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N-[2-(2-Chlorophenyl)-2-hydroxyethyl]-propan-2-aminium nitrate

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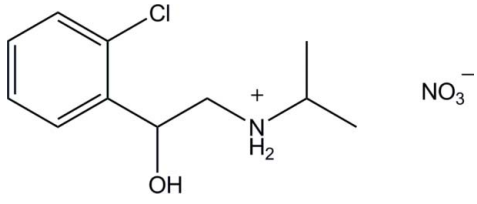
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.045; wR factor = 0.145; data-to-parameter ratio = 19.0.

In the title compound, $C_{11}H_{17}ClNO^+\cdot NO_3^-$, the side chain of the ethylammonium group is orientated approximately perpendicular to the benzene ring, the dihedral angle between the C/C/N plane of the ethylammonium group and the benzene ring being 79.40 (18)°. In the crystal structure, intermolecular O—H···O and N—H···O hydrogen bonds are observed between the cation and the anion.

Related literature

For related structures, see: Tang, Xu, Zhang & Feng (2009); Tang, Xu, Zheng & Feng (2009).



Experimental

Crystal data

$C_{11}H_{17}ClNO^+\cdot NO_3^-$
M_r = 276.72
Monoclinic, $P2_1/n$
a = 11.9551 (6) Å
b = 10.4563 (5) Å
c = 12.2968 (7) Å
β = 115.109 (1)°
V = 1391.91 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.28 mm^{-1}
T = 296 K
0.38 × 0.36 × 0.22 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.900, T_{max} = 0.940
13380 measured reflections
3179 independent reflections
1833 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.145
S = 1.00
3179 reflections
167 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H2A···O4	0.90	1.97	2.843 (2)	163
N1—H2B···O2^{i}	0.90	1.93	2.8234 (19)	170
O1—H101···O4^{ii}	0.82	1.98	2.7614 (19)	158

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

Data collection: *PROCESS-AUTO* (Rigaku/MSC, 2006); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2007); 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, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2504).

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Tang, Z., Xu, M., Zhang, H.-C. & Feng, H. (2009). *Acta Cryst.* E**65**, o1670.
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supporting information

Acta Cryst. (2010). E**66**, o391 [https://doi.org/10.1107/S1600536809054506]

N-[2-(2-Chlorophenyl)-2-hydroxyethyl]propan-2-aminium nitrate

Hai Feng, Zhan Tang, Lin-Jun Xie and Bin-Tao Xing

S1. Comment

A recent study reports the structure of bis{*N*-[2-(2-chlorophenyl)-2-hydroxyethyl]propan-2-aminium} oxalate (Tang, Xu, Zhang & Feng, 2009), which was synthesized by oxalic acid and clorprenaline (Tang, Xu, Zheng & Feng, 2009). Here using nitric acid instead of oxalic acid and following a similar synthetic procedure yields the title compound, (I).

In the molecular structure (Fig. 1), the Cl atom and the phenyl plane is almost planar with a deviation of 0.0118 Å. The dihedral angle between the plane formed by C1/C2/C8 and the benzene plane is 81.23 (18)°, which shows that the two planes are almost perpendicular. O—H···O and N—H···O hydrogen bonds are found in the crystal structure.

S2. Experimental

Racemic clorprenaline was prepared by clorprenaline hydrochloride purchased from ShangHai Shengxin Medicine & Chemical Co., Ltd. ShangHai, China. Clorprenaline hydrochloride and NaOH in a molar ratio of 1:1 were mixed and dissolved in a methanol-water solution (1:1 *v*/*v*). The precipitate formed was filtered off, washed with water and dried. It was used without further purification. Racemic clorprenaline (3.0 g, 0.014 mol) was dissolved in ethanol (30 ml), then nitric acid was added to give pH of about 2. The resulting solution was concentrated and colorless crystals of (I) were obtained within one day at ambient temperature.

S3. Refinement

All H atoms were placed in calculated positions and allowed to ride on their parent atoms, with C—H = 0.93 (aromatic), 0.98 (methine), 0.97 (methylene), 0.96 Å (methyl), O—H = 0.82 Å and N—H = 0.90 Å, and with U_{iso}(H) = 1.2–1.5 times U_{eq} of the parent atoms.

