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The 1:1 proton-transfer monohydrate salt of 3,5-dinitrosalicylic acid with 1-amino-5-hydroxy-naphthalene

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Key indicators

Single-crystal X-ray study
T = 297 K
Mean σ(C–C) = 0.008 Å
R factor = 0.066
wR factor = 0.228
Data-to-parameter ratio = 11.5

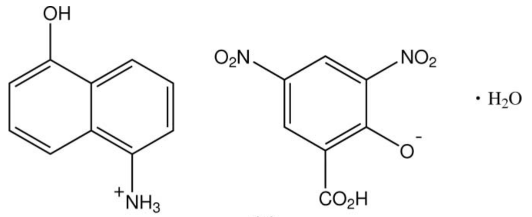
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title complex, 5-hydroxynaphthalen-1-aminium 2-carboxy-4,6-dinitrophenolate monohydrate, $C_{10}H_{10}NO^+\cdot C_7H_3N_2O_7^-\cdot H_2O$, shows a three-dimensional hydrogen-bonded framework structure in which columns comprising π-stacked cations and anions are interconnected by conventional hydrogen bonds.

Comment

Among the proton-transfer compounds of 3,5-dinitrosalicylic acid (DNSA) with aromatic Lewis bases we have observed that, while conventional hydrogen bonding is of primary importance in the molecular assembly in the solid state (Smith *et al.*, 2003), weak aromatic C—H···O interactions become increasingly important for polycyclic compounds (Smith *et al.*, 2007). Cation–anion π–π interactions are not particularly significant within the overall series, being restricted to polycyclic analogues such as quinoline, quinaldic acid and 1,10-phenanthroline (Smith *et al.*, 2007), and benzidine (Smith *et al.*, 2006).

The reaction of DNSA with the moderately weak base 1-amino-5-hydroxynaphthalene (5-amino-1-naphthol = NAPH) (pK_a 3.96, *cf.* 2.2 for DNSA) might be expected to result in proton transfer, and this was found to be the case with the isolation of the title compound, (I). In (I) (Fig. 1), the alternating NAPH cations and DNSA anions give partial aromatic ring overlap [ring centroid separation and inter-ring dihedral angle (α) for rings C1–C6 (DNSA) and C5*A*–C10*A* (NAPH) are 3.779 (4) Å, 1.90 (1)° (intra) and 3.526 (4) Å, 1.90 (1)° (inter), respectively]. These form columns which extend down the *c*-axial direction in the cell (Fig. 2) and are linked by a number of hydrogen-bonded interactions, including a three-centre $R_1^2(4)$ N^+—H···$(O,O')_{\text{nitro group}}$ association (Table 1), giving a three-dimensional framework structure.



(I)

The DNSA anion is essentially planar [torsion angles C2—C1—C7—O71 = −173.0 (6)°; C2—C3—N3—O32 = −176.4 (6)°; C4—C5—N5—O52 = 178.6 (6)°], while an usual intramolecular O(carboxyl)···O(phenol) hydrogen bond [2.520 (7) Å] is also present.

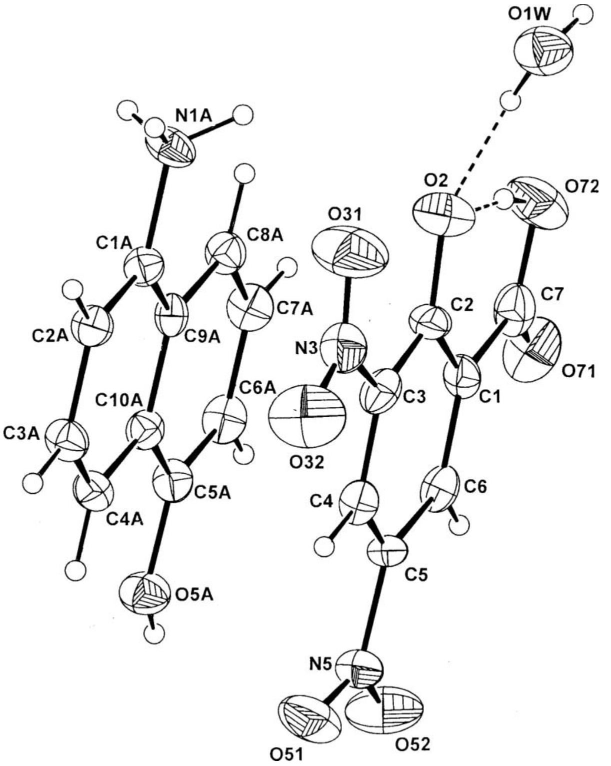


Figure 1
The asymmetric unit of (I) with hydrogen bonds shown as dashed lines. Displacement ellipsoids are drawn at the 30% probability level.

