Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-(2-Hydroxybenzyl)adamantan-1-aminium chloride

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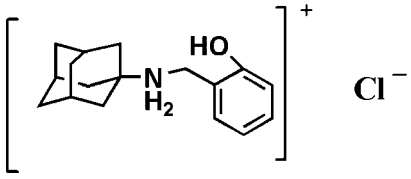
Received 23 April 2011; accepted 30 May 2011

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.074; wR factor = 0.217; data-to-parameter ratio = 17.8.

The asymmetric unit of the title compound, $C_{17}H_{24}NO^{+}\cdot Cl^{-}$, consists of a discrete *N*-(2-hydroxybenzyl)adamantan-1-aminium cation and a Cl^{-} anion. Intermolecular N—H···Cl and O—H···Cl hydrogen bonds occurring between the organic cation and the Cl^{-} anion generate a layered structure.

Related literature

For general background to ferroelectric organic frameworks, see: Ye *et al.* (2006, 2009); Fu *et al.* (2007); Zhao *et al.* (2008). For phase transitions of ferroelectric materials, see: Zhang *et al.* (2008).



Experimental

Crystal data

$C_{17}H_{24}NO^{+}\cdot Cl^{-}$
M_r = 293.82
Monoclinic, $P2_1/c$
a = 12.262 (3) Å
b = 10.202 (2) Å
c = 12.845 (3) Å
β = 98.43 (3)°
V = 1589.5 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 293 K
0.20 × 0.20 × 0.20 mm

Data collection

Rigaku SCXmini diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.954, T_{max} = 0.954
15541 measured reflections
3632 independent reflections
2249 reflections with $I > 2\sigma(I)$
R_{int} = 0.088

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.217
S = 1.06
3632 reflections
204 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···Cl1^{i}	0.90	2.58	3.260 (2)	133
N1—H1C···Cl1^{ii}	0.90	2.38	3.118 (2)	139
O1—H1B···Cl1^{iii}	0.85	2.27	3.049 (2)	152

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *SHELXL97*.

The authors are grateful to the Starter Fund of Southeast University, Nanjing, People's Republic of China.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KP2325).

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supporting information

Acta Cryst. (2011). E**67**, o1602 [doi:10.1107/S1600536811020794]

N-(2-Hydroxybenzyl)adamantan-1-aminium chloride

Tao Rong

S1. Comment

The study of ferroelectric materials has received much attention. Some materials have predominantly dielectric-ferroelectric performances.The title compound was studied as part of our work to obtain potential ferroelectric phase-change materials (Ye *et al.*, 2006; Fu *et al.*, 2007; Zhao *et al.* 2008; Zhang *et al.*, 2008; Ye *et al.*, 2009). Unluckily, the compound has no dielectric anomalies in the temperature range 93–453 K, suggesting that it might be only a paraelectric. The title compound (Fig. 1), consists of protonated N-(2-hydroxybenzyl)–1–adamantylammonium cation and Cl^- anion. In the crystal structure, the N—H···Cl and O—H···Cl hydrogen bonds between the organic cations and the Cl^- anions stabilise crystal packing. In the cation, the groups NH_2^+ and OH are proton donors to Cl^- forming hydrogen bonds (Table 1, Fig. 2).

S2. Experimental

KOH (20 mmol)and salicylaldehyde (20 mmol) were added into a solution of amantadine hydrochloride (20 mmol) in ethanol. Then a little of anhydrous magnesium sulfate was added into the mixture, after 6 h the yellow precipitate came out. The yellow solid of amantadine shrink Yang Schiff was obtained by filtration, collection and drying.

$NaBH_4$(3.78 g) was added into a solution of amantadine shrink Yang Schiff (25 mmol) in anhydrous methanol(120 mL). After 5 h reaction, a white solid 2-(adamantane-1-aminomethyl)phenol was obtained by reduced pressure distillation, extraction and drying.

A solution of hydrochloric acid (10 mmol) was added to a solution of 2-(adamantane-1-aminomethyl)phenol (10 mmol) in ethanol (20 mL). Crystals suitable for structure determination were grown by slow evaporation of the mixture at room temperature.

