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Adamantane-1-ammonium benzoate

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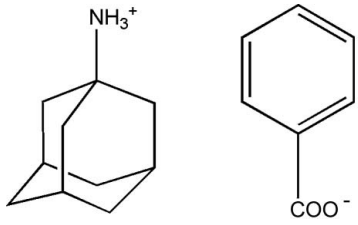
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.055; wR factor = 0.147; data-to-parameter ratio = 19.0.

In the title molecular salt, $C_{10}H_{15}NH_3^+\cdot C_7H_5O_2^-$, both carboxyl O atoms act as acceptors for strong N—H···O intermolecular hydrogen-bond interactions with the ammonium group in the cation, generating infinite chains along the b axis. A weak C—H···π interaction is also present.

Related literature

For related structures, see: Tukada & Mochizuki (2003); Zhao *et al.* (2003); He & Wen (2006). For puckering parameters, see: Cremer & Pople (1975).



Experimental

Crystal data

$C_{10}H_{18}N^+\cdot C_7H_5O_2^-$
M_r = 273.36
Monoclinic, $P2_1/n$
a = 10.918 (2) Å
b = 6.5664 (13) Å
c = 21.197 (4) Å
β = 100.07 (3)°
V = 1496.3 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 298 K
0.20 × 0.20 × 0.20 mm

Data collection

Rigaku SCXmini diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.774, T_{max} = 1.000
15027 measured reflections
3437 independent reflections
2453 reflections with $I > 2\sigma(I)$
R_{int} = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.147
S = 1.04
3437 reflections
181 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 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	1.83	2.7134 (17)	176
N1—H1B···O2^{ii}	0.89	1.90	2.7840 (18)	173
N1—H1C···O2	0.89	1.92	2.7915 (18)	166
C16—H16A···$Cg1^{iii}$	0.97	2.74	3.702 (2)	174

Symmetry codes: (i) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (iii) $x+1, y, z$. $Cg1$ is the centroid of the C2–C7 ring.

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: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *PRPKAPPA* (Ferguson, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JJ2008).

References

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Ferguson, G. (1999). *PRPKAPPA*. University of Guelph, Canada.
He, Y.-H. & Wen, Y.-H. (2006). *Acta Cryst.* E**62**, o1312–o1313.
Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Tukada, H. & Mochizuki, K. (2003). *J. Mol. Struct.* **655**, 473–478.
Zhao, G. L., Feng, Y. L., Hu, X. C. & Kong, L. C. (2003). *Chin. J. Appl. Chem.* **20**, 806–808.

supplementary materials

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Adamantane-1-ammonium benzoate

W.-N. Zheng and B. Wang

Comment

Owing to its highly symmetrical and stable structure, adamantane and its derivatives have generated much interest in the past and continue to be actively studied as evidenced by the large number of compounds containing amantadine that have been synthesized (Tukada & Mochizuki, 2003; Zhao *et al.*, 2003; He & Wen, 2006). Here we report the synthesis and crystal structure of the title compound,(I), $C_{10}H_{15}NH_3^+ . C_7H_5O_2^-$, a salt obtained from the reaction of adamantane-1-ammonium hydrochloride and sodium benzoate (Fig. 1).

The adamantane-1-ammonium cation contains four 6-membered rings in a cage-like structure each in a slightly distorted boat conformation and with a protonated *N* atom at the 1-position. Puckering parameters (Cremer & Pople, 1975) Q, θ and φ are for rings 1–4 [(1) 0.630 (2) Å, 1.48 (18)°,272 (54)°; (2) 0.6247 (19)Å,178.36 (17)°, 251 (423)°; (3) 0.621 (4)Å,0.67 (18)°, 240 (54)°; (4)0.6207 (19)Å, 0.55 (18)°, 218 (39)°] where (1) = C7–C9/C13–C15, (2) = C7/C8/C10/C11/C16/C15, (3) = C8/C9/C13/C12/C11/C10 and (4) = C11–C16. C—C distances range from 1.518 (2)Å to 1.531 (3)Å and C—C—C angles range from 108.93 (13)° to 109.91 (13)°, while the exocyclic C—N bond length is 1.4924 (19)Å. These values are similar to that observed in adamantane-1-ammonium 2-nitrobenzoate (C—C = 1.5254 (18)Å to 1.532 (2)Å, C—C—C 109.06 (13)° to 109.84 (11)°, C—N = 1.4967 (18)Å (He & Wen, 2006). Both the negativly charged and neutral oxygen atoms in the benzoate anion are involved in strong N—H···O intermolecular hydrogen bond interactions with the ammonium cation group generating infinite one-dimensional chains along the *b* axis of the unit cell (Fig. 2, Table 1). In addition, weak π-ring C16–H16A···Cg1 interactions exist which contribute to crystal stability (Cg1 is the center of gravity of ring 1).

