

1*H*-Nicotinamidium 3,5-dinitrosalicylate

Marian Koman,^{a*} Ladislav Martiška,^a Dušan Valigura^a and Tadeuš Glowiak^b

^aDepartment of Inorganic Chemistry, Slovak Technical University, Radlinského 9, 812 37 Bratislava, Slovakia, and ^bInstitut of Chemistry, University of Wrocław, F. Joliot-Curie 14, 50 383 Wrocław, Poland

Correspondence e-mail: koman@chtf.stuba.sk

Key indicators

Single-crystal X-ray study

T = 293 K

Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$

Disorder in main residue

R factor = 0.040

w*R* factor = 0.110

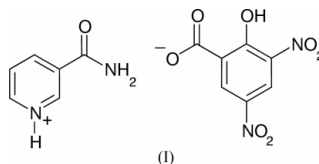
Data-to-parameter ratio = 9.6

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

The crystal and molecular structure of the title compound, $\text{C}_6\text{H}_7\text{N}_2\text{O}^+ \cdot \text{C}_7\text{H}_3\text{N}_2\text{O}_7^-$, has been studied by single-crystal X-ray diffraction at 293 (2) K. The structure consists of pairs of 1*H*-nicotinamidium cations and 3,5-dinitrosalicylate anions which are held together by ionic interactions and by additional hydrogen-bond interactions. The ions are also held together by weaker hydrogen bonds and by van der Waals interactions.

Comment

As part of our study of new copper(II) complexes with 3,5-dinitrosalicylate anions and nicotinamide, the title compound, (I), was also prepared. Later, the same compound were also prepared by reaction of nicotinamide with 3,5-dinitrosalicylic acid in aqueous solution. We believe that salt formation is typical for 3,5-dinitrosalicylic acid, *e.g.* with 3-amino-1,2,4-triazole (Smith *et al.*, 1996) or even with different amino-benzoic acids (Smith *et al.*, 1995).



Neighbouring protonated molecules of nicotinamide and 3,5-dinitrosalicylate anions are linked by hydrogen bonds $\text{N4}-\text{H41} \cdots \text{O1}^{\text{iv}}$ (see Table 2). Additional hydrogen bonds are formed by the amide H atoms of the nicotinamide cation and by the O atom of the nitro groups of the salicylate anion (Table 2). Atom H3 forms an intramolecular hydrogen bond with atoms O2 and O3. Atom O3 is statistically distributed between two positions.

Experimental

Crystals of the title compound suitable for X-ray crystal structure analysis were obtained by slow crystallization of the yellow–green reaction mixture containing copper(II) sulfate (0.0926 g $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, 0.37 mmol), 3,5-dinitrosalicylic acid (0.2811 g, 0.74 mmol) and nicotinamide (0.186 g, 1.52 mmol) in aqueous solution (50 ml), in an Erlenmeyer flask at room temperature for three months.

Crystal data

$\text{C}_6\text{H}_7\text{N}_2\text{O}^+ \cdot \text{C}_7\text{H}_3\text{N}_2\text{O}_7^-$

$M_r = 350.25$

Monoclinic, $P2_1/n$

$a = 4.740 (1) \text{ \AA}$

$b = 21.968 (4) \text{ \AA}$

$c = 13.858 (3) \text{ \AA}$

$\beta = 94.62 (3)^\circ$

$V = 1438.3 (5) \text{ \AA}^3$

$Z = 4$

$D_x = 1.617 \text{ Mg m}^{-3}$

Cu $K\alpha$ radiation

Cell parameters from 25

reflections

$\theta = 4.5\text{--}11.8^\circ$

$\mu = 1.20 \text{ mm}^{-1}$

$T = 293 (2) \text{ K}$

Plate, yellow

$0.40 \times 0.20 \times 0.15 \text{ mm}$

Data collection

Syntex P2₁ diffractometer
 θ -2 θ scans
 Absorption correction: ψ scan
 (North *et al.*, 1968)
 $T_{\min} = 0.544$, $T_{\max} = 0.741$
 3511 measured reflections
 2212 independent reflections
 1631 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.031$
 $\theta_{\text{max}} = 65.1^\circ$
 $h = -5 \rightarrow 2$
 $k = 0 \rightarrow 25$
 $l = -16 \rightarrow 16$
 2 standard reflections
 every 100 reflections
 intensity decay: 15%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.110$
 $S = 1.07$
 2212 reflections
 231 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0618P)^2 + 0.1656P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.15 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\text{min}} = -0.18 \text{ e } \text{Å}^{-3}$

Table 1

Selected geometric parameters (Å, °).

