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Adamantane-1-ammonium 2-nitrobenzoate

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.002 Å
R factor = 0.045
wR factor = 0.125
Data-to-parameter ratio = 17.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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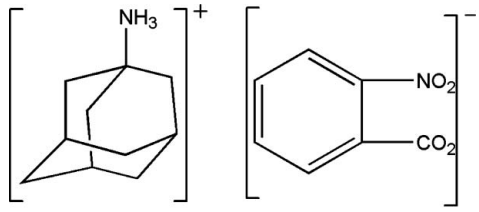
The title compound, $C_{17}H_{22}N_2O_4$, was obtained by the diffusion method. Strong N—H···O hydrogen bonds generate a chain-like structure.

Comment

Owing to its highly symmetrical and stable structure, adamantane and its derivatives are widely used in several fields, such as medicine and macromolecular materials. Amantadine has been used successfully for the prevention and treatment of the influenza A virus by blocking the M2 protein ion channel (Bright *et al.*, 2005). A large number of compounds containing amantadine have been synthesized (Tukada & Mochizuki, 2003; Zhao *et al.*, 2003). Here we report the synthesis and crystal structure of the title compound, (I), illustrated in Fig. 1.



(I)

In the cation, C—C distances range from 1.5254 (18) to 1.532 (2) Å and C—C—C angles range from 109.06 (13) to 109.84 (11)°, while the exocyclic C—N bond length is 1.4967 (18) Å.

There are N—H···O hydrogen-bonding interactions between the cations and anions. The protonated atom N2 forms three hydrogen bonds with carboxyl O atoms from adjacent nitrobenzoate anions (Table 1). As a result, the

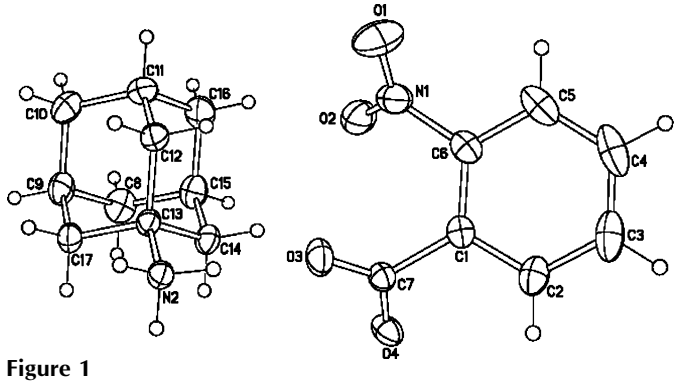


Figure 1
A view of the structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are shown at the 30% probability level.

hydrogen bonding generates a one-dimensional chain structure (Fig. 2).

Experimental

2-Nitrobenzoic acid (0.1671 g, 1 mmol) and 1-aminoadamantane (0.1512 g, 1 mmol) were mixed in 20 ml absolute ethanol with continuous stirring for 0.5 h, resulting in a clear colourless solution. Colourless single crystals of the title compound suitable for X-ray diffraction were obtained by slow evaporation of the solvent over a period of 7 d (yield 60%). Analysis calculated for $C_{17}H_{22}N_2O_4$: C 64.13, H 6.97, N 8.80%; found: C 64.02, H 6.77, N 8.98%.

Crystal data

$C_{10}H_{18}N^+\cdot C_7H_4NO_4^-$
M_r = 318.37
Monoclinic, $P2_1/c$
a = 13.155 (3) Å
b = 6.4940 (13) Å
c = 20.608 (4) Å
β = 105.63 (3)°
V = 1695.4 (7) Å^3
Z = 4
D_x = 1.247 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 11565 reflections
θ = 3.2–27.5°
μ = 0.09 mm^{-1}
T = 296 (2) K
Chunk, colourless
0.46 × 0.33 × 0.29 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.965, T_{max} = 0.975
15796 measured reflections
3878 independent reflections
2597 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 27.5°
$h = -17 \rightarrow 17$
$k = -8 \rightarrow 8$
$l = -26 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.125
S = 1.04
3878 reflections
217 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0655P)^2 + 0.0743P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2*C*···O3	0.922 (9)	1.913 (9)	2.8355 (16)	178.4 (15)
N2—H2*B*···O4^{i}	0.919 (9)	1.846 (9)	2.7638 (16)	176.9 (14)
N2—H2*D*···O4^{ii}	0.919 (9)	1.899 (9)	2.8015 (15)	166.7 (14)

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

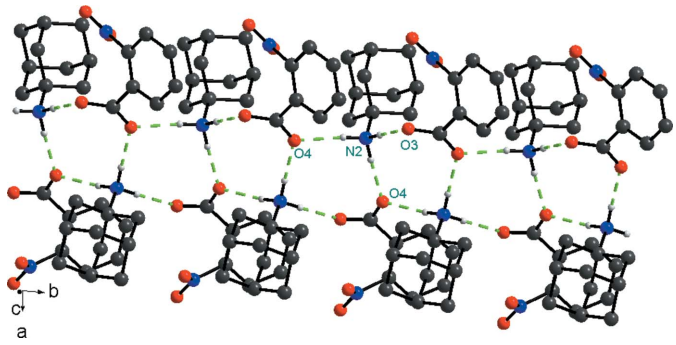


Figure 2
A packing diagram for (I). N—H···O interactions are depicted by dashed lines. H atoms not involved in hydrogen bonding have been omitted.

H atoms bonded to C atoms were positioned geometrically (aromatic C—H = 0.93 Å and aliphatic C—H = 0.97 Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C). H atoms bonded to N2 were located in difference Fourier maps and refined with an N—H distance restraint of 0.91(1) Å and with U_{iso}(H) = 1.2U_{eq}(N).

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXL97*.

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