

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

4-Methylbenzylammonium nitrate

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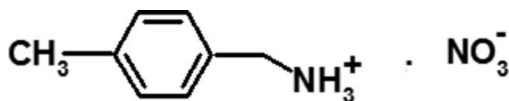
Received 13 August 2013; accepted 13 August 2013

 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.071; wR factor = 0.216; data-to-parameter ratio = 36.3.

In the title salt, $\text{C}_8\text{H}_{12}\text{N}^+\text{NO}_3^-$, 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 $\text{N}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots(\text{O},\text{O})$ hydrogen bonds, generating a layered network parallel to (100). A weak $\text{C}-\text{H}\cdots\text{O}$ 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 $\pi-\pi$ stacking interactions, see: Janiak (2000). For graph-set notation of hydrogen-bonding patterns, see: Bernstein *et al.* (1995).



Experimental

Crystal data

 $\text{C}_8\text{H}_{12}\text{N}^+\text{NO}_3^-$
 $M_r = 184.20$

 Monoclinic, $P2_1/c$
 $a = 15.097$ (2) Å

 $b = 5.8121$ (10) Å

 $c = 10.486$ (2) Å

 $\beta = 99.75$ (2)°

 $V = 906.8$ (3) Å³
 $Z = 4$

 Ag $K\alpha$ radiation

 $\lambda = 0.56083$ Å

 $\mu = 0.06$ mm⁻¹
 $T = 293$ K

 $0.40 \times 0.35 \times 0.30$ mm

Data collection

 Enraf–Nonius CAD-4
diffractometer

6579 measured reflections

4430 independent reflections

 2415 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.033$

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

 $\Delta\rho_{\text{max}} = 0.31$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.17$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1A}\cdots\text{O1}^{\text{i}}$	0.89	2.07	2.936 (3)	164
$\text{N1}-\text{H1A}\cdots\text{O3}^{\text{i}}$	0.89	2.52	3.065 (2)	120
$\text{N1}-\text{H1B}\cdots\text{O3}^{\text{ii}}$	0.89	2.12	2.9378 (19)	153
$\text{N1}-\text{H1C}\cdots\text{O3}$	0.89	2.01	2.900 (2)	179
$\text{N1}-\text{H1C}\cdots\text{O2}$	0.89	2.55	3.158 (3)	126
$\text{C8}-\text{H8A}\cdots\text{O1}^{\text{iii}}$	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).

This work was supported by the Tunisian Ministry of HEScR.

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

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supplementary materials

Acta Cryst. (2013). E69, o1453 [doi:10.1107/S1600536813022836]

4-Methylbenzylammonium nitrate

Sofian Gatfaoui, Houda Marouani and Mohamed Rzaigui

1. Comment

We report here the preparation and the crystal structure of the title compound, C₈H₁₂N·NO₃ (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.

2. Experimental

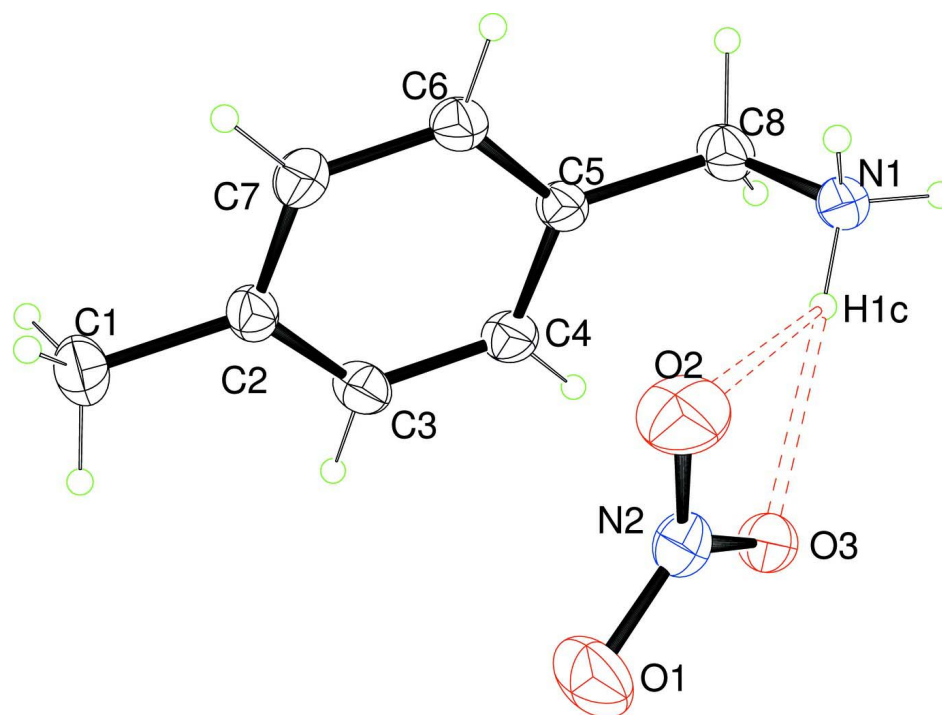
An aqueous solution containing 1 mmol of HNO₃ in 10 ml of water, was added to 1 mmol of 4-xylylamine in 10 ml of ethanol. The obtained solution was stirred for 20 min and then left to stand at room temperature. Colorless prisms of the title compound were obtained after some days.

