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4-Methylbenzylammonium nitrate

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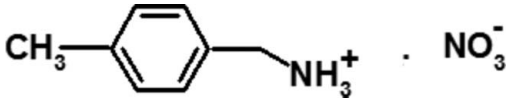
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.071; wR factor = 0.216; data-to-parameter ratio = 36.3.

In the title salt, $C_8H_{12}N^+\cdot NO_3^-$, the N atom of the 4-methylbenzylammonium cation is displaced by 1.366 (2) Å from the mean plane of the other atoms. In the crystal, the cations are connected to the anions by N—H···O and N—H···(O,O) hydrogen bonds, generating a layered network parallel to (100). A weak C—H···O interaction also occurs.

Related literature

For related structures, see: Kefi *et al.* (2011); Rahmouni *et al.* (2011). For a discussion on hydrogen bonding, see: Brown (1976); Blessing (1986). For aromatic π–π stacking interactions, see: Janiak (2000). For graph-set notation of hydrogen-bonding patterns, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_8H_{12}N^+\cdot NO_3^-$
M_r = 184.20
Monoclinic, $P2_1/c$
a = 15.097 (2) Å
b = 5.8121 (10) Å
c = 10.486 (2) Å
β = 99.75 (2)°
V = 906.8 (3) Å^3
Z = 4
Ag $K\alpha$ radiation
λ = 0.56083 Å
μ = 0.06 mm^{-1}
T = 293 K
0.40 × 0.35 × 0.30 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
6579 measured reflections
4430 independent reflections
2415 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
2 standard reflections every 120 min
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.071
$wR(F^2)$ = 0.216
S = 0.96
4430 reflections
122 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.89	2.07	2.936 (3)	164
N1—H1A···O3^{i}	0.89	2.52	3.065 (2)	120
N1—H1B···O3^{ii}	0.89	2.12	2.9378 (19)	153
N1—H1C···O3	0.89	2.01	2.900 (2)	179
N1—H1C···O2	0.89	2.55	3.158 (3)	126
C8—H8A···O1^{iii}	0.97	2.45	3.234 (2)	138

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB7131).

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supporting information

Acta Cryst. (2013). E**69**, o1453 [doi:10.1107/S1600536813022836]

4-Methylbenzylammonium nitrate

Sofian Gatfaoui, Houda Marouani and Mohamed Rzaigui

S1. Comment

We report here the preparation and the crystal structure of the title compound, $C_8H_{12}N{\cdot}NO_3$ (I).

The asymmetric unit of (I) consists of one nitrate anion and one 4-methylbenzylammonium cation (Figure 1). The 4-methylbenzylammonium cations are connected to the nitrate anions through weak N—H···O and C—H···O hydrogen bonds with donor-acceptor distances varying between 2.900 (2) and 3.234 (2) Å [d (N(C)···O) > 2.73 Å] (Brown, 1976); (Blessing, 1986) (Table 1, Figure 2).

In the nitrate anion, the distance N2—O2 is significantly shorter than the N2—O1 and N2—O3 distances because O2 is applied in only one hydrogen bond (table1) while O1 and O3 are applied in two and three hydrogen bonds, respectively. These geometrical features have also been noticed in other crystal structures (Rahmouni, *et al.*, 2011).

Each organic entity is bounded to three different nitrate anions through five N—H···O hydrogen bonds forming $R_1^2(4)$ and $R_4^2(8)$ motifs (Fig. 3) (Bernstein, *et al.*, 1995). Examination of the 4-methylbenzylammonium cation shows that the bond distances and angles show no significant difference from those obtained in other structures involving the same organic groups (Kefi, *et al.*, 2011). The aromatic ring of the organic cation is essentially planar with an r.m.s deviation of 0.0099 Å. The inter-planar distance between nearby phenyl rings is in the vicinity of 5.925 Å, which is much longer than 3.80 Å, value required for the formation of π–π interactions (Janiak, 2000).

The crystal cohesion and stability are ensured by electrostatic and van der Waals interactions which, together with N—H···O and C—H···O hydrogen bonds, build up a two-dimensional network.

S2. Experimental

An aqueous solution containing 1 mmol of HNO_3 in 10 ml of water, was added to 1 mmol of 4-xylylamine in 10 ml of ethanol. The obtained solution was stirred for 20 min and then left to stand at room temperature. Colorless prisms of the title compound were obtained after some days.

S3. Refinement

All H atoms were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic) or 0.97 Å (methylene) or 0.96 Å (methyl), N—H = 0.89 Å with U_{iso}(H) = 1.2Ueq(C or N).