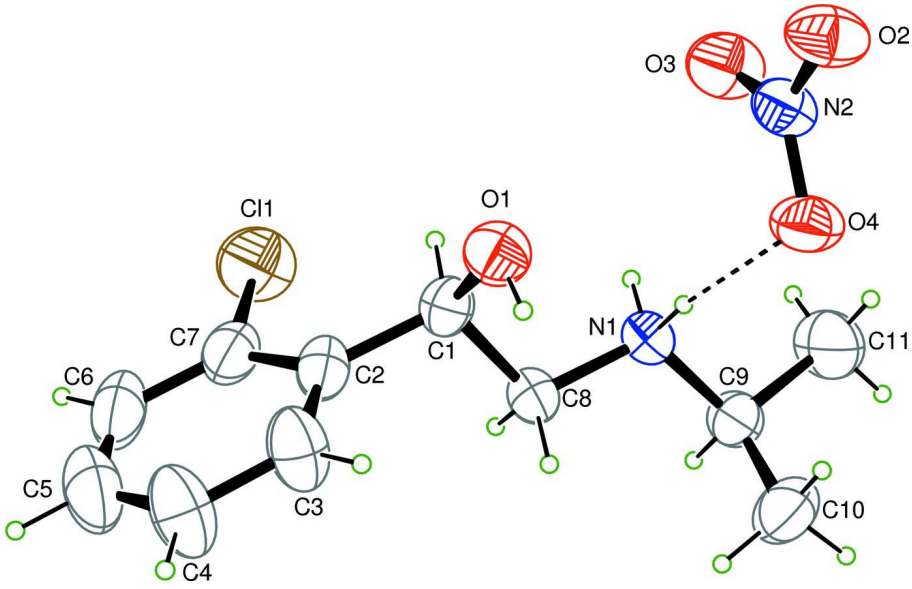

Figure 1

The molecular structure of the title compound, with atom labels, showing 40% probability displacement ellipsoids.

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Crystal data

$C_{11}H_{17}ClNO^{+}\cdot NO_3^{-}$
$M_r = 276.72$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 11.9551\ (6)$ Å
$b = 10.4563\ (5)$ Å
$c = 12.2968\ (7)$ Å
$\beta = 115.109\ (1)°$
$V = 1391.91\ (12)$ Å^3
$Z = 4$
$F(000) = 584$
$D_x = 1.320$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7750 reflections
$\theta = 3.1–27.4°$
$\mu = 0.28$ mm^{-1}
$T = 296$ K
Chunk, colorless
0.38 × 0.36 × 0.22 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Radiation source: rolling anode
Graphite monochromator
Detector resolution: 10.00 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.900$, $T_{max} = 0.940$
13380 measured reflections
3179 independent reflections
1833 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 27.4°$, $\theta_{min} = 3.1°$
$h = -15\rightarrow15$
$k = -13\rightarrow13$
$l = -15\rightarrow15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.145$
$S = 1.00$
3179 reflections
167 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
$w = 1/[\sigma^2(F_o^2) + (0.0508P)^2 + 0.8267P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.49$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.031 (2)

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}
Cl1	0.50104 (7)	0.95849 (7)	0.13939 (6)	0.1022 (3)
N2	0.28600 (14)	0.45390 (15)	0.33777 (14)	0.0580 (4)
N1	0.37095 (12)	0.76881 (13)	0.40807 (13)	0.0468 (4)
H2A	0.3748	0.6832	0.4169	0.056*
H2B	0.3128	0.7862	0.3339	0.056*
C8	0.49200 (14)	0.81542 (17)	0.41706 (16)	0.0489 (4)
H8A	0.5552	0.7990	0.4973	0.059*
H8B	0.4877	0.9071	0.4039	0.059*
O1	0.53694 (12)	0.61603 (12)	0.34203 (12)	0.0634 (4)
H101	0.5812	0.5995	0.4126	0.095*
O2	0.28728 (13)	0.33459 (13)	0.33095 (13)	0.0724 (5)
O3	0.23497 (15)	0.52046 (14)	0.24813 (13)	0.0787 (5)
O4	0.33694 (14)	0.50469 (14)	0.43991 (12)	0.0734 (4)
C9	0.33109 (16)	0.82528 (19)	0.49838 (17)	0.0568 (5)
H8	0.3382	0.9186	0.4967	0.068*
C1	0.52762 (16)	0.75053 (17)	0.32594 (16)	0.0521 (4)
H1	0.4615	0.7673	0.2461	0.063*
C2	0.64380 (17)	0.81197 (18)	0.33057 (17)	0.0560 (5)
C7	0.6422 (2)	0.9062 (2)	0.25042 (18)	0.0650 (5)
C3	0.75844 (18)	0.7782 (2)	0.4181 (2)	0.0776 (7)
H3	0.7634	0.7155	0.4735	0.093*
C10	0.4135 (2)	0.7788 (3)	0.62253 (19)	0.0801 (7)
H9A	0.4150	0.6870	0.6232	0.096*
H9B	0.3825	0.8089	0.6782	0.096*
H9C	0.4957	0.8109	0.6455	0.096*
C6	0.7495 (2)	0.9625 (2)	0.2564 (2)	0.0830 (6)
H6	0.7457	1.0243	0.2006	0.100*
C5	0.8610 (2)	0.9267 (3)	0.3447 (3)	0.0923 (7)
H5	0.9333	0.9647	0.3496	0.111*
C11	0.19628 (19)	0.7912 (3)	0.4610 (2)	0.0950 (9)

