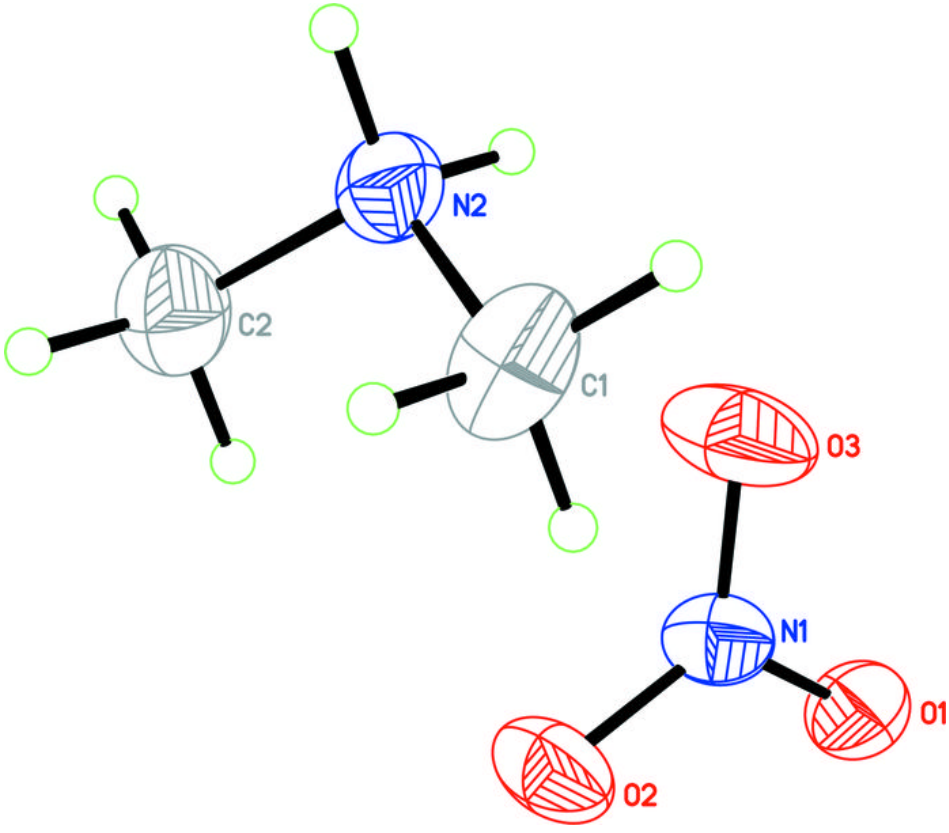

Fig. 2