O1—C7	1.254 (2)	C1—C6	1.383 (3)
O2—C7	1.258 (3)	C1—C2	1.410 (3)
O3—C2	1.297 (3)	C1—C7	1.497 (3)
O4—N1	1.205 (3)	C2—C3	1.402 (3)
O5—N1	1.196 (3)	C3—C4	1.379 (3)
O6—N2	1.220 (3)	C4—C5	1.372 (3)
O7—N2	1.211 (3)	C5—C6	1.390 (3)
O8—C13	1.228 (2)	C8—C9	1.379 (3)
N1—C3	1.453 (3)	C8—C12	1.382 (3)
N2—C5	1.462 (3)	C8—C13	1.501 (2)
N3—C13	1.318 (3)	C10—C11	1.364 (3)
N4—C9	1.337 (3)	C11—C12	1.377 (3)
N4—C10	1.332 (3)		
O5—N1—O4	122.0 (2)	C4—C5—C6	121.96 (19)
O5—N1—C3	119.1 (2)	C4—C5—N2	117.87 (18)
O4—N1—C3	118.8 (2)	C6—C5—N2	120.16 (18)
O7—N2—O6	123.0 (2)	O1—C7—O2	123.63 (18)
O7—N2—C5	119.1 (2)	O1—C7—C1	118.15 (19)
O6—N2—C5	117.9 (2)	O2—C7—C1	118.22 (17)
C9—N4—C10	122.82 (17)	C9—C8—C12	118.35 (17)
C6—C1—C2	121.02 (18)	C9—C8—C13	121.89 (17)
C6—C1—C7	120.29 (17)	C12—C8—C13	119.73 (17)
C2—C1—C7	118.68 (18)	N4—C9—C8	119.81 (18)
O3—C2—C3	122.82 (18)	N4—C10—C11	119.19 (18)
O3—C2—C1	119.74 (19)	C10—C11—C12	119.95 (19)
C3—C2—C1	117.40 (18)	C8—C12—C11	119.87 (19)
C4—C3—C2	122.08 (17)	O8—C13—N3	124.10 (18)
C4—C3—N1	117.56 (19)	O8—C13—C8	119.74 (18)
C2—C3—N1	120.35 (19)	N3—C13—C8	116.15 (17)
C5—C4—C3	118.61 (18)		

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N3—H31 ⁱ ...O4 ⁱ	0.86	2.17	3.018 (2)	167
N3—H32 ⁱⁱ ...O8 ⁱⁱ	0.86	2.32	2.995 (2)	136
N3—H32 ⁱⁱⁱ ...O6 ⁱⁱⁱ	0.86	2.35	3.038 (3)	138
N4—H41 ^{iv} ...O1 ^{iv}	0.86	1.76	2.619 (2)	176

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

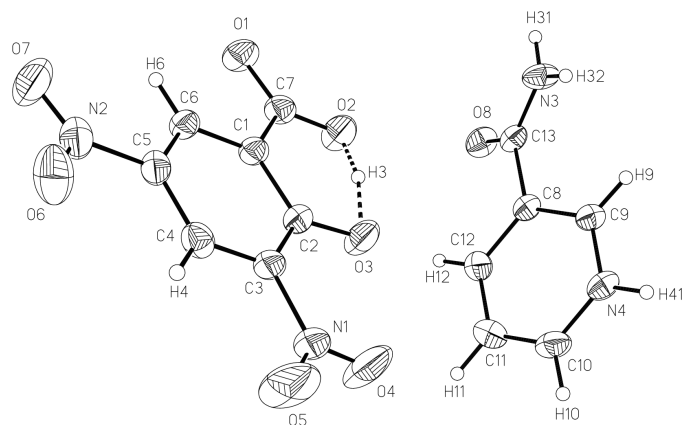


Figure 1

The anion and cation in the crystal structure of the title compound. Displacement ellipsoids are drawn at the 35% probability level and H atoms are shown as spheres of arbitrary radii. The alternative disordered atoms O3' and H3' have been omitted for clarity.

The O atom of the hydroxy group is disordered, occupying two alternative positions represented by O3 and O3' with occupancy factors of 0.758 (5) and 0.242 (5), respectively. Atom O3 was refined anisotropically and O3' isotropically. For atom O3 (anisotropic refinement) in a single position, $R1 = 0.071$ for 1631 reflections and $\Delta\rho_{\text{max}} = 1.58 \text{ e } \text{Å}^{-3}$. For the alternative atom O3 (isotropically refined), $R1$ and $\Delta\rho_{\text{max}}$ were 0.044 and $0.43 \text{ e } \text{Å}^{-3}$, respectively. The H atoms were placed in calculated positions, except for H3, which was located from a difference Fourier map.

Data collection: P2₁ Software (Syntex, 1974); cell refinement: P2₁ Software; data reduction: XP21 (Pavelčík, 1993); program(s) used to solve structure: SHELXS86 (Sheldrick, 1985); program(s) used to refine structure: SHELXL93 (Sheldrick, 1994); molecular graphics: ORTEP (Johnson, 1965); software used to prepare material for publication: SHELXL93.

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