

Ethane-1,2-diammonium bis(4-nitrobenzoate)
dihydrateDaniel E. Lynch^{a*} and Peter C. Healy^b^aSchool of Science and the Environment, Coventry University, Coventry CV1 5FB, England, and ^bSchool of Science and Technology, Griffith University, Nathan Q4111, AustraliaCorrespondence e-mail:
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Key indicators

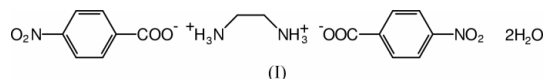
Single-crystal X-ray study
T = 298 K
Mean $\sigma(C-C)$ = 0.004 Å
R factor = 0.044
wR factor = 0.150
Data-to-parameter ratio = 11.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.The structure of the title compound, $C_2H_{10}N_2^{2+} \cdot 2C_7H_4NO_4^{-} \cdot 2H_2O$, comprises interdigitated two-dimensional hydrogen-bonded networks derived from a repeat unit that contains the bisammonium, two benzoates and two water molecules.

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Comment

A search of the Cambridge Structural Database (Allen, 2002) reveals that there are 39 reported organic salt structures containing ethane-1,2-diamine and a carboxylate group. Of these, 11 contain a metal and can be considered as coordination complexes. The remainder can be separated into the 19 that contain an alkyl carboxylate whereas the rest (nine) contain substituted benzoates. Interestingly, the majority (five) of these are nitrobenzoates, namely 4-nitroanthranilate (Smith, Wermuth & White, 2002), 3,5-dinitrobenzoate (Nethaji *et al.*, 1992; Lynch *et al.*, 1994; Burchell *et al.*, 2001) and 3,5-dinitrosalicylate (Smith, Wermuth, Bott *et al.*, 2002). Here we report the 1:2 organic salt dihydrate of ethane-1,2-diammonium with 4-nitrobenzoate, (I) (Fig. 1).The solid-state packing of (I) involves two symmetry-independent two-dimensional hydrogen-bonded networks individually derived from the repeat unit represented in Fig. 2 by molecules *A*–*C*, *O1_w* and *O2_w*. Both networks, comprising molecules *A*–*C*, *O1_w* and *O2_w*, and *D*–*F*, *O3_w* and *O4_w*, respectively, are essentially identical, the symmetry between the groupings being broken by the interdigitation of the 4-NBA molecules. Crosslinking through the associations of the water molecules to adjacent carboxylate O atoms creates the second dimension in the network. Hydrogen-bonding interactions are listed in Table 1.

Experimental

1:2 molar amounts of ethane-1,2-diamine and 4-nitrobenzoic acid were refluxed in ethanol for 20 min. Total evaporation of the solvent gave colourless needles (m.p. 465 K).

Crystal data

 $C_2H_{10}N_2^{2+} \cdot 2C_7H_4NO_4^{-} \cdot 2H_2O$ $M_r = 430.38$ Triclinic, *P1* $a = 12.213$ (2) Å $b = 22.317$ (2) Å $c = 7.091$ (5) Å $\alpha = 89.47$ (2)° $\beta = 89.17$ (2)° $\gamma = 84.72$ (8)° $V = 1924.2$ (14) Å³ $Z = 4$ $D_x = 1.486$ Mg m⁻³Mo $K\alpha$ radiation

Cell parameters from 25 reflections

 $\theta = 13.0$ – 19.2 ° $\mu = 0.13$ mm⁻¹ $T = 298$ (2) K

Needle, colourless

 $0.40 \times 0.20 \times 0.10$ mm

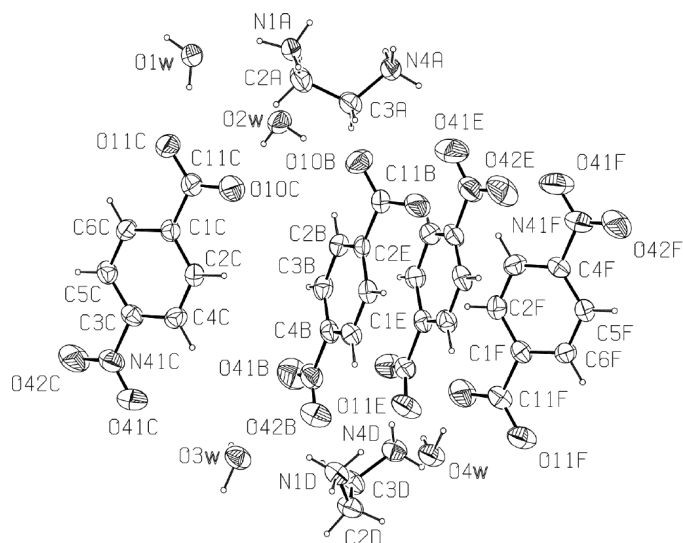


Figure 1
The asymmetric unit of the title compound, showing the atom-numbering scheme and 50% probability displacement ellipsoids.

