Crystal structure of bis[diaqua(1,10-phenanthroline)(μ -4-sulfo-benzoato)cadmium(II)], $[\text{Cd}(\text{H}_2\text{O})_2(\text{C}_{12}\text{H}_8\text{N}_2)(\text{C}_7\text{H}_4\text{O}_5\text{S})]_2$

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Abstract

$\text{C}_{38}\text{H}_{32}\text{Cd}_2\text{N}_4\text{O}_{14}\text{S}_2$, triclinic, $P\bar{1}$ (no. 2), $a = 8.2384(8)$ Å, $b = 10.1299(9)$ Å, $c = 12.969(1)$ Å, $\alpha = 102.000(2)^\circ$, $\beta = 101.049(2)^\circ$, $\gamma = 113.453(1)^\circ$, $V = 924.7$ Å³, $Z = 1$, $R_{\text{gt}}(F) = 0.052$, $wR_{\text{ref}}(F^2) = 0.100$, $T = 293$ K.

Source of material

A mixture of $\text{Cd}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ (0.067 g, 0.25 mmol), potassium hydrogen 4-sulfobenzoate (0.063 g, 0.26 mmol), 1,10-phenanthroline (0.045 g, 0.23 mmol), and water (15 ml) was sealed in a 30 ml stainless-steel reactor with Teflon liner and heated to 423 K for 24 h. After cooling, colorless needle crystals of the title compound were collected by filtration.

Experimental details

H atoms bonded to the C atoms were placed geometrically and treated as riding, with C—H distances of 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent})$. The water H atoms found from Fourier difference maps were refined with restraints for O—H distances (0.85 Å) and $U_{\text{iso}}(\text{H})$ fixed at 0.05 Å².

Discussion

Recently complexes with sulfonate groups have attracted much attention due to their wide applications as surfactants and dyes and could exhibit abundant coordination topologies [1–2]. However, complexes with 4-sulfobenzoic acid (H_2sb), a ligand with sulfonate and carboxyl groups, are still rare [3,4]. Here, we present a dimer with sb ligand, $[\text{Cd}_2(\text{sb})_2(\text{phen})_2(\text{H}_2\text{O})_4]$.

The molecular structure is a centrosymmetrical dimeric loop, in which the cadmium atom has a environment defined by two O atom donors from two water molecules, two N atoms from one 1,10-phenanthroline, and three atoms from two sb ligands ($\text{CN} = 7$). The sulfonate group and the carboxyl group of each sb ligand in the title compound are in monodentate and chelating modes, re-

spectively, while in $[\text{Cd}(\text{sb})(2,2'\text{-bipy})(\text{H}_2\text{O})]$ [3] the carboxyl group is in bridging-chelating mode. Thus, the title compound is a dimeric species with the Cd...Cd separation of 9.931(1) Å and the other compound with bipyridine is a 1D polymer.

The bond distances of Cd—O(SO_3^-) [2.324(4) Å] and Cd—N [2.311(4) Å and 2.403(4) Å] in the title structure are remarkably similar to those of the cited structure [3]. The chelating Cd—O distances are also quite similar. The dihedral angle between the planes of the sb ring and its carboxyl group is 19.2(6)°. The C13—O1 bond length [1.277(7) Å] is longer than the C13—O2 bond length [1.235(5) Å] indicating more *keto* character. Two sb ligands in the dimeric species are arranged into *cis* and are fully coplanar with slightly $\pi\cdots\pi$ interaction due to partly stacking. It is worth noting that the coordinated water molecule forms an intramolecular hydrogen bond with the sulfonate O5 oxygen [O7—H7B...O5ⁱ, 3.039(7) Å, symmetry code: (i) $-x+2, -y+2, -z$]. Moreover, hydrogen bonds are formed among the dimeric loops via water molecules O6 and O7, carboxylate O1 oxygen atom, and sulfonate O5 oxygen atom [O6—H6B...O1ⁱⁱ, 2.872(6) Å; O6—H6A...O5ⁱⁱⁱ, 2.929(7) Å; O7—H7A...O1ⁱⁱ, 2.825(6) Å; O7—H7B...O5^{iv}, 2.885(6) Å; symmetry codes: (ii) $-x+1, -y+1, -z$; (iii) $-x+1, -y+2, -z$; (iv) $x, y-1, z$]. Therefore, a 2D hydrogen bonded network is formed. From above discussed detail it is obvious that the intra- and intermolecular hydrogen bonded interactions enhance the stability of the structure.

Table 1. Data collection and handling.