S3. Refinement

Positional parameters of all the H atoms bonded to C atoms were calculated geometrically and were allowed to ride on the C atoms to which they are bonded, with U_{iso}(H) = 1.2Ueq(C) and U_{iso}(H) = 1.5Ueq(C) for the methyl group. The other H bonded to O/N atoms were calculated geometrically and were allowed to ride on the O/N atoms with U_{iso}(H) = 1.2Ueq(N) and U_{iso}(H) = 1.5Ueq(O).

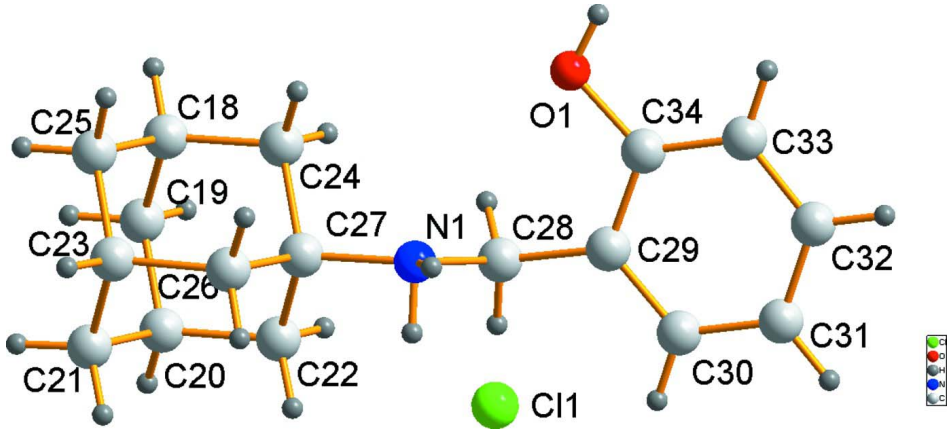

Figure 1

The molecular structure of the title compound with the atomic numbering scheme.

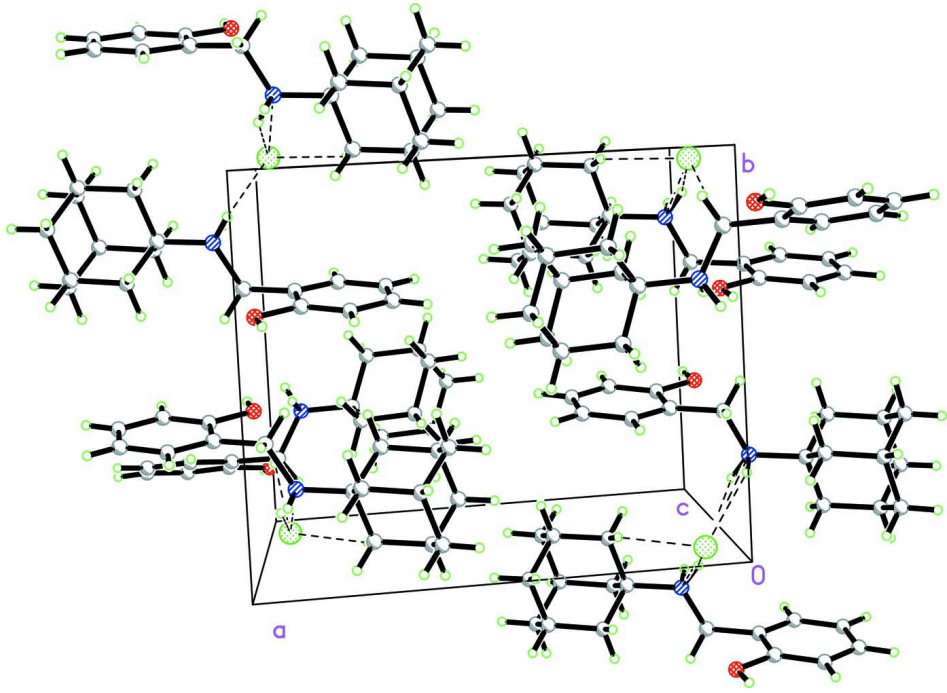

Figure 2

A view of the packing of the title compound, showing hydrogen-bonded helices along the axis b.