Data collection

Rigaku AFC-7R diffractometer
 ω -2 θ scans
 Absorption correction: none
 6810 measured reflections
 6810 independent reflections
 3360 reflections with $I > 2\sigma(I)$
 $\theta_{\max} = 25.4^\circ$

$h = 0 \rightarrow 14$
 $k = -26 \rightarrow 26$
 $l = -8 \rightarrow 8$
 3 standard reflections
 every 150 reflections
 intensity decay: 0.4%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.150$
 $S = 1.00$
 6810 reflections
 577 parameters

H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0767P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.21 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.30 \text{ e } \text{\AA}^{-3}$

Table 1

Hydrogen-bonding geometry (\AA , $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1A—H11A \cdots O2W	0.89	2.03	2.833 (4)	149
N1A—H12A \cdots O1W	0.89	1.99	2.786 (3)	148
N1A—H13A \cdots O11B ⁱ	0.89	1.85	2.744 (3)	178
N4A—H41A \cdots O1W ⁱⁱ	0.89	2.09	2.881 (3)	147
N4A—H42A \cdots O2W ⁱⁱ	0.89	2.09	2.861 (3)	145
N4A—H43A \cdots O10B ⁱⁱ	0.89	1.96	2.847 (3)	175
N1D—H11D \cdots O4W ⁱⁱⁱ	0.89	2.06	2.846 (3)	147
N1D—H12D \cdots O3W	0.89	1.97	2.781 (3)	150
N1D—H13D \cdots O11E	0.89	1.85	2.728 (3)	171
N4D—H41D \cdots O10E	0.89	1.87	2.753 (3)	173
N4D—H42D \cdots O3W ⁱⁱ	0.89	2.10	2.881 (4)	146
N4D—H43D \cdots O4W ^{iv}	0.89	2.04	2.839 (4)	149
O1W—H11 \cdots O11C ^v	0.87 (4)	1.86 (4)	2.693 (3)	160 (3)
O1W—H12 \cdots O11C	0.91 (3)	1.77 (4)	2.658 (3)	166 (3)
O2W—H21 \cdots O10C	0.94 (4)	1.72 (4)	2.653 (3)	171 (4)
O2W—H22 \cdots O10B	0.85 (3)	1.87 (4)	2.704 (3)	168 (3)
O3W—H31 \cdots O11F ^{vi}	0.80 (3)	1.99 (3)	2.785 (3)	169 (3)
O3W—H32 \cdots O11F ⁱⁱⁱ	0.97 (3)	1.70 (3)	2.663 (3)	174 (3)
O4W—H41 \cdots O11E	0.87 (4)	1.88 (4)	2.756 (3)	176 (3)
O4W—H42 \cdots O10F	1.02 (4)	1.65 (4)	2.674 (3)	177 (3)

Symmetry codes: (i) $1-x, 1-y, 1-z$; (ii) $x, y, z-1$; (iii) $1-x, -y, 1-z$; (iv) $1-x, -y, -z$; (v) $-x, 1-y, 2-z$; (vi) $x-1, y, z$.

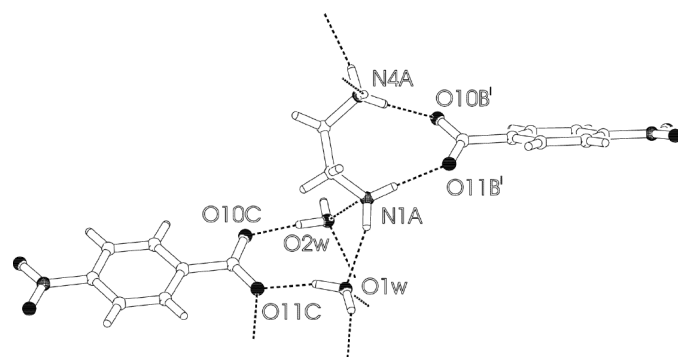


Figure 2
One instance of the hydrogen-bonded unit that extends to form the two-dimensional networks of (I). [Symmetry code: (i) $1-x, 1-y, 1-z$.]

All H atoms on the amines were initially located in difference syntheses but were then included in the refinement (along with all other H atoms) at calculated positions as riding models, with N—H set to 0.89 \AA and C—H set to 0.97 (CH₂) and 0.93 \AA (Ar—H), while the isotropic displacement parameters were set equal to 1.25 U_{eq} of the carrier atom. All water H atoms were located in difference syntheses and both positional and isotropic displacement parameters were refined.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1985); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLUTON94* (Spek, 1994) and *PLATON97* (Spek, 1997); software used to prepare material for publication: *SHELXL97*.

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References

- Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.
 Burchell, C. J., Glidewell, C., Lough, A. J. & Ferguson, G. (2001). *Acta Cryst.* **B57**, 201–212.
 Lynch, D. E., Smith, G., Byriell, K. A. & Kennard, C. H. L. (1994). *Acta Cryst.* **C50**, 1259–1262.
 Molecular Structure Corporation (1985). *TEXSAN*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
 Molecular Structure Corporation (1993). *MSC/AFC Diffractometer Control Software*. Version 5.1.10. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
 Nethaji, M., Pattabhi, V., Chhabra, N., Poonia, N. S. (1992). *Acta Cryst.* **C48**, 2207–2209.
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
 Smith, G., Wermuth, U. D., Bott, R. C., Healy, P. C. & White, J. M. (2002). *Aust. J. Chem.* **55**, 349–356.
 Smith, G., Wermuth, U. D. & White, J. M. (2002). *Acta Cryst.* **E58**, o1088–o1090.
 Spek, A. L. (1994). *PLUTON94*. University of Utrecht, The Netherlands.
 Spek, A. L. (1997). *PLATON97*. University of Utrecht, The Netherlands.