Experimental

A mixture of adamantane-1- ammonium hydrochloride (10 mmol), sodiumbenzoate (10 mmol) and methanol (50 ml) was stirred in a beaker. There were many solid powders produced and the solution was filtered. Colorless single crystals of the title compound suitable for X-ray analysis were obtained by slow evaporation of the solvents over a period of 20 h.

Refinement

Positional parameters of all the H atoms were calculated geometrically (aromatic C–H = 0.93 A°, aliphatic C–H = 0.97 A°) & N–H = 0.89Å) and were allowed to ride on the C,N atoms to which they are bonded, with U_{iso}(H) = 1.2-1.5U_{eq}(C,N).

Figures

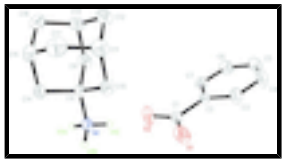

Fig. 1. The molecular structure of the title compound, with the atomic numbering scheme and displacement ellipsoids drawn at the 30% probability level. All H atoms except those on the N atom have been omitted for clarity.

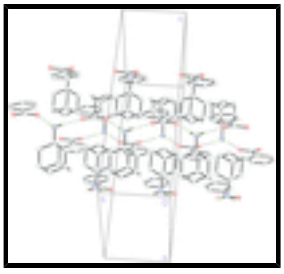

Fig. 2. A view of the crystal packing of the title compound. Dashed lines indicate N–H···O hydrogen bonds which form infinite, one-dimensional chains along the *b* axis of the unit cell. H atoms not involved in hydrogen bonding have been omitted for clarity.

adamantane-1-ammonium benzoate

Crystal data

$C_{10}H_{18}N^+·C_7H_5O_2^-$	F_{000} = 592
M_r = 273.36	D_x = 1.214 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 12490 reflections
a = 10.918 (2) Å	θ = 3.2–27.7º
b = 6.5664 (13) Å	μ = 0.08 mm^{-1}
c = 21.197 (4) Å	T = 298 K
β = 100.07 (3)º	Prism, colourless
V = 1496.3 (5) Å^3	0.20 × 0.20 × 0.20 mm
Z = 4	

Data collection

Rigaku SCXmini diffractometer	3437 independent reflections
Radiation source: fine-focus sealed tube	2453 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.043
Detector resolution: 13.6612 pixels mm^{-1}	θ_{max} = 27.5º
T = 298 K	θ_{min} = 3.2º
CCD_Profile_fitting scans	h = −14→14
Absorption correction: multi-scan (CrystalClear; Rigaku, 2005)	k = −8→8
T_{min} = 0.774, T_{max} = 1.000	l = −27→26
15027 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.055	H-atom parameters constrained
$wR(F^2)$ = 0.147	$w = 1/[\sigma^2(F_o^2) + (0.0654P)^2 + 0.3147P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max} < 0.001$