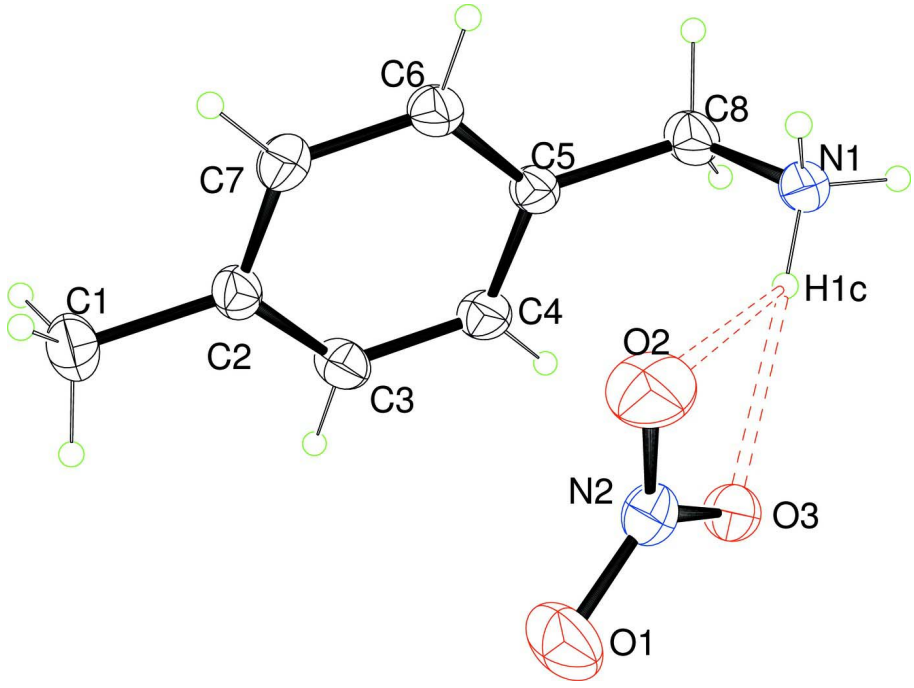

Figure 1

An *ORTEP* view of (I) with displacement ellipsoids drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii. Hydrogen bonds are shown as dotted lines.

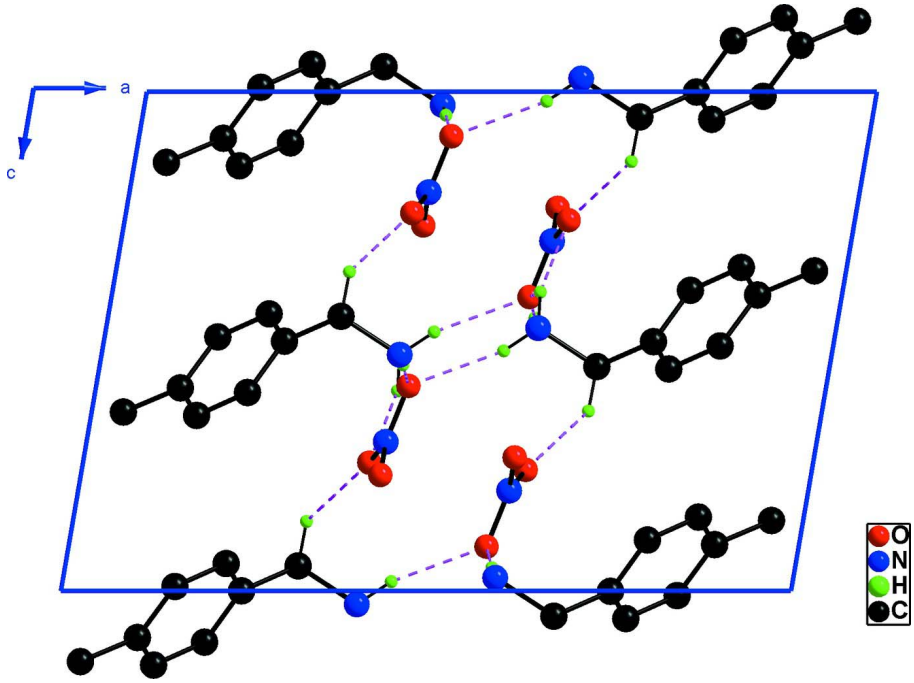

Figure 2

Projection of (I) along the *b* axis. The H-atoms not involved in H-bonding are omitted.

