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3,4-Dihydroxybenzylammonium 3,5-dinitrobenzoate monohydrate

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In the title compound, $C_7H_{10}NO_2^+\cdot C_7H_3N_2O_6^-\cdot H_2O$, the aromatic rings of the cation and anion are stacked alternately along the *a* axis. The ammonium group of the cation, the carboxylate moiety of the anion and a water molecule of crystallization are connected *via* N—H···O and O—H···O hydrogen bonds, forming hydrophilic columns along the *c* axis.

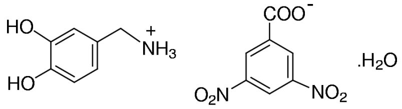
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.033
wR factor = 0.090
Data-to-parameter ratio = 7.5

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

Comment

Crystal structures of some charge-transfer complexes involving dopamine and its analogs as donors have been reported by us (Ohba & Ito, 2002*a*,*b*,*c*). The structure of the title compound, (I), has been determined to study the packing mode of the molecules and the hydrogen-bonding pattern.



(I)

The organic molecules show π–π stacking (Fig. 1), as expected from the yellow color of the crystal. The dihedral angle between benzene rings C14–C19 and C21–C26 is 5.50 (8)°, and these rings are arranged alternately along the *a* axis (Fig. 2), with center-to-center distances of 3.616 and

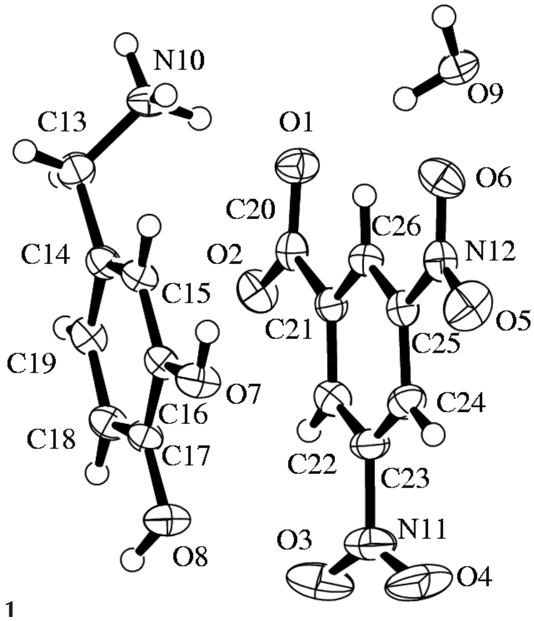


Figure 1
The molecular structure of (I), showing displacement ellipsoids at the 50% probability level.

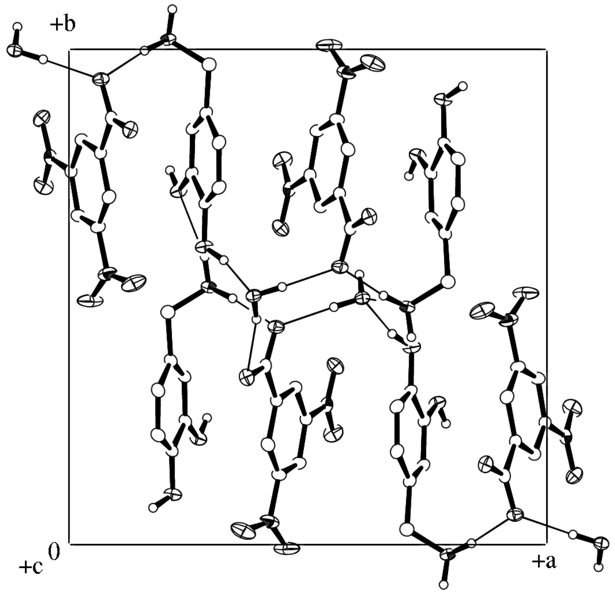


Figure 2
The crystal structure of (I), projected along the *c* axis. Thin lines indicate hydrogen bonds. H atoms bonded to C atoms have been omitted for clarity.