3437 reflections	$\Delta\rho_{max}$ = 0.20 e Å^{-3}
181 parameters	$\Delta\rho_{min}$ = −0.26 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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}
C1	0.56217 (14)	0.6892 (3)	0.15342 (7)	0.0405 (4)
C2	0.47020 (13)	0.7614 (2)	0.09647 (7)	0.0365 (3)
C3	0.41146 (17)	0.6184 (3)	0.05392 (8)	0.0551 (5)
H3A	0.4282	0.4807	0.0612	0.066*
C4	0.3278 (2)	0.6789 (3)	0.00041 (9)	0.0718 (6)
H4A	0.2899	0.5820	−0.0286	0.086*
C5	0.30094 (19)	0.8799 (4)	−0.00983 (9)	0.0707 (6)
H5A	0.2439	0.9201	−0.0455	0.085*
C6	0.35764 (18)	1.0225 (3)	0.03223 (9)	0.0651 (5)
H6A	0.3385	1.1596	0.0253	0.078*
C7	0.44324 (15)	0.9646 (3)	0.08504 (8)	0.0481 (4)
H7A	0.4828	1.0629	0.1130	0.058*
C8	0.93359 (16)	0.8861 (2)	0.16653 (8)	0.0463 (4)
H8A	0.9568	1.0003	0.1952	0.056*
H8B	0.8483	0.9057	0.1452	0.056*
C9	0.94432 (13)	0.6891 (2)	0.20453 (7)	0.0347 (3)
C10	0.90627 (16)	0.5096 (2)	0.15964 (8)	0.0472 (4)
H10A	0.9123	0.3838	0.1840	0.057*
H10B	0.8207	0.5260	0.1383	0.057*
C11	0.99218 (19)	0.5007 (3)	0.11014 (9)	0.0591 (5)
H11A	0.9681	0.3859	0.0811	0.071*
C12	0.98200 (19)	0.6977 (3)	0.07187 (9)	0.0633 (5)
H12A	0.8972	0.7158	0.0496	0.076*
H12B	1.0360	0.6915	0.0401	0.076*
C13	1.01933 (18)	0.8766 (3)	0.11689 (9)	0.0548 (5)
H13A	1.0126	1.0035	0.0922	0.066*
C14	1.07795 (14)	0.6598 (3)	0.23908 (8)	0.0474 (4)
H14A	1.0846	0.5347	0.2638	0.057*

H14B	1.1021	0.7723	0.2683	0.057*
C15	1.16369 (16)	0.6503 (3)	0.18941 (9)	0.0596 (5)
H15A	1.2497	0.6317	0.2113	0.071*
C16	1.15367 (17)	0.8477 (3)	0.15120 (10)	0.0628 (5)
H16A	1.2089	0.8428	0.1200	0.075*
H16B	1.1783	0.9616	0.1797	0.075*
C17	1.1262 (2)	0.4714 (3)	0.14452 (11)	0.0689 (6)
H17A	1.1330	0.3455	0.1688	0.083*
H17B	1.1813	0.4629	0.1134	0.083*
N1	0.85951 (11)	0.69816 (19)	0.25257 (6)	0.0393 (3)
H1A	0.8810	0.8025	0.2790	0.059*
H1B	0.8654	0.5828	0.2749	0.059*
H1C	0.7815	0.7146	0.2324	0.059*
O1	0.57302 (14)	0.5030 (2)	0.16200 (6)	0.0744 (5)
O2	0.62592 (11)	0.81896 (19)	0.18784 (6)	0.0594 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0380 (8)	0.0485 (9)	0.0338 (8)	0.0034 (7)	0.0030 (6)	−0.0005 (7)
C2	0.0332 (7)	0.0430 (8)	0.0325 (7)	−0.0025 (6)	0.0039 (6)	0.0033 (6)
C3	0.0573 (10)	0.0494 (10)	0.0514 (10)	−0.0022 (8)	−0.0102 (8)	−0.0026 (8)
C4	0.0713 (13)	0.0782 (15)	0.0541 (11)	−0.0070 (11)	−0.0218 (10)	−0.0098 (10)
C5	0.0622 (12)	0.0890 (16)	0.0518 (11)	0.0029 (11)	−0.0148 (9)	0.0201 (11)
C6	0.0635 (12)	0.0581 (12)	0.0670 (12)	0.0025 (9)	−0.0073 (10)	0.0234 (10)
C7	0.0473 (9)	0.0454 (9)	0.0482 (9)	−0.0030 (7)	−0.0012 (7)	0.0045 (7)
C8	0.0529 (9)	0.0362 (9)	0.0467 (9)	0.0043 (7)	−0.0002 (7)	0.0037 (7)
C9	0.0346 (7)	0.0325 (7)	0.0343 (7)	−0.0002 (6)	−0.0019 (6)	−0.0008 (6)
C10	0.0521 (9)	0.0385 (9)	0.0493 (9)	−0.0068 (7)	0.0046 (8)	−0.0090 (7)
C11	0.0723 (12)	0.0516 (11)	0.0546 (11)	−0.0037 (9)	0.0137 (9)	−0.0182 (8)
C12	0.0675 (12)	0.0823 (14)	0.0398 (9)	−0.0007 (10)	0.0083 (9)	−0.0031 (9)
C13	0.0648 (11)	0.0487 (10)	0.0507 (10)	−0.0016 (8)	0.0092 (9)	0.0150 (8)
C14	0.0391 (8)	0.0527 (10)	0.0461 (9)	0.0016 (7)	−0.0045 (7)	0.0046 (7)
C15	0.0375 (8)	0.0759 (13)	0.0635 (11)	0.0061 (8)	0.0036 (8)	0.0076 (10)
C16	0.0528 (10)	0.0718 (13)	0.0651 (12)	−0.0150 (9)	0.0134 (9)	0.0019 (10)
C17	0.0717 (13)	0.0601 (12)	0.0810 (14)	0.0191 (10)	0.0297 (11)	0.0009 (10)
N1	0.0371 (6)	0.0397 (7)	0.0375 (7)	0.0005 (5)	−0.0030 (5)	−0.0023 (5)
O1	0.0912 (11)	0.0536 (9)	0.0663 (9)	0.0002 (7)	−0.0200 (8)	0.0209 (6)
O2	0.0484 (7)	0.0625 (8)	0.0575 (7)	0.0142 (6)	−0.0177 (6)	−0.0197 (6)

