Crystal structure of 1,1′-dihydro[4,4′-bipyridine]-1,1′-diium-bis(2-sulfonatoterephthalic acid) — water (1:4), $(C_{10}H_{10}N_2)(C_8H_5O_7S)_2 \cdot 4H_2O$

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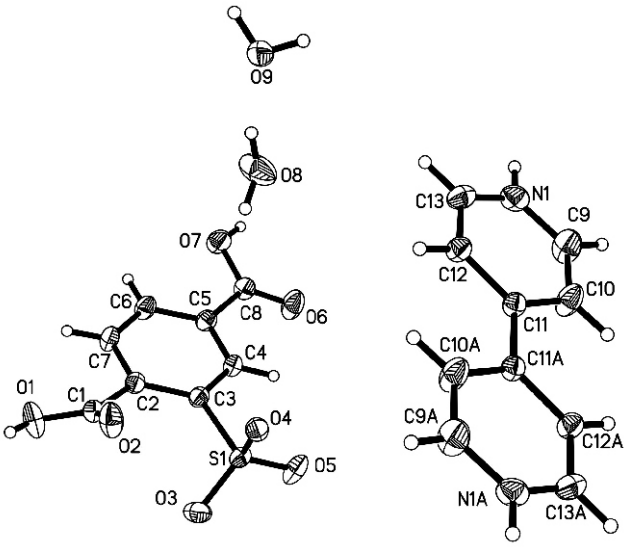

Abstract

$C_{26}H_{28}N_2O_{18}S_2$, triclinic, $P\bar{1}$ (no. 2),
a = 8.4687(5) Å, b = 9.4670(5) Å, c = 10.4823(6) Å,
α = 73.007(1)°, β = 71.567(1)°, γ = 89.582(1)°, V = 759.1 Å^3,
Z = 1, $R_{gt}(F)$ = 0.035, $wR_{ref}(F^2)$ = 0.110, T = 298 K.

Source of material

2-sulfoterephthalic acid (0.246 g, 1.0 mmol) was dissolved in hot ethanol (5 mL, 95%), and a solution of 4,4′-bipyridine (0.156 g, 1.0 mmol) in the same solvent (5 mL) was added. After one hour of reflux the solution was filtered. After two weeks colorless crystals had formed, which were suitable for X-ray diffraction experiments.

Expermental details

All H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of d(C sp^2—H) = 0.93 Å with U_{iso} = 1.2 U_{eq}(parent atom), d(N—H) = 0.86 Å with U_{iso} = 1.2 U_{eq}(parent atom) and d(O—H) = 0.82 Å with U_{iso} = 1.5 U_{eq}(parent atom).

Discussion

The design and synthesis of supramolecular compounds, especially those constructed by hydrogen bonding interactions have been more and more attractive due to their special physical properties and potential application. A well known and effective design strategy is the matching of suitable hydrogen bonding donors and acceptors [1,2]. 2-sulfoterephthalic acid may act as an excellent, readily available hydrogen bond donor. The 2-sulfoterephthalic acid possesses several interesting characteristics: (a) it has two carboxyl groups and one sulfonate group which may be completely or partially deprotonated, inducing rich coordination modes and allowing interesting structures with higher dimensions; (b) it can act not only as hydrogen-bond acceptor but also as hydrogen-bond donor, depending upon the number of deprotonated groups [3-7].

In the 2-sulfoterephthalic acid ligand, the sulfonic group is deprotonated, two carboxyl groups are protonated. The distances d(C—O) of the two carboxyl groups are 1.202(3) Å, 1.310(3) Å and 1.199(4) Å, 1.310(3) Å, which is shorter than a typical d(C—O) = 1.439(2) Å, but longer than a typical d(C=O) = 1.173(5) Å [8,9]. d(S1—O3), d(S1—O4) and d(S1—O5) are 1.446(2) Å, 1.453(2) Å and 1.440(2) Å, respectively. d(N1—C9) = 1.377(4) Å and d(N1—C13) = 1.384(4) Å are shorter than typical d(N—C) = 1.443(4) Å, but longer than typical d(N=C) = 1.269(2) Å. In order to balance the anion charge of the 2-sulfonatoterephthalatic acid, the N atoms of the 4,4′-bipyridine ligand are protonated.

The adjacent water molecules and 2-sulfonatoterephthalatic acid ligands are linked by intermolecular O—H···O bond interactions (O9—H9B···O4^{i}, O9—H9A···O5^{ii}, O9—H9A···O3^{ii}, O8—H8B···O9, O8—H8A···O2^{iii}, O8—H8A···O3^{iii}, O7—H7···O9^{iv}, O1—H1···O8^{v}, symmetry codes: (i) $-x+1,-y+1,-z+1$; (ii) $x-1,y+1,z$; (iii) $-x-2,-y+1,-z+1$; (iv) $x,y-1,z+1$; (v) $x+1,y,z$). The adjacent 4,4′-bipridinediium and 2-sulfonatoterephthalatic acid ligands are linked by N1—H1A···O2 (symmetry code: (vi) $x-1,y,z$) hydrogen-bond interactions. All above hydrogen bond interactions may enchance the stability of the solid-state structure of the title compound and generate a three-dimemsional architecture.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.15 × 0.21 × 0.26 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.64 cm^{-1}
Diffractometer, scan mode:	Bruker APEX II, φ/ω
$2\theta_{max}$:	50.2°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8997, 2686
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2506
$N(param)_{refined}$:	220
Programs:	SHELXS-97, SHELXL-97 [10]