N-(2-Hydroxybenzyl)adamantan-1-aminium chloride

Crystal data

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$M_r = 293.82$
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$c = 12.845\ (3)$ Å
$\beta = 98.43\ (3)°$
$V = 1589.5\ (6)$ Å^3
$Z = 4$
$F(000) = 632$
$D_x = 1.228$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$\theta = 3.0–27.5°$
$\mu = 0.24$ mm^{-1}
$T = 293$ K
Prism, colourless
0.20 × 0.20 × 0.20 mm

Data collection

Rigaku SCXmini diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm^{-1}
CCD_Profile_fitting scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.954, T_{max} = 0.954

15541 measured reflections
3632 independent reflections
2249 reflections with $I > 2\sigma(I)$
R_{int} = 0.088
θ_{max} = 27.5°, θ_{min} = 3.2°
h = −15→15
k = −13→13
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.217
S = 1.06
3632 reflections
204 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
$w = 1/[\sigma^2(F_o^2) + (0.0986P)^2 + 0.3244P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

Special details

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	0.04801 (8)	0.97166 (8)	0.33131 (6)	0.0627 (3)
O1	−0.01490 (19)	0.8664 (2)	−0.12965 (18)	0.0641 (7)
H1B	−0.0321	0.8850	−0.1947	0.15 (2)*
N1	0.07621 (18)	0.6830 (2)	0.05578 (17)	0.0391 (5)
H1C	0.0596	0.6471	0.1155	0.047*
H1A	0.0400	0.6179	0.0181	0.23 (3)*
C18	0.3843 (3)	0.7592 (5)	0.0829 (4)	0.0832 (12)
H18A	0.4245	0.8220	0.0479	0.113 (16)*
C19	0.4046 (3)	0.7838 (4)	0.1997 (4)	0.0854 (13)
H19A	0.4819	0.7749	0.2250	0.093 (13)*
H19B	0.3827	0.8715	0.2141	0.086 (12)*
C20	0.3412 (3)	0.6858 (4)	0.2566 (3)	0.0709 (11)
H20A	0.3543	0.7027	0.3309	0.089 (12)*
C21	0.3796 (3)	0.5474 (4)	0.2345 (3)	0.0752 (11)
H21A	0.4562	0.5379	0.2621	0.072 (11)*
H21B	0.3384	0.4840	0.2679	0.100 (15)*
C22	0.2176 (3)	0.7000 (4)	0.2164 (2)	0.0604 (9)
H22A	0.1762	0.6394	0.2523	0.086 (13)*
H22B	0.1938	0.7873	0.2297	0.079 (12)*