Geometric parameters (Å, °)

C1—O1	1.238 (2)	C10—H10B	0.9700
C1—O2	1.2512 (19)	C11—C12	1.520 (3)
C1—C2	1.505 (2)	C11—C17	1.528 (3)
C2—C7	1.378 (2)	C11—H11A	0.9800
C2—C3	1.380 (2)	C12—C13	1.523 (3)
C3—C4	1.384 (3)	C12—H12A	0.9700
C3—H3A	0.9300	C12—H12B	0.9700

C4—C5	1.361 (3)	C13—C16	1.531 (3)
C4—H4A	0.9300	C13—H13A	0.9800
C5—C6	1.365 (3)	C14—C15	1.528 (2)
C5—H5A	0.9300	C14—H14A	0.9700
C6—C7	1.380 (2)	C14—H14B	0.9700
C6—H6A	0.9300	C15—C17	1.522 (3)
C7—H7A	0.9300	C15—C16	1.522 (3)
C8—C9	1.518 (2)	C15—H15A	0.9800
C8—C13	1.527 (2)	C16—H16A	0.9700
C8—H8A	0.9700	C16—H16B	0.9700
C8—H8B	0.9700	C17—H17A	0.9700
C9—N1	1.4925 (19)	C17—H17B	0.9700
C9—C14	1.526 (2)	N1—H1A	0.8900
C9—C10	1.526 (2)	N1—H1B	0.8900
C10—C11	1.526 (2)	N1—H1C	0.8900
C10—H10A	0.9700		
O1—C1—O2	123.88 (15)	C17—C11—H11A	109.4
O1—C1—C2	117.52 (14)	C11—C12—C13	109.58 (14)
O2—C1—C2	118.55 (15)	C11—C12—H12A	109.8
C7—C2—C3	118.90 (15)	C13—C12—H12A	109.8
C7—C2—C1	122.54 (14)	C11—C12—H12B	109.8
C3—C2—C1	118.55 (14)	C13—C12—H12B	109.8
C2—C3—C4	120.31 (18)	H12A—C12—H12B	108.2
C2—C3—H3A	119.8	C12—C13—C8	109.53 (15)
C4—C3—H3A	119.8	C12—C13—C16	109.35 (16)
C5—C4—C3	120.12 (18)	C8—C13—C16	109.25 (15)
C5—C4—H4A	119.9	C12—C13—H13A	109.6
C3—C4—H4A	119.9	C8—C13—H13A	109.6
C4—C5—C6	120.05 (17)	C16—C13—H13A	109.6
C4—C5—H5A	120.0	C9—C14—C15	108.92 (13)
C6—C5—H5A	120.0	C9—C14—H14A	109.9
C5—C6—C7	120.41 (18)	C15—C14—H14A	109.9
C5—C6—H6A	119.8	C9—C14—H14B	109.9
C7—C6—H6A	119.8	C15—C14—H14B	109.9
C2—C7—C6	120.18 (16)	H14A—C14—H14B	108.3
C2—C7—H7A	119.9	C17—C15—C16	109.76 (16)
C6—C7—H7A	119.9	C17—C15—C14	109.41 (15)
C9—C8—C13	109.32 (13)	C16—C15—C14	109.63 (15)
C9—C8—H8A	109.8	C17—C15—H15A	109.3
C13—C8—H8A	109.8	C16—C15—H15A	109.3
C9—C8—H8B	109.8	C14—C15—H15A	109.3
C13—C8—H8B	109.8	C15—C16—C13	109.38 (15)
H8A—C8—H8B	108.3	C15—C16—H16A	109.8
N1—C9—C8	109.18 (12)	C13—C16—H16A	109.8
N1—C9—C14	109.45 (12)	C15—C16—H16B	109.8
C8—C9—C14	109.91 (13)	C13—C16—H16B	109.8
N1—C9—C10	108.82 (12)	H16A—C16—H16B	108.2
C8—C9—C10	109.89 (12)	C15—C17—C11	109.36 (15)
C14—C9—C10	109.58 (13)	C15—C17—H17A	109.8

