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[2-Hydroxy-1,1-bis(hydroxymethyl)ethyl]-bis(2-hydroxyethyl)ammonium 4-nitrobenzoate monohydrate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.050
wR factor = 0.141
Data-to-parameter ratio = 12.6

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

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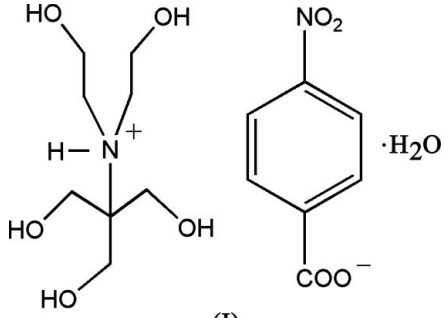
The title salt, $C_8H_{20}NO_5^+\cdot C_7H_4NO_4^-\cdot H_2O$, is composed of a 4-nitrobenzoate anion, a [2-hydroxy-1,1-bis(hydroxymethyl)-ethyl]bis(2-hydroxyethyl)ammonium (NBHHP) cation and a water molecule. The three units are linked by O—H···O hydrogen bonds.

Comment

4-Nitrobenzoic acid can easily lose its acidic H atom and form complexes with other compounds through hydrogen bonding. Complexes of 4-nitrobenzoic acid (forming dimers through hydrogen bonding; Tonogaki *et al.*, 1993), pyridinium 4-nitrobenzoate 4-nitrobenzoic acid (Carrow & Wheeler, 1998), tris(2-benzimidazylmethyl) ammonium 4-nitrobenzoate (Ji *et al.*, 2000), 4-nitrobenzoic acid–3-amino-1,2,4-triazole (Byriel *et al.*, 1992) and 4-nitrobenzoic acid–4-nitropyridine *N*-oxide (Moreno-Fuquen *et al.*, 2000) have already been reported. In these complexes, the hydrogen bonds play an important role in both building and stabilizing the structure. We have synthesized another such complex, (I), and report its structure here (Fig. 1).



(I)

Complex (I) is composed of a 4-nitrobenzoate anion, a [2-hydroxy-1,1-bis(hydroxymethyl)ethyl]bis(2-hydroxyethyl)-ammonium (NBHHP) cation and a water molecule. The three units are linked by O—H···O hydrogen bonds. As expected, the O1···O6 and O2···O7 distances are short and similar, since both acceptors are from the carboxylate group, and the O5···O10 distance is longer (Table 2). In addition to the O—H···O hydrogen bonds, there are some weaker intramolecular N—H···O hydrogen bonds between N1 and three of the hydroxyl groups (Table 2).

O—H···O and N—H···O hydrogen bonds play an important role in building the structure, while a number of C—H···O hydrogen bonds (Fig. 2) play a subordinate role in stabilizing the structure. In the unit cell, neighbouring benzene

rings of the 4-nitrobenzoate are parallel to one another. The relevant centroid–centroid separation between two adjacent benzene rings related by the symmetry operation ($-x$, $-y+1$, $-z+1$) is 3.687 (4) Å.

Experimental

The title compound was synthesized from a mixture of 4-nitrobenzoic acid (1 mmol, 0.17 g) and BHHP (1 mmol, 0.21 g). The mixture was dissolved in a mixed solvent of anhydrous ethanol (10 ml) and water (10 ml), then heated to 273 K and stirred for half an hour. The reaction system was cooled to room temperature and colourless crystals were collected after 5 d.

Crystal data

$C_8H_{20}NO_5^+\cdot C_7H_4NO_4^-\cdot H_2O$
M_r = 394.38
Triclinic, $P\overline{1}$
a = 6.5419 (6) Å
b = 11.5935 (10) Å
c = 13.4632 (12) Å
α = 67.290 (2)°
β = 77.302 (2)°
γ = 82.612 (2)°
V = 917.81 (14) Å^3
Z = 2
D_x = 1.427 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2504 reflections
θ = 3.0–24.6°
μ = 0.12 mm^{-1}
T = 298 (2) K
Block, colourless
0.37 × 0.29 × 0.26 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*, Bruker, 2000)
T_{min} = 0.953, T_{max} = 0.965
4825 measured reflections
3242 independent reflections
2739 reflections with $I > 2\sigma(I)$
R_{int} = 0.011
θ_{max} = 25.0°
$h = -7 \rightarrow 7$
$k = -12 \rightarrow 13$
$l = -15 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.142
S = 1.04
3242 reflections
258 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0803P)^2 + 0.3186P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.409 (2)	N1—C4	1.539 (2)
O4—C6	1.416 (2)	C1—C4	1.526 (3)
O6—C9	1.238 (3)	C5—C6	1.504 (3)
O7—C9	1.256 (3)	C9—C10	1.512 (3)
N1—C5	1.522 (2)	C10—C11	1.379 (3)
C7—N1—C5	109.85 (14)	C1—C4—N1	112.42 (14)
C7—N1—C4	112.90 (14)	C6—C5—N1	113.00 (15)
C5—N1—C4	115.69 (13)	O4—C6—C5	109.44 (16)
O1—C1—C4	113.91 (16)	O6—C9—O7	125.51 (19)
O2—C2—C4	108.77 (15)	O6—C9—C10	117.01 (18)
O3—C3—C4	110.54 (15)	O7—C9—C10	117.48 (17)
O1—C1—C4—N1	66.8 (2)	O7—C9—C10—C11	163.1 (2)
N1—C5—C6—O4	−60.5 (2)	O6—C9—C10—C15	162.4 (2)
O6—C9—C10—C11	−16.3 (3)	O7—C9—C10—C15	−18.2 (3)