C23	0.3609 (3)	0.5257 (4)	0.1154 (3)	0.0704 (11)
H23A	0.3858	0.4396	0.0999	0.095 (13)*
C24	0.2618 (3)	0.7734 (4)	0.0421 (3)	0.0679 (10)
H24A	0.2377	0.8606	0.0551	0.111 (16)*
H24B	0.2479	0.7574	−0.0323	0.103 (15)*
C25	0.4232 (3)	0.6267 (5)	0.0625 (4)	0.0852 (13)
H25A	0.4139	0.6101	−0.0119	0.130 (19)*
H25B	0.5004	0.6207	0.0892	0.093 (13)*
C26	0.2374 (3)	0.5371 (3)	0.0762 (3)	0.0639 (10)
H26A	0.2226	0.5201	0.0020	0.087 (13)*
H26B	0.1981	0.4737	0.1116	0.052 (9)*
C27	0.1979 (2)	0.6736 (3)	0.0991 (2)	0.0409 (6)
C28	0.0184 (3)	0.8077 (3)	0.0800 (2)	0.0500 (7)
H28A	0.0613	0.8815	0.0634	0.082 (12)*
H28B	0.0135	0.8109	0.1539	0.043 (8)*
C29	−0.0946 (2)	0.8170 (3)	0.0198 (2)	0.0437 (7)
C30	−0.1871 (3)	0.8027 (3)	0.0692 (3)	0.0625 (9)
H30A	−0.1780	0.7829	0.1430	0.082 (12)*
C31	−0.2914 (3)	0.8181 (4)	0.0146 (4)	0.0774 (11)
H31A	−0.3550	0.8068	0.0493	0.100 (15)*
C32	−0.3042 (3)	0.8490 (4)	−0.0905 (4)	0.0794 (12)
H32A	−0.3770	0.8604	−0.1288	0.105 (14)*
C33	−0.2136 (3)	0.8645 (3)	−0.1424 (3)	0.0653 (10)
H33A	−0.2225	0.8857	−0.2160	0.101 (14)*
C34	−0.1099 (3)	0.8494 (3)	−0.0868 (2)	0.0492 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.1004 (7)	0.0557 (5)	0.0339 (4)	0.0196 (4)	0.0159 (4)	0.0021 (3)
O1	0.0775 (16)	0.0677 (16)	0.0505 (14)	0.0146 (12)	0.0211 (11)	0.0142 (11)
N1	0.0488 (14)	0.0375 (12)	0.0313 (11)	0.0016 (10)	0.0073 (10)	0.0006 (9)
C18	0.056 (2)	0.090 (3)	0.107 (3)	−0.010 (2)	0.022 (2)	0.016 (3)
C19	0.055 (2)	0.062 (3)	0.133 (4)	−0.0044 (18)	−0.008 (2)	−0.019 (2)
C20	0.059 (2)	0.101 (3)	0.0477 (19)	0.014 (2)	−0.0074 (16)	−0.0226 (19)
C21	0.057 (2)	0.078 (3)	0.086 (3)	0.0117 (19)	−0.007 (2)	0.014 (2)
C22	0.061 (2)	0.083 (3)	0.0361 (16)	0.0120 (18)	0.0050 (15)	−0.0123 (16)
C23	0.067 (2)	0.055 (2)	0.085 (3)	0.0188 (17)	−0.003 (2)	−0.0219 (19)
C24	0.057 (2)	0.076 (3)	0.073 (3)	−0.0042 (18)	0.0178 (18)	0.017 (2)
C25	0.059 (3)	0.113 (4)	0.087 (3)	0.006 (2)	0.022 (2)	−0.006 (3)
C26	0.068 (2)	0.0471 (19)	0.073 (2)	0.0063 (16)	−0.0012 (18)	−0.0148 (17)
C27	0.0461 (16)	0.0396 (15)	0.0379 (14)	−0.0007 (12)	0.0091 (12)	−0.0024 (11)
C28	0.0632 (19)	0.0418 (17)	0.0436 (16)	0.0065 (14)	0.0024 (14)	−0.0075 (13)
C29	0.0478 (16)	0.0352 (14)	0.0481 (16)	0.0033 (12)	0.0072 (13)	0.0004 (12)
C30	0.067 (2)	0.059 (2)	0.066 (2)	0.0046 (16)	0.0223 (18)	0.0098 (17)
C31	0.053 (2)	0.070 (3)	0.112 (4)	−0.0011 (18)	0.020 (2)	0.005 (2)
C32	0.053 (2)	0.062 (2)	0.114 (4)	0.0038 (17)	−0.016 (2)	−0.006 (2)
C33	0.076 (2)	0.054 (2)	0.060 (2)	0.0130 (17)	−0.0111 (18)	−0.0020 (16)

C34	0.0628 (19)	0.0361 (15)	0.0481 (17)	0.0059 (13)	0.0058 (15)	−0.0010 (12)

Geometric parameters (Å, º)

