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A cation–anion complex: 1,10-phenanthrolin-1-ium 5-sulfonatosalicylic acid

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.005 Å
R factor = 0.043
wR factor = 0.106
Data-to-parameter ratio = 11.2

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

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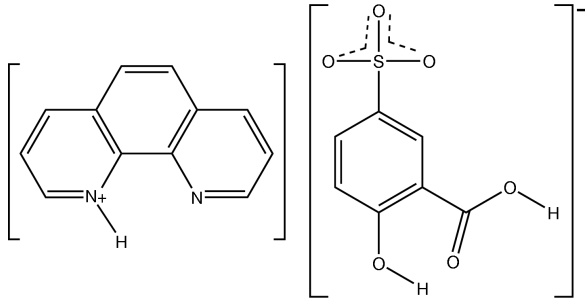
The title compound, $C_{12}H_9N_2^+\cdot C_7H_5O_6S^-$, consists of a 1,10-phenanthrolin-1-ium cation and a 5-sulfosalicylate anion. Hydrogen bonds link the anions into a one-dimensional chain. Furthermore, hydrogen bonds between the NH groups of 1,10-phenanthrolin-1-ium cations and the sulfonate O atoms attach the cations to the one-dimensional chain.

Comment

Recently, some protonated 1,10-phenanthrolin-1-ium complexes have been reported and structurally characterized (Wang *et al.*, 1999; Hensen *et al.*, 1998; Hensen *et al.*, 2000; Bonfim *et al.*, 2003). We report here a cation–anion 1,10-phenathroline complex, (phenH)(H_2ssal) (phenH and H_2ssal are the 1,10-phenanthrolin-1-ium cation and the 5-sulfosalicylate anion, respectively), (I).



(I)

In the anion (Fig. 1 and Table 1), the carboxyl group is nearly coplanar with the benzene ring [the dihedral angle is 3.2 (2) Å] and there is an intramolecular hydrogen bond involving the hydroxy group and carboxyl atom O2. Moreover, intermolecular hydrogen bonds between a sulfonate O atom and a carboxylate O atom connect the anions into a one-dimensional chain (Fig. 2 and Table 2). In most reported cases, every sulfonate O atom is coordinated by two or three hydrogen bonds (Chertanova & Pascard, 1996), while in the title complex two sulfonate O atoms (O4 and O5) accept only one hydrogen bond. Furthermore, in the title complex the cation is essentially planar and the protonated N atom forms a hydrogen bond with the sulfonate O atom (Fig. 3). The hydrogen-bonded N···O distance in (I), 2.780 (4) Å, is significantly shorter than the mean N···O distance (2.946 Å) for hydrogen bonds of N-donors to sulfonate groups (Pirard *et al.*, 1995; Haynes *et al.*, 2004).

Experimental

An aqueous solution (20 ml) of 5-sulfosalicylic acid dihydrate (0.127 g, 0.50 mmol) and $La(NO_3)\cdot 6H_2O$ (0.199 g, 0.46 mmol) was

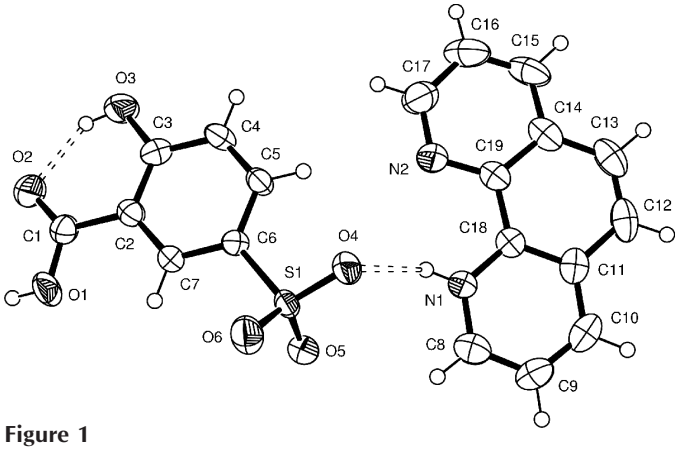


Figure 1
ORTEP-3 diagram of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines.