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Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	2*i*	1.3344	0.5161	0.7060	0.092
H(7)	2*i*	0.4633	0.0052	1.3277	0.082
H(8A)	2*i*	0.5717	0.6643	0.5169	0.115
H(8B)	2*i*	0.4278	0.7333	0.5560	0.115
H(9A)	2*i*	0.2705	0.9521	0.5437	0.069
H(9B)	2*i*	0.2266	0.8660	0.4787	0.069
H(1A)	2*i*	0.1187	0.4029	0.4115	0.053
H(4)	2*i*	0.7754	–0.0009	0.9106	0.043
H(6)	2*i*	0.7992	0.2327	1.1683	0.051
H(7A)	2*i*	1.0269	0.3774	0.9937	0.050
H(9)	2*i*	0.1402	0.2373	0.2982	0.069
H(10)	2*i*	0.3394	0.2850	0.0795	0.064
H(12)	2*i*	0.4739	0.6750	0.1099	0.043
H(13)	2*i*	0.2741	0.6169	0.3294	0.051

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1)	2*i*	1.02057(6)	0.12620(5)	0.65556(5)	0.0403(3)	0.0398(3)	0.0363(3)	0.0046(2)	–0.0100(2)	–0.0194(2)
O(1)	2*i*	1.2722(2)	0.4481(2)	0.7702(2)	0.057(1)	0.061(1)	0.061(1)	–0.0227(8)	–0.0297(8)	0.0009(8)
O(2)	2*i*	1.1559(2)	0.4496(2)	0.6079(2)	0.066(1)	0.068(1)	0.0377(8)	–0.0265(8)	–0.0132(7)	–0.0063(7)
O(3)	2*i*	1.2015(2)	0.1433(2)	0.6043(2)	0.0398(8)	0.078(1)	0.0594(9)	0.0167(7)	–0.0117(7)	–0.0363(8)
O(4)	2*i*	0.9487(2)	0.2313(2)	0.5621(1)	0.0385(7)	0.0493(8)	0.0323(7)	–0.0004(6)	–0.0088(5)	–0.0146(6)
O(5)	2*i*	0.9510(2)	–0.0235(2)	0.6900(2)	0.088(1)	0.0414(8)	0.0523(9)	–0.0011(7)	–0.0208(8)	–0.0247(7)
O(6)	2*i*	0.5592(2)	–0.1061(2)	1.1565(2)	0.075(1)	0.058(1)	0.058(1)	–0.0269(8)	0.0022(8)	–0.0251(8)
O(7)	2*i*	0.5444(2)	0.0609(2)	1.2698(2)	0.0543(9)	0.0516(9)	0.0471(8)	–0.0055(7)	0.0020(7)	–0.0188(7)
O(8)	2*i*	0.4797(2)	0.6612(2)	0.5755(2)	0.0435(9)	0.055(1)	0.100(1)	–0.0017(7)	–0.0076(9)	0.0046(9)
O(9)	2*i*	0.3060(2)	0.8983(2)	0.4942(1)	0.0377(7)	0.0572(9)	0.0478(8)	0.0055(6)	–0.0111(6)	–0.0257(7)
N(1)	2*i*	0.1935(2)	0.4229(2)	0.3294(2)	0.0379(8)	0.056(1)	0.0312(8)	0.0025(7)	–0.0027(6)	–0.0105(7)
C(1)	2*i*	1.1611(2)	0.3997(2)	0.7257(2)	0.0349(9)	0.0359(9)	0.043(1)	0.0010(7)	–0.0121(8)	–0.0151(8)
C(2)	2*i*	1.0299(2)	0.2863(2)	0.8415(2)	0.0365(9)	0.0319(9)	0.0371(9)	0.0030(7)	–0.0142(7)	–0.0117(7)
C(3)	2*i*	0.9545(2)	0.1691(2)	0.8185(2)	0.0362(9)	0.0328(9)	0.0345(9)	0.0054(7)	–0.0140(7)	–0.0135(7)
C(4)	2*i*	0.8233(2)	0.0782(2)	0.9257(2)	0.0404(9)	0.0313(9)	0.0372(9)	0.0008(7)	–0.0138(8)	–0.0129(7)
C(5)	2*i*	0.7610(2)	0.1031(2)	1.0565(2)	0.042(1)	0.0327(9)	0.0346(9)	0.0030(7)	–0.0122(8)	–0.0101(7)
C(6)	2*i*	0.8382(3)	0.2162(2)	1.0808(2)	0.054(1)	0.042(1)	0.0336(9)	–0.0004(8)	–0.0123(8)	–0.0157(8)
C(7)	2*i*	0.9729(3)	0.3043(2)	0.9752(2)	0.051(1)	0.0367(9)	0.041(1)	–0.0041(8)	–0.0155(8)	–0.0168(8)
C(8)	2*i*	0.6117(2)	0.0064(2)	1.1660(2)	0.045(1)	0.040(1)	0.038(1)	0.0009(8)	–0.0117(8)	–0.0110(8)
C(9)	2*i*	0.2098(3)	0.3254(3)	0.2590(2)	0.059(1)	0.053(1)	0.045(1)	–0.021(1)	0.001(1)	–0.012(1)
C(10)	2*i*	0.3287(3)	0.3538(2)	0.1287(2)	0.062(1)	0.046(1)	0.042(1)	–0.019(1)	0.0020(9)	–0.0193(9)
C(11)	2*i*	0.4334(2)	0.4844(2)	0.0696(2)	0.0336(9)	0.0316(8)	0.0310(9)	0.0004(7)	–0.0099(7)	–0.0097(7)
C(12)	2*i*	0.4087(2)	0.5846(2)	0.1470(2)	0.0395(9)	0.0317(9)	0.0367(9)	0.0027(7)	–0.0098(7)	–0.0122(7)
C(13)	2*i*	0.2888(2)	0.5506(2)	0.2773(2)	0.047(1)	0.046(1)	0.038(1)	0.0124(8)	–0.0114(8)	–0.0190(8)

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