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

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

The title complex, 5-hydroxynaphthalen-1-aminium 2-carboxy-4,6-dinitrophenolate monohydrate, $\text{C}_{10}\text{H}_{10}\text{NO}^+\text{C}_{7}\text{H}_3\text{N}_2\text{O}_7^-\text{H}_2\text{O}$, shows a three-dimensional hydrogen-bonded framework structure in which columns comprising $\pi$-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 $\text{C}--\text{H} \cdot \cdot \cdot \text{O}$ interactions become increasingly important for polycyclic compounds (Smith et al., 2007). Cation–anion $\pi--\pi$ 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) ($\text{pK}_a$ 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 (\(\alpha\)) for rings C1–C6 (DNSA) and C5A–C10A (NAPH) are 3.779 (4) Å, 1.90 (1)\(^\circ\) (intra) and 3.526 (4) Å, 1.90 (1)\(^\circ\) (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 $R_2^2(4)$ $\text{N}^+--\text{H} \cdot \cdot \cdot (\text{O, O})$ nitro group association (Table 1), giving a three-dimensional framework structure.

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

C_{10}H_{10}NO^{+}\cdot C_{7}H_{3}N_{2}O_{7}^{-}\cdot H_{2}O

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) Å³

\(Z = 4\)

Mo Kα radiation

\(\mu = 0.13\) mm⁻¹

\(T = 297\) (2) K

0.50 × 0.34 × 0.15 mm

Data collection

Rigaku AFC-7R diffractometer

Absorption correction: ψ scan

(TEXSAN for Windows; Molecular Structure Corporation, 1999)

3006 independent reflections

2096 reflections with \(I > 2\sigma(I)\)

3 standard reflections

frequency: 150 min

intensity decay: 2.4%

Refinement

\(R[F^2 > 2\sigma(F^2)] = 0.066\)

\(wR(F^2) = 0.228\)

\(S = 1.14\)

3545 measured reflections

262 parameters

H-atom parameters constrained

\(\Delta\rho_{\text{max}} = 0.38\) e Å⁻³

\(\Delta\rho_{\text{min}} = -0.26\) e Å⁻³

Table 1

Hydrogen-bond geometry (Å⁻¹).

<table>
<thead>
<tr>
<th>D—H—A</th>
<th>D—H</th>
<th>H—A</th>
<th>D—A</th>
<th>D—H—A</th>
</tr>
</thead>
<tbody>
<tr>
<td>O72—H72···O2</td>
<td>0.91</td>
<td>1.62</td>
<td>2.520 (7)</td>
<td>170</td>
</tr>
<tr>
<td>O54—H54···O71i</td>
<td>0.92</td>
<td>1.79</td>
<td>2.708 (6)</td>
<td>180</td>
</tr>
<tr>
<td>O1W—H11W···O5Aiv</td>
<td>0.81</td>
<td>2.21</td>
<td>2.951 (6)</td>
<td>152</td>
</tr>
<tr>
<td>O1W—H12W···O2</td>
<td>0.91</td>
<td>2.07</td>
<td>2.975 (7)</td>
<td>174</td>
</tr>
<tr>
<td>N1A—H11A···O3i</td>
<td>0.92</td>
<td>2.03</td>
<td>2.834 (7)</td>
<td>145</td>
</tr>
<tr>
<td>N1A—H12A···O1Wii</td>
<td>0.88</td>
<td>1.87</td>
<td>2.754 (7)</td>
<td>173</td>
</tr>
<tr>
<td>N1A—H13A···O3ii</td>
<td>0.93</td>
<td>2.0800</td>
<td>2.997 (7)</td>
<td>166</td>
</tr>
<tr>
<td>N1A—H13A···O3ii</td>
<td>0.93</td>
<td>2.56</td>
<td>3.289 (8)</td>
<td>135</td>
</tr>
</tbody>
</table>

Symmetry codes: (i) \(-x + \frac{1}{2}, y - \frac{1}{2}, -z + 1\); (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 \(U_{eq}(H) = 1.2U_{eq}(C)\).

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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References