O1—C34	1.370 (4)	C23—C26	1.529 (5)
O1—H1B	0.8530	C23—H23A	0.9600
N1—C28	1.511 (3)	C24—C27	1.535 (4)
N1—C27	1.517 (3)	C24—H24A	0.9601
N1—H1C	0.9000	C24—H24B	0.9600
N1—H1A	0.9001	C25—H25A	0.9600
C18—C25	1.470 (6)	C25—H25B	0.9600
C18—C19	1.505 (6)	C26—C27	1.517 (4)
C18—C24	1.524 (5)	C26—H26A	0.9601
C18—H18A	0.9599	C26—H26B	0.9601
C19—C20	1.518 (6)	C28—C29	1.488 (4)
C19—H19A	0.9600	C28—H28A	0.9601
C19—H19B	0.9599	C28—H28B	0.9600
C20—C21	1.528 (5)	C29—C30	1.386 (4)
C20—C22	1.534 (4)	C29—C34	1.394 (4)
C20—H20A	0.9600	C30—C31	1.375 (5)
C21—C23	1.529 (6)	C30—H30A	0.9600
C21—H21A	0.9600	C31—C32	1.373 (6)
C21—H21B	0.9599	C31—H31A	0.9601
C22—C27	1.515 (4)	C32—C33	1.386 (6)
C22—H22A	0.9599	C32—H32A	0.9602
C22—H22B	0.9600	C33—C34	1.373 (5)
C23—C25	1.504 (6)	C33—H33A	0.9598
C34—O1—H1B	108.6	C18—C24—H24B	110.4
C28—N1—C27	116.3 (2)	C27—C24—H24B	109.5
C28—N1—H1C	89.7	H24A—C24—H24B	108.4
C27—N1—H1C	89.7	C18—C25—C23	110.5 (3)
C28—N1—H1A	121.6	C18—C25—H25A	110.2
C27—N1—H1A	122.1	C23—C25—H25A	109.6
H1C—N1—H1A	90.2	C18—C25—H25B	108.9
C25—C18—C19	108.7 (4)	C23—C25—H25B	109.5
C25—C18—C24	110.8 (4)	H25A—C25—H25B	108.1
C19—C18—C24	109.8 (3)	C27—C26—C23	109.6 (3)
C25—C18—H18A	109.1	C27—C26—H26A	109.8
C19—C18—H18A	109.5	C23—C26—H26A	110.3
C24—C18—H18A	108.9	C27—C26—H26B	109.2
C18—C19—C20	110.4 (3)	C23—C26—H26B	109.5
C18—C19—H19A	109.5	H26A—C26—H26B	108.3
C20—C19—H19A	109.0	C22—C27—N1	111.0 (2)
C18—C19—H19B	109.7	C22—C27—C26	110.3 (3)
C20—C19—H19B	110.1	N1—C27—C26	108.2 (2)
H19A—C19—H19B	108.3	C22—C27—C24	109.7 (3)
C19—C20—C21	109.0 (3)	N1—C27—C24	109.1 (2)

C19—C20—C22	109.1 (3)	C26—C27—C24	108.5 (3)
C21—C20—C22	109.9 (3)	C29—C28—N1	112.1 (2)
C19—C20—H20A	109.7	C29—C28—H28A	109.4
C21—C20—H20A	109.9	N1—C28—H28A	109.0
C22—C20—H20A	109.3	C29—C28—H28B	108.8
C20—C21—C23	108.4 (3)	N1—C28—H28B	109.4
C20—C21—H21A	109.6	H28A—C28—H28B	108.0
C23—C21—H21A	110.5	C30—C29—C34	118.3 (3)
C20—C21—H21B	110.1	C30—C29—C28	121.2 (3)
C23—C21—H21B	109.8	C34—C29—C28	120.4 (3)
H21A—C21—H21B	108.5	C31—C30—C29	121.1 (3)
C27—C22—C20	108.9 (3)	C31—C30—H30A	119.5
C27—C22—H22A	110.2	C29—C30—H30A	119.4
C20—C22—H22A	110.1	C32—C31—C30	119.4 (4)
C27—C22—H22B	109.5	C32—C31—H31A	120.1
C20—C22—H22B	109.8	C30—C31—H31A	120.5
H22A—C22—H22B	108.4	C31—C32—C33	121.0 (3)
C25—C23—C21	110.0 (3)	C31—C32—H32A	119.6
C25—C23—C26	110.1 (3)	C33—C32—H32A	119.4
C21—C23—C26	108.3 (3)	C34—C33—C32	119.0 (4)
C25—C23—H23A	109.6	C34—C33—H33A	120.0
C21—C23—H23A	109.6	C32—C33—H33A	121.1
C26—C23—H23A	109.3	O1—C34—C33	123.7 (3)
C18—C24—C27	108.6 (3)	O1—C34—C29	115.1 (3)
C18—C24—H24A	110.0	C33—C34—C29	121.2 (3)
C27—C24—H24A	109.9		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···Cl1^{i}	0.90	2.58	3.260 (2)	133
N1—H1*C*···Cl1^{ii}	0.90	2.38	3.118 (2)	139
O1—H1*B*···Cl1^{iii}	0.85	2.27	3.049 (2)	152

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