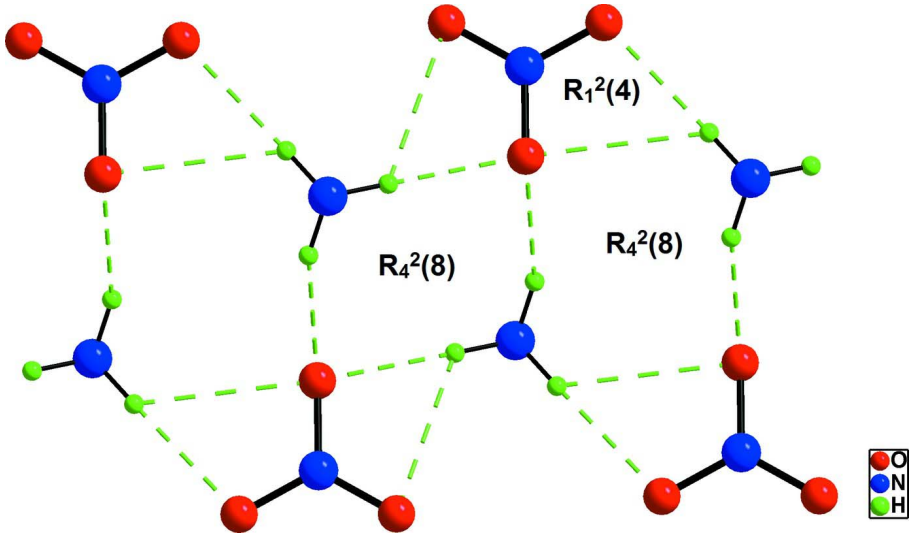

Figure 3

Hydrogen bond motifs in (I).

4-Methylbenzylammonium nitrate

Crystal data

$C_8H_{12}N^+\cdot NO_3^-$
M_r = 184.20
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 15.097 (2) Å
b = 5.8121 (10) Å
c = 10.486 (2) Å
β = 99.75 (2)°
V = 906.8 (3) Å^3
Z = 4
$F(000)$ = 392
D_x = 1.349 Mg m^{-3}
Ag $K\alpha$ radiation, λ = 0.56083 Å
Cell parameters from 25 reflections
θ = 9–11°
μ = 0.06 mm^{-1}
T = 293 K
Prism, colorless
0.40 × 0.35 × 0.30 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
non–profiled ω scans
6579 measured reflections
4430 independent reflections
2415 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 28.0°, θ_{min} = 2.2°
h = −2→25
k = −9→2
l = −17→17
2 standard reflections every 120 min
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.071
$wR(F^2)$ = 0.216
S = 0.96
4430 reflections
122 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

$w = 1/[\sigma^2(F_o^2) + (0.0879P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.011$

$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-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 $F^2 > \sigma(F^2)$ 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}
O3	0.42764 (9)	0.7571 (2)	0.58898 (11)	0.0491 (4)
N2	0.40982 (10)	0.7432 (3)	0.70119 (15)	0.0495 (4)
N1	0.40818 (11)	1.2425 (3)	0.52659 (18)	0.0576 (5)
H1A	0.4136	1.3250	0.5990	0.086*
H1B	0.4500	1.2859	0.4811	0.086*
H1C	0.4151	1.0940	0.5464	0.086*
C6	0.20891 (13)	1.2197 (3)	0.60241 (18)	0.0485 (5)
H6	0.2267	1.3600	0.6411	0.058*
C2	0.11570 (12)	0.8824 (3)	0.59300 (17)	0.0455 (5)
C7	0.14446 (13)	1.0904 (4)	0.64823 (18)	0.0510 (5)
H7	0.1198	1.1447	0.7180	0.061*
C5	0.24736 (12)	1.1432 (3)	0.49952 (16)	0.0420 (4)
C4	0.21981 (13)	0.9329 (3)	0.44485 (18)	0.0494 (5)
H4	0.2453	0.8768	0.3763	0.059*
C8	0.31895 (14)	1.2799 (4)	0.44969 (19)	0.0538 (5)
H8A	0.3200	1.2369	0.3606	0.065*
H8B	0.3042	1.4423	0.4511	0.065*
C3	0.15502 (13)	0.8059 (3)	0.49093 (19)	0.0517 (5)
H3	0.1373	0.6654	0.4525	0.062*
O1	0.39214 (12)	0.5519 (3)	0.74280 (15)	0.0818 (6)
O2	0.41003 (13)	0.9155 (3)	0.76752 (16)	0.0859 (6)
C1	0.04360 (14)	0.7443 (4)	0.6414 (2)	0.0632 (6)
H1D	0.0477	0.7676	0.7329	0.095*
H1E	0.0516	0.5840	0.6244	0.095*
H1F	−0.0144	0.7933	0.5977	0.095*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.0556 (8)	0.0473 (8)	0.0478 (7)	0.0014 (6)	0.0184 (6)	0.0008 (6)
N2	0.0483 (9)	0.0536 (10)	0.0499 (9)	0.0024 (8)	0.0175 (7)	−0.0024 (8)
N1	0.0558 (10)	0.0390 (9)	0.0870 (12)	−0.0007 (8)	0.0379 (9)	0.0063 (9)