C11—C10—C9	108.97 (13)	C11—C17—H17A	109.8
C11—C10—H10A	109.9	C15—C17—H17B	109.8
C9—C10—H10A	109.9	C11—C17—H17B	109.8
C11—C10—H10B	109.9	H17A—C17—H17B	108.3
C9—C10—H10B	109.9	C9—N1—H1A	109.5
H10A—C10—H10B	108.3	C9—N1—H1B	109.5
C12—C11—C10	109.76 (15)	H1A—N1—H1B	109.5
C12—C11—C17	109.64 (17)	C9—N1—H1C	109.5
C10—C11—C17	109.22 (15)	H1A—N1—H1C	109.5
C12—C11—H11A	109.4	H1B—N1—H1C	109.5
C10—C11—H11A	109.4		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O1^{i}	0.89	1.83	2.7134 (17)	176
N1—H1B···O2^{ii}	0.89	1.90	2.7840 (18)	173
N1—H1C···O2	0.89	1.92	2.7915 (18)	166
C16—H16A···Cg1^{iii}	0.97	2.74	3.702 (2)	174

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

Fig. 1

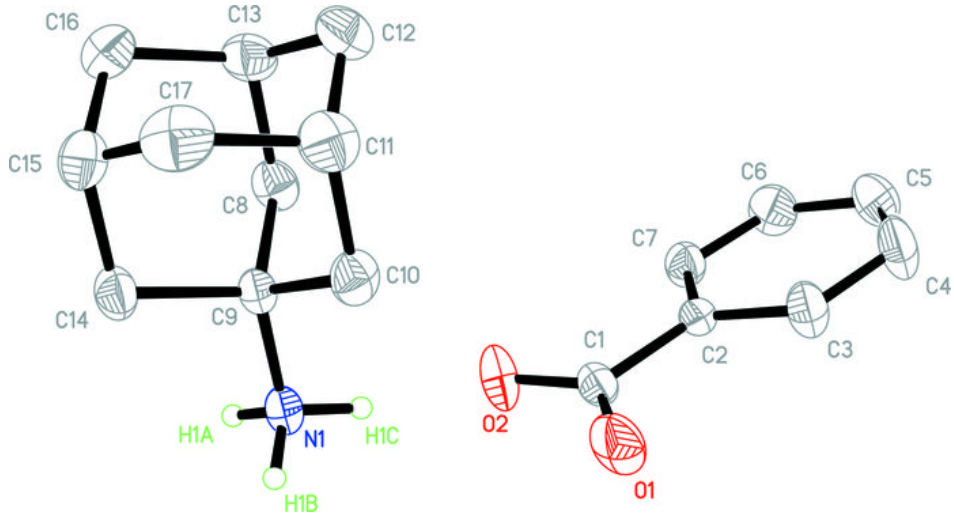
C16
C13
C12
C17
C15
C11
C8
C10
C14
C9
H1A
N1
H1C
H1B
C6
C5
C7
C4
C1
C2
C3
O2
O1

Fig. 2