3. Refinement

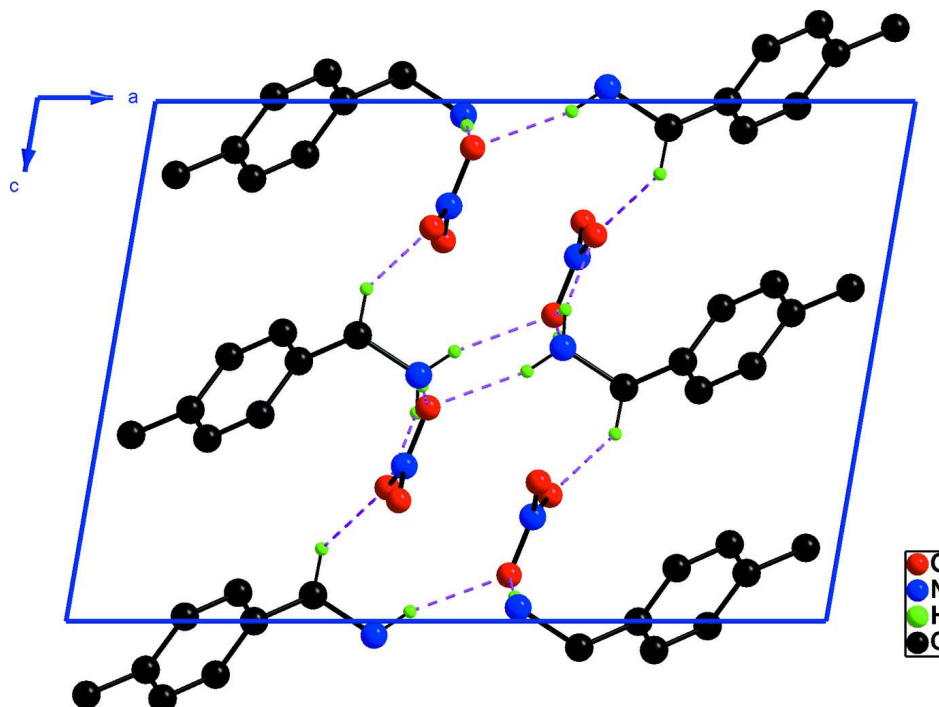
All H atoms were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic) or 0.97 Å (methylene) or 0.96 Å (methyl), N—H = 0.89 Å with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C or N})$.

Computing details

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); 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).

**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.

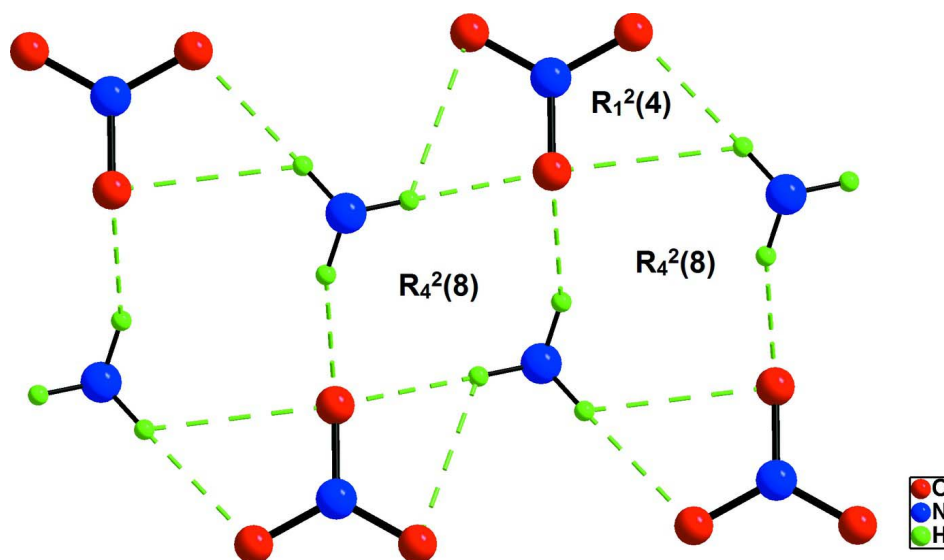


Figure 3
Hydrogen bond motifs in (I).

4-Methylbenzylammonium nitrate

Crystal data

$C_8H_{12}N^+ \cdot NO_3^-$

$M_r = 184.20$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2ybc$

$a = 15.097\ (2)\ \text{\AA}$

$b = 5.8121\ (10)\ \text{\AA}$

$c = 10.486\ (2)\ \text{\AA}$

$\beta = 99.75\ (2)^\circ$

$V = 906.8\ (3)\ \text{\AA}^3$

$Z = 4$

$F(000) = 392$

$D_x = 1.349\ \text{Mg m}^{-3}$

Ag $K\alpha$ radiation, $\lambda = 0.56083\ \text{\AA}$

Cell parameters from 25 reflections

$\theta = 9\text{--}11^\circ$

$\mu = 0.06\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Prism, colorless

$0.40 \times 0.35 \times 0.30\ \text{mm}$

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 $I > 2\sigma(I)$

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

$\theta_{\text{max}} = 28.0^\circ$, $\theta_{\text{min}} = 2.2^\circ$

$h = -2 \rightarrow 25$

$k = -9 \rightarrow 2$

$l = -17 \rightarrow 17$

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 $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.011$

$$\Delta\rho_{\max} = 0.31 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.17 \text{ e } \text{\AA}^{-3}$$

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
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 (\AA^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, °)

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, °)

<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—H1A \cdots O1 ⁱ	0.89	2.07	2.936 (3)	164
N1—H1A \cdots 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
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Symmetry codes: (i) $x, y+1, z$; (ii) $-x+1, -y+2, -z+1$; (iii) $x, -y+3/2, z-1/2$.