C6	0.0555 (11)	0.0418 (10)	0.0504 (10)	−0.0018 (9)	0.0157 (9)	−0.0056 (9)
C2	0.0400 (9)	0.0471 (11)	0.0494 (10)	0.0003 (9)	0.0073 (8)	0.0096 (9)
C7	0.0523 (11)	0.0537 (12)	0.0506 (10)	0.0038 (10)	0.0197 (9)	−0.0026 (9)
C5	0.0471 (10)	0.0377 (9)	0.0428 (9)	−0.0005 (9)	0.0117 (8)	0.0029 (8)
C4	0.0628 (12)	0.0432 (11)	0.0459 (10)	0.0024 (10)	0.0195 (9)	−0.0029 (8)
C8	0.0662 (13)	0.0471 (12)	0.0531 (10)	−0.0053 (10)	0.0245 (10)	0.0021 (9)
C3	0.0607 (12)	0.0409 (11)	0.0541 (11)	−0.0055 (9)	0.0116 (10)	−0.0006 (9)
O1	0.1100 (14)	0.0670 (11)	0.0772 (11)	−0.0136 (10)	0.0413 (10)	0.0139 (9)
O2	0.1169 (15)	0.0737 (12)	0.0739 (10)	0.0027 (11)	0.0353 (10)	−0.0280 (9)
C1	0.0542 (12)	0.0675 (15)	0.0705 (13)	−0.0100 (11)	0.0178 (11)	0.0083 (12)

Geometric parameters (Å, º)

O3—N2	1.2533 (19)	C2—C1	1.508 (3)
N2—O2	1.219 (2)	C7—H7	0.9300
N2—O1	1.240 (2)	C5—C4	1.384 (3)
N1—C8	1.464 (3)	C5—C8	1.505 (2)
N1—H1A	0.8900	C4—C3	1.376 (3)
N1—H1B	0.8900	C4—H4	0.9300
N1—H1C	0.8900	C8—H8A	0.9700
C6—C7	1.379 (3)	C8—H8B	0.9700
C6—C5	1.383 (2)	C3—H3	0.9300
C6—H6	0.9300	C1—H1D	0.9600
C2—C7	1.378 (3)	C1—H1E	0.9600
C2—C3	1.382 (3)	C1—H1F	0.9600
O2—N2—O1	121.05 (17)	C4—C5—C8	120.39 (16)
O2—N2—O3	120.18 (18)	C3—C4—C5	120.63 (17)
O1—N2—O3	118.77 (17)	C3—C4—H4	119.7
C8—N1—H1A	109.5	C5—C4—H4	119.7
C8—N1—H1B	109.5	N1—C8—C5	112.22 (16)
H1A—N1—H1B	109.5	N1—C8—H8A	109.2
C8—N1—H1C	109.5	C5—C8—H8A	109.2
H1A—N1—H1C	109.5	N1—C8—H8B	109.2
H1B—N1—H1C	109.5	C5—C8—H8B	109.2
C7—C6—C5	120.72 (18)	H8A—C8—H8B	107.9
C7—C6—H6	119.6	C4—C3—C2	121.56 (19)
C5—C6—H6	119.6	C4—C3—H3	119.2
C7—C2—C3	117.47 (17)	C2—C3—H3	119.2
C7—C2—C1	121.30 (18)	C2—C1—H1D	109.5
C3—C2—C1	121.2 (2)	C2—C1—H1E	109.5
C2—C7—C6	121.50 (18)	H1D—C1—H1E	109.5
C2—C7—H7	119.3	C2—C1—H1F	109.5
C6—C7—H7	119.3	H1D—C1—H1F	109.5
C6—C5—C4	118.11 (17)	H1E—C1—H1F	109.5
C6—C5—C8	121.48 (17)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1^{i}	0.89	2.07	2.936 (3)	164
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N1—H1*C*···O3	0.89	2.01	2.900 (2)	179
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C8—H8*A*···O1^{iii}	0.97	2.45	3.234 (2)	138

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