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1,3-Propanediammonium bis(3′-nitro-*trans*-cinnamate) and *trans*-1,2-cyclohexanediammonium bis(3′-nitro-*trans*-cinnamate)

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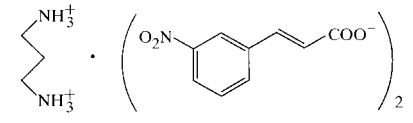
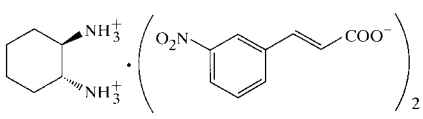
In the title two adducts, $C_3H_{12}N_2^{2+}\cdot 2C_9H_6NO_4^-$, (I), and $C_6H_{16}N_2^{2+}\cdot 2C_9H_6NO_4^-$, (II), hydrogen bonds between the diammonium and carboxylate ions form a two-dimensional network parallel to the *ab* plane in (I) and one-dimensional chains along the *c* axis in (II). The cyclohexanediammonium ion in (II) has a crystallographic twofold axis.

Comment

The crystal structure and photoreactivity of ethylenediammonium bis(3′-nitro-*trans*-cinnnamate) was reported by Ito *et al.* (1995). The structures of 1,3-propanediammonium bis(3′-nitro-*trans*-cinnamate) {IUPAC name: propane-1,3-diammonium bis[(*E*)-3-(3-nitrophenyl)propenoate]}, (I), and *trans*-1,2-cyclohexanediammonium bis(3′-nitro-*trans*-cinnamate) {IUPAC name: *trans*-cyclohexane-1,2-diammonium bis[(*E*)-3-(3-nitrophenyl)propenoate]}, (II), are reported here.



(I)



(II)

Experimental

Ether/methanol or ether/ethanol was used as the solvent for mixing and recrystallization.

Compound (I)

Crystal data

$C_3H_{12}N_2^{2+}\cdot 2C_9H_6NO_4^-$
M_r = 460.44
Orthorhombic, *Pbca*
a = 7.136 (3) Å
b = 23.710 (3) Å
c = 25.964 (4) Å
V = 4393.0 (18) Å^3
Z = 8
D_x = 1.392 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 12.3–14.8°
μ = 0.108 mm^{-1}
T = 298 (1) K
Prism, pale yellow
0.7 × 0.4 × 0.4 mm

Data collection

Rigaku AFC-7*R* diffractometer
ω scans
5308 measured reflections
5040 independent reflections
3317 reflections with $I > 2\sigma(I)$
R_{int} = 0.011
θ_{max} = 27.5°
$h = 0 \rightarrow 9$
$k = -11 \rightarrow 31$
$l = 0 \rightarrow 34$
3 standard reflections
every 150 reflections
intensity decay: 1.5%

Refinement

Refinement on F^2
$R(F)$ = 0.056
$wR(F^2)$ = 0.167
S = 1.03
5040 reflections
322 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0829P)^2 + 1.5750P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Table 1
Selected geometric parameters (Å, °) for (I).

O1—C13	1.245 (2)	O6—C22	1.259 (3)
O2—C13	1.250 (3)	O7—N10	1.213 (3)
O3—N9	1.205 (4)	O8—N10	1.219 (3)
O4—N9	1.219 (3)	N11—C31	1.484 (3)
O5—C22	1.243 (3)	N12—C33	1.483 (3)
O3—N9—O4	123.6 (2)	O1—C13—O2	125.8 (2)
O7—N10—O8	124.1 (2)	O5—C22—O6	124.9 (2)

Table 2
Hydrogen-bonding geometry (Å, °) for (I).

D—H···A	D—H	H···A	D···A	D—H···A
N11—H11A···O2	0.87 (3)	1.96 (3)	2.782 (2)	158 (2)
N11—H11B···O5^{i}	0.99 (3)	1.77 (3)	2.731 (3)	164 (2)
N11—H11C···O2^{ii}	0.84 (3)	2.11 (2)	2.830 (3)	144 (2)
N12—H12A···O6^{iii}	0.90 (2)	1.94 (2)	2.820 (3)	164 (2)
N12—H12B···O6^{iv}	0.83 (2)	1.99 (2)	2.816 (2)	174 (2)
N12—H12C···O1^{v}	0.98 (2)	1.80 (2)	2.756 (2)	163 (2)

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

Compound (II)

Crystal data

$C_6H_{16}N_2^{2+}\cdot 2C_9H_6NO_4^-$
$M_r = 500.51$
Orthorhombic, *Pbcn*
$a = 26.919\ (4)$ Å
$b = 7.572\ (3)$ Å
$c = 11.966\ (3)$ Å
$V = 2439.1\ (10)$ Å^3
$Z = 4$
$D_x = 1.363$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 12.2$–$14.6°$
$\mu = 0.103$ mm^{-1}
$T = 298\ (1)$ K
Prism, colourless
$0.7 \times 0.3 \times 0.3$ mm

Data collection

Rigaku AFC-7*R* diffractometer
ω scans
2942 measured reflections
2798 independent reflections
1507 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 27.5°$
$h = 0 \rightarrow 35$
$k = -10 \rightarrow 3$
$l = 0 \rightarrow 16$
3 standard reflections
every 150 reflections
intensity decay: 0.7%

Refinement

Refinement on F^2
$R(F) = 0.074$
$wR(F^2) = 0.190$
$S = 1.77$
2798 reflections
183 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.05P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.44$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

Table 3
Selected geometric parameters (Å, °) for (II).

O1—C7	1.233 (4)	O4—N5	1.207 (4)
O2—C7	1.248 (3)	N6—C16	1.470 (4)
O3—N5	1.207 (6)		
O3—N5—O4	124.1 (3)	O1—C7—O2	125.3 (2)

Table 4
Hydrogen-bonding geometry (Å, °) for (II).

D—H···A	D—H	H···A	D···A	D—H···A
N6—H6A···O1^{i}	0.95 (3)	1.80 (3)	2.723 (3)	166 (3)
N6—H6B···O2	0.90 (3)	1.89 (3)	2.786 (3)	170 (2)
N6—H6C···O2^{ii}	0.74 (3)	2.09 (3)	2.771 (3)	153 (3)

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

In (II), there is an orientational disorder of the C═C double bond of the 3′-nitro-cinnamate ion. The occupancy factors were 0.768 (9) and 0.232 (9) for C8/C9 and C8*/C9*, respectively. The H atoms bonded to N11 and N12 of (I), and N6 of (II) were located from difference syntheses and were refined isotropically. The N—H bond distances are 0.83 (2)–0.99 (3) Å in (I) and 0.74 (3)–0.95 (3) Å in (II). The other H-atom positional parameters were calculated geometrically and fixed with $U_{iso}(H) = 1.2U_{eq}$(parent atom).

For both compounds, data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1999); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *TEXSAN*.

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