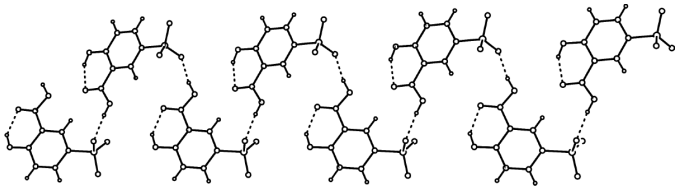


Figure 2
View of the one-dimensional hydrogen bonded chain of 5-sulfosalicylate anions. Hydrogen bonds are shown as dashed lines.

mixed with a previously prepared solution of 1,10-phenanthroline (0.098 g, 0.50 mmol) in methanol–water (10 ml, 1:9 *v*/*v*). The resulting solution was stirred and refluxed for 5 h and then cooled to room temperature. After slow evaporation for two days, colorless block-shaped crystals of (I) were obtained.

Crystal data

$C_{12}H_9N_2^+\cdot C_7H_5O_6S^-$
M_r = 398.38
Monoclinic, $P2_1$
a = 7.0492 (9) Å
b = 10.6716 (14) Å
c = 11.7464 (15) Å
β = 103.426 (2)°
V = 859.49 (19) Å^3
Z = 2
D_x = 1.539 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5405
θ = 0.9–28.3°
μ = 0.23 mm^{-1}
T = 295 (2) K
Block, colorless
0.34 × 0.31 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.926, T_{max} = 0.964
4555 measured reflections
2938 independent reflections
2910 reflections with $I > 2\sigma(I)$
R_{int} = 0.014
θ_{max} = 25.2°
$h = -5 \rightarrow 8$
$k = -12 \rightarrow 12$
$l = -14 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.106
S = 1.18
2938 reflections
262 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0565P)^2 + 0.1823P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}
Absolute structure: Flack (1983), 1312 Friedel pairs
Flack parameter = 0.04 (9)

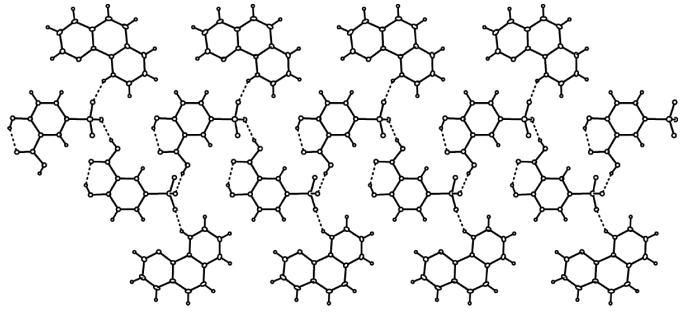


Figure 3
View of the cations hydrogen-bonded to the anion chain in (I). Hydrogen bonds are shown as dashed lines.

Table 1
Selected bond lengths (Å).

S1—O6	1.439 (2)	O1—C1	1.306 (4)
S1—O4	1.454 (2)	O2—C1	1.216 (4)
S1—O5	1.465 (2)	O3—C3	1.344 (4)
S1—C6	1.777 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O5^{i}	0.85 (4)	1.79 (4)	2.623 (3)	168 (5)
O3—H3···O2	0.85 (3)	1.86 (3)	2.587 (4)	142 (5)
N1—H2···O4	0.82 (4)	2.10 (3)	2.780 (4)	141 (4)

Symmetry code: (i) $2-x, y-\frac{1}{2}, 1-z$.

H atoms bonded to C atoms were positioned geometrically and treated as riding, with C—H distances of 0.93 Å and U_{iso}(H) = 1.2U_{eq}(parent). H atoms bound to O and N atoms were located in a difference Fourier map and refined with restraints for O—H and N—H distances [0.85 (1) and 0.82 (1) Å], with fixed isotropic displacement parameters [U_{iso}(H) = 0.08 Å^2].

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT* and *SHELXTL* (Bruker, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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