H10A	0.1473	0.8255	0.3826	0.114*
H10B	0.1690	0.8266	0.5176	0.114*
H10C	0.1874	0.6999	0.4590	0.114*
C4	0.8659 (2)	0.8349 (3)	0.4258 (3)	0.0942 (9)
H4	0.9416	0.8106	0.4861	0.113*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.1190 (5)	0.0985 (5)	0.0691 (3)	−0.0122 (4)	0.0207 (3)	0.0293 (3)
N2	0.0537 (8)	0.0524 (9)	0.0573 (9)	−0.0069 (7)	0.0134 (7)	0.0004 (7)
N1	0.0444 (7)	0.0437 (7)	0.0513 (7)	−0.0031 (6)	0.0193 (6)	−0.0005 (6)
C8	0.0448 (8)	0.0489 (9)	0.0546 (9)	−0.0036 (7)	0.0225 (7)	−0.0004 (7)
O1	0.0721 (8)	0.0509 (7)	0.0686 (8)	−0.0004 (6)	0.0312 (6)	−0.0032 (6)
O2	0.0794 (9)	0.0485 (7)	0.0677 (8)	0.0020 (7)	0.0105 (7)	−0.0035 (6)
O3	0.0865 (10)	0.0630 (8)	0.0633 (8)	−0.0017 (8)	0.0093 (7)	0.0125 (7)
O4	0.0934 (10)	0.0564 (8)	0.0567 (8)	−0.0187 (7)	0.0185 (7)	−0.0061 (6)
C9	0.0538 (9)	0.0598 (11)	0.0636 (10)	−0.0034 (8)	0.0315 (8)	−0.0127 (9)
C1	0.0557 (9)	0.0533 (10)	0.0497 (9)	0.0023 (8)	0.0247 (7)	0.0062 (8)
C2	0.0629 (9)	0.0551 (10)	0.0619 (10)	0.0007 (8)	0.0378 (8)	0.0024 (8)
C7	0.0842 (12)	0.0617 (12)	0.0610 (10)	−0.0040 (10)	0.0422 (9)	−0.0010 (9)
C3	0.0563 (10)	0.0833 (15)	0.0978 (15)	0.0062 (11)	0.0371 (10)	0.0233 (12)
C10	0.0859 (13)	0.1027 (18)	0.0609 (11)	−0.0001 (13)	0.0401 (10)	−0.0077 (11)
C6	0.1148 (14)	0.0703 (14)	0.0984 (13)	−0.0136 (13)	0.0785 (11)	−0.0028 (11)
C5	0.0847 (12)	0.0848 (16)	0.1380 (19)	−0.0117 (13)	0.0768 (13)	−0.0115 (15)
C11	0.0604 (11)	0.130 (2)	0.1072 (17)	−0.0126 (13)	0.0480 (11)	−0.0340 (16)
C4	0.0587 (11)	0.1000 (19)	0.132 (2)	0.0021 (13)	0.0478 (13)	0.0155 (16)

Geometric parameters (Å, º)