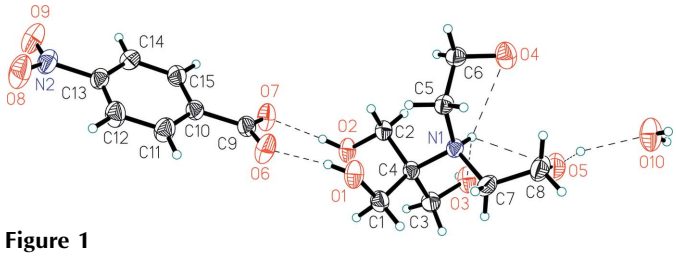


Figure 1
The asymmetric unit of (I) with atom labels, showing 40% probability displacement ellipsoids. The dashed lines denote hydrogen bonds.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O6	0.82	1.81	2.608 (3)	166
O2—H2···O7	0.82	1.78	2.595 (3)	176
O3—H3···O2^{i}	0.82	1.87	2.684 (2)	175
O4—H4···O1^{ii}	0.82	1.94	2.742 (2)	164
O5—H5···O10	0.82	1.98	2.774 (6)	162
O10—H10A···O7^{iii}	0.84 (2)	1.98 (2)	2.811 (6)	175
N1—H1N···O3	0.86 (2)	2.23 (2)	2.690 (2)	113
N1—H1N···O4	0.86 (2)	2.51 (2)	2.917 (2)	110
N1—H1N···O5	0.86 (2)	2.47 (2)	2.895 (4)	112
C3—H3B···O2	0.97	2.46	2.873 (3)	105
C5—H5A···O1	0.97	2.38	3.077 (3)	129
C5—H5A···O9^{iv}	0.97	2.58	3.428 (6)	146
C7—H7B···O5^{v}	0.97	2.55	3.276 (3)	131
C11—H11···O6	0.93	2.49	2.781 (4)	98
C12—H12···O8	0.93	2.45	2.726 (3)	97
C14—H14···O9	0.93	2.45	2.720 (2)	97
C15—H15···O7	0.93	2.55	2.831 (6)	98

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

The H atoms attached to N1 and O10 were refined with the distance restraints 0.86 and 0.82 Å, respectively. All other H atoms were placed in calculated positions and allowed to ride on their parent atoms at distances of 0.82 (O—H), 0.97 (methylene C—H) and 0.93 Å (aromatic C—H). For all H atoms, U_{iso} = 1.2–1.5U_{eq}(C,N,O).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve

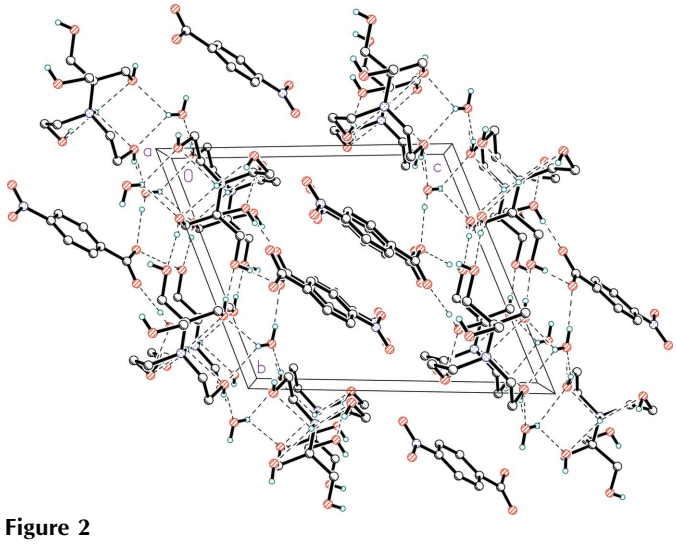


Figure 2
The packing of (I), viewed along the c axis. Dashed lines indicate hydrogen bonds.

structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXL97*.

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