3.748 Å. The ammonium group of the cation, the carboxylate group of the anion and the water molecule of crystallization are connected *via* hydrogen bonds (Table 2), forming hydrophilic columns along the *c* axis. The two hydroxy groups of the benzylammonium ion (O7 and O8) act not only as hydrogen-bond donors, but also as hydrogen-bond acceptors for one of the ammonium H atoms (H10*C*).

Experimental

A hot solution containing 3,4-dihydroxybenzylamine hydrobromide (36.4 mg, 0.162 mmol) in MeOH (10 ml) was mixed with a hot solution containing 3,5-dinitrobenzoic acid (36.3 mg, 0.167 mmol) in MeOH (10 ml) and then with a 1 *N* NaOH aqueous solution (0.15 ml). The mixture was filtered and the filtrate was left overnight at room temperature to obtain yellow crystals of (I) [yield 20 mg, 31%; m.p. 462–463 K (decomposition)].

Crystal data

$C_7H_{10}NO_2^+ \cdot C_7H_3N_2O_6^- \cdot H_2O$
$M_r = 369.29$
Orthorhombic, $Pna2_1$
$a = 14.546$ (3) Å
$b = 15.065$ (4) Å
$c = 6.930$ (2) Å
$V = 1518.6$ (7) Å^3
$Z = 4$
$D_x = 1.615$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 10.9$–$13.6°$
$\mu = 0.14$ mm^{-1}
$T = 298$ K
Plate, yellow
0.5 × 0.4 × 0.2 mm

Data collection

Rigaku AFC-7*R* diffractometer
ω–2θ scans
Absorption correction: by integration (*ABSCOR*; Higashi, 1999)
$T_{\min} = 0.950$, $T_{\max} = 0.974$
32296 measured reflections
1883 independent reflections
1654 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.017$
$\theta_{\max} = 27.5°$
$h = -7 \rightarrow 18$
$k = 0 \rightarrow 19$
$l = -3 \rightarrow 9$
3 standard reflections every 150 reflections intensity decay: 0.2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.090$
$S = 1.06$
1883 reflections
251 parameters
H atom treated by a mixture of constrained and independent refinement

$w = 1/[\sigma^2(F_o^2) + (0.0564P)^2 + 0.1249P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.18$ e Å^{-3}
$\Delta\rho_{\min} = -0.27$ e Å^{-3}

Table 1
Selected torsion angles (°).

O1—C20—C21—C26	3.1 (3)	O5—N12—C25—C24	−4.5 (3)
O3—N11—C23—C22	−16.7 (4)	N10—C13—C14—C15	52.3 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
O7—H7···O2$^{\rm i}$	0.80 (3)	1.96 (3)	2.733 (2)	161 (3)
O8—H8···O9$^{\rm ii}$	0.89 (4)	1.89 (4)	2.770 (3)	167 (4)
O9—H9*A*···O1	0.93 (4)	1.90 (4)	2.825 (3)	176 (4)
O9—H9*B*···O2$^{\rm iii}$	0.88 (3)	2.07 (3)	2.930 (3)	166 (3)
N10—H10*A*···O1	0.95	1.87	2.816 (3)	173
N10—H10*B*···O9$^{\rm iii}$	0.95	2.00	2.901 (3)	158
N10—H10*C*···O7$^{\rm iv}$	0.95	2.22	3.155 (2)	167
N10—H10*C*···O8$^{\rm iv}$	0.95	2.41	2.880 (3)	110

Symmetry codes: (i) $x, y, z-1$; (ii) $x-\frac{1}{2}, \frac{1}{2}-y, z$; (iii) $2-x, -y, z-\frac{1}{2}$; (iv) $\frac{3}{2}-x, y-\frac{1}{2}, \frac{1}{2}+z$.

Friedel-pair reflections were merged before the final refinement, since anomalous scattering effects were negligible. The hydroxyl and water H atoms were located in difference syntheses and refined isotropically. The ammonium H atoms were also located from difference syntheses, but their positional parameters were recalculated geometrically and refined as riding, with N—H = 0.95 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm N})$. The other H atoms were positioned geometrically and refined as riding, with C—H = 0.95 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

Data collection: *WinAFC Diffractometer Control Software* (Rigaku, 1999); cell refinement: *WinAFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 2001); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *TEXSAN*.

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