Cl1—C7	1.748 (2)	C1—H1	0.9800
N2—O3	1.225 (2)	C2—C3	1.382 (3)
N2—O2	1.251 (2)	C2—C7	1.388 (3)
N2—O4	1.258 (2)	C7—C6	1.385 (3)
N1—C8	1.486 (2)	C3—C4	1.381 (3)
N1—C9	1.503 (2)	C3—H3	0.9300
N1—H2A	0.9000	C10—H9A	0.9600
N1—H2B	0.9000	C10—H9B	0.9600
C8—C1	1.517 (3)	C10—H9C	0.9600
C8—H8A	0.9700	C6—C5	1.366 (3)
C8—H8B	0.9700	C6—H6	0.9300
O1—C1	1.418 (2)	C5—C4	1.368 (4)
O1—H101	0.8200	C5—H5	0.9300
C9—C10	1.504 (3)	C11—H10A	0.9600
C9—C11	1.519 (3)	C11—H10B	0.9600
C9—H8	0.9800	C11—H10C	0.9600
C1—C2	1.509 (3)	C4—H4	0.9300

O3—N2—O2	121.46 (16)	C3—C2—C1	120.90 (18)
O3—N2—O4	120.26 (16)	C7—C2—C1	122.68 (17)
O2—N2—O4	118.27 (16)	C6—C7—C2	122.1 (2)
C8—N1—C9	114.86 (13)	C6—C7—Cl1	118.28 (17)
C8—N1—H2A	108.6	C2—C7—Cl1	119.66 (16)
C9—N1—H2A	108.6	C4—C3—C2	122.0 (2)
C8—N1—H2B	108.6	C4—C3—H3	119.0
C9—N1—H2B	108.6	C2—C3—H3	119.0
H2A—N1—H2B	107.5	C9—C10—H9A	109.5
N1—C8—C1	111.59 (14)	C9—C10—H9B	109.5
N1—C8—H8A	109.3	H9A—C10—H9B	109.5
C1—C8—H8A	109.3	C9—C10—H9C	109.5
N1—C8—H8B	109.3	H9A—C10—H9C	109.5
C1—C8—H8B	109.3	H9B—C10—H9C	109.5
H8A—C8—H8B	108.0	C5—C6—C7	119.7 (2)
C1—O1—H101	109.5	C5—C6—H6	120.2
N1—C9—C10	110.28 (16)	C7—C6—H6	120.2
N1—C9—C11	108.21 (16)	C6—C5—C4	119.8 (2)
C10—C9—C11	112.7 (2)	C6—C5—H5	120.1
N1—C9—H8	108.5	C4—C5—H5	120.1
C10—C9—H8	108.5	C9—C11—H10A	109.5
C11—C9—H8	108.5	C9—C11—H10B	109.5
O1—C1—C2	113.60 (15)	H10A—C11—H10B	109.5
O1—C1—C8	111.75 (15)	C9—C11—H10C	109.5
C2—C1—C8	108.90 (15)	H10A—C11—H10C	109.5
O1—C1—H1	107.4	H10B—C11—H10C	109.5
C2—C1—H1	107.4	C5—C4—C3	120.0 (2)
C8—C1—H1	107.4	C5—C4—H4	120.0
C3—C2—C7	116.40 (19)	C3—C4—H4	120.0

C9—N1—C8—C1	177.99 (14)	C1—C2—C7—C6	179.4 (2)
C8—N1—C9—C10	−68.4 (2)	C3—C2—C7—Cl1	−178.09 (18)
C8—N1—C9—C11	167.93 (18)	C1—C2—C7—Cl1	0.5 (3)
N1—C8—C1—O1	−59.77 (18)	C7—C2—C3—C4	0.0 (4)
N1—C8—C1—C2	173.93 (14)	C1—C2—C3—C4	−178.7 (2)
O1—C1—C2—C3	−44.5 (3)	C2—C7—C6—C5	−1.1 (4)
C8—C1—C2—C3	80.7 (2)	Cl1—C7—C6—C5	177.8 (2)
O1—C1—C2—C7	136.94 (19)	C7—C6—C5—C4	0.6 (4)
C8—C1—C2—C7	−97.8 (2)	C6—C5—C4—C3	0.2 (4)
C3—C2—C7—C6	0.8 (3)	C2—C3—C4—C5	−0.5 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H2*A*···O4	0.90	1.97	2.843 (2)	163

N1—H2*B*···O2^{i}	0.90	1.93	2.8234 (19)	170
O1—H101···O4^{ii}	0.82	1.98	2.7614 (19)	158

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