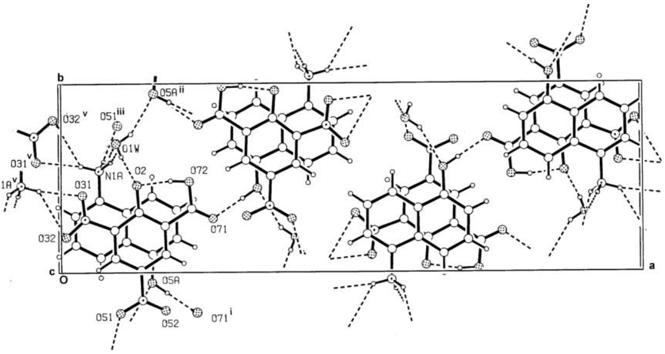


Figure 2
Perspective view of the packing in the unit cell viewed approximately down the *c*-axis direction, showing cation–anion stacks with associated hydrogen-bonding interactions (dashed lines). For symmetry codes, see Table 1.

Experimental

The title compound was synthesized by heating 1 mmol quantities of DNSA and NAPH in 80% ethanol/water (50 ml) for 10 min under reflux. After concentration to *ca* 30 ml, partial room-temperature evaporation of the hot-filtered solution gave large dark-brown crystals (m.p. 467.9–469.5 K). Characteristic IR absorption frequencies (cm^{-1}): ν(OH), 3090, 3527; ν(C—O) 1684; NO_2(asym), 1570, 1521; NO_2(sym), 1375, 1318; ν(C—OH), 1166.

Crystal data

$C_{10}H_{10}NO^+\cdot C_7H_3N_2O_7^-\cdot H_2O$
$M_r = 405.32$
Monoclinic, $P2_1/n$
$a = 27.324$ (6) Å
$b = 8.879$ (4) Å
$c = 7.037$ (4) Å
$\beta = 94.98$ (3)°
$V = 1700.8$ (13) $Å^3$
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.13\ \mathrm{mm}^{-1}$
$T = 297$ (2) K
$0.50 \times 0.34 \times 0.15$ mm

Data collection

Rigaku AFC-7R diffractometer
Absorption correction: ψ scan (*TEXSAN for Windows*; Molecular Structure Corporation, 1999)
$T_{\min} = 0.938$, $T_{\max} = 0.981$
3545 measured reflections
3006 independent reflections
2096 reflections with $I > 2\sigma(I)$
$R_{\mathrm{int}} = 0.025$
3 standard reflections
frequency: 150 min
intensity decay: 2.4%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.066$
$wR(F^2) = 0.228$
$S = 1.14$
3006 reflections
262 parameters
H-atom parameters constrained
$\Delta\rho_{\max} = 0.38$ e $Å^{-3}$
$\Delta\rho_{\min} = -0.26$ e $Å^{-3}$

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

D—H···A	D—H	H···A	D···A	D—H···A
O72—H72···O2	0.91	1.62	2.520 (7)	170
O5A—H5A···O71$^{\mathrm{i}}$	0.92	1.79	2.708 (6)	180
O1W—H11W···O5A^{ii}	0.81	2.21	2.951 (6)	152
O1W—H12W···O2	0.91	2.07	2.975 (7)	174
N1A—H11A···O51$^{\mathrm{iii}}$	0.92	2.03	2.834 (7)	145
N1A—H12A···O1W^{iv}	0.88	1.87	2.754 (7)	173
N1A—H13A···O31$^{\mathrm{v}}$	0.93	2.0800	2.997 (7)	166
N1A—H13A···O32$^{\mathrm{v}}$	0.93	2.56	3.289 (8)	135

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

H atoms involved in hydrogen-bonding interactions were located by difference methods but their positional and isotropic displacement parameters were fixed. Other H atoms were included in the refinement in calculated positions (C—H = 0.95 Å) using a riding-model approximation, with $U_{\mathrm{iso}}(\mathrm{H}) = 1.2U_{\mathrm{eq}}(\mathrm{C})$.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1999); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN for Windows* (Molecular Structure Corporation, 1999); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

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