Crystal:	colorless plate, size 0.08 × 0.14 × 0.16 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	13.43 cm ⁻¹
Diffractionmeter, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{\text{max}}$:	50.2°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	6782, 3274
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 3208
$N(\text{param})_{\text{refined}}$:	283
Programs:	SHELX-97 [5], ORTEP-3 [6], WinGX [7]

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Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U _{iso}
H(1)	2i	0.7562	0.9430	0.3797	0.052
H(2)	2i	0.7530	1.0354	0.5558	0.064
H(3)	2i	0.7378	0.8940	0.6732	0.063
H(5)	2i	0.7104	0.6484	0.7027	0.063
H(6)	2i	0.7162	0.4246	0.6418	0.068
H(8)	2i	0.7604	0.2429	0.4955	0.066
H(9)	2i	0.8069	0.1845	0.3274	0.062
H(10)	2i	0.8176	0.3413	0.2199	0.051

Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(15)	2i	0.5278	0.8081	−0.0969	0.050
H(16)	2i	0.5430	0.9636	−0.2061	0.047
H(18)	2i	1.0022	1.2972	0.0254	0.044
H(19)	2i	0.9813	1.1445	0.1359	0.043
H(7A)	2i	0.624(4)	0.396(6)	0.026(4)	0.050
H(6B)	2i	0.400(9)	0.420(6)	0.101(3)	0.050
H(6A)	2i	0.372(8)	0.533(6)	0.157(6)	0.050
H(7B)	2i	0.821(5)	0.466(7)	0.055(5)	0.050

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Cd(1)	2i	0.75349(6)	0.65201(4)	0.21276(3)	0.0436(3)	0.0278(2)	0.0243(2)	0.0158(2)	0.0107(2)	0.0126(2)
S(1)	2i	0.7912(2)	1.2593(2)	−0.1899(1)	0.0387(8)	0.0361(8)	0.0407(8)	0.0196(7)	0.0177(7)	0.0246(7)
O(1)	2i	0.6528(6)	0.7159(5)	0.0561(4)	0.060(3)	0.036(2)	0.048(3)	0.019(2)	0.019(2)	0.025(2)
O(2)	2i	0.8334(6)	0.9152(5)	0.2016(3)	0.062(3)	0.054(3)	0.034(2)	0.024(2)	0.009(2)	0.024(2)
O(3)	2i	0.9279(6)	1.2497(5)	−0.2426(3)	0.055(3)	0.042(2)	0.043(2)	0.022(2)	0.024(2)	0.018(2)
O(4)	2i	0.6105(7)	1.1973(6)	−0.2678(5)	0.043(3)	0.097(4)	0.090(4)	0.020(3)	0.007(3)	0.073(4)
O(5)	2i	0.8554(7)	1.4128(5)	−0.1185(4)	0.095(4)	0.041(3)	0.065(3)	0.043(3)	0.049(3)	0.031(2)
O(6)	2i	0.4268(6)	0.4814(5)	0.1612(4)	0.048(3)	0.045(3)	0.040(2)	0.027(2)	0.020(2)	0.021(2)
O(7)	2i	0.7259(6)	0.4484(5)	0.0724(4)	0.042(3)	0.043(3)	0.045(3)	0.019(2)	0.022(2)	0.014(2)
N(1)	2i	0.7601(6)	0.7553(5)	0.3910(4)	0.037(3)	0.032(3)	0.029(2)	0.016(2)	0.010(2)	0.013(2)
N(2)	2i	0.7840(6)	0.4925(5)	0.3210(4)	0.035(3)	0.029(2)	0.035(3)	0.013(2)	0.011(2)	0.014(2)
C(1)	2i	0.7558(9)	0.8860(7)	0.4274(5)	0.049(4)	0.042(4)	0.038(3)	0.021(3)	0.008(3)	0.016(3)
C(2)	2i	0.7511(1)	0.9413(8)	0.5328(5)	0.073(5)	0.049(4)	0.042(4)	0.033(4)	0.022(4)	0.010(3)
C(3)	2i	0.7426(9)	0.8580(8)	0.6023(5)	0.057(4)	0.067(5)	0.034(3)	0.030(4)	0.021(3)	0.008(3)
C(4)	2i	0.7415(8)	0.7178(7)	0.5673(5)	0.034(3)	0.059(4)	0.030(3)	0.018(3)	0.009(3)	0.020(3)
C(5)	2i	0.7246(9)	0.6200(9)	0.6333(5)	0.039(4)	0.076(5)	0.037(4)	0.016(4)	0.011(3)	0.031(4)
C(6)	2i	0.7289(9)	0.4868(9)	0.5972(6)	0.048(4)	0.076(5)	0.048(4)	0.018(4)	0.011(3)	0.050(4)
C(7)	2i	0.7526(8)	0.4398(7)	0.4920(5)	0.034(3)	0.049(4)	0.045(4)	0.010(3)	0.006(3)	0.030(3)
C(8)	2i	0.7680(9)	0.3069(8)	0.4526(6)	0.047(4)	0.044(4)	0.069(5)	0.011(3)	0.006(4)	0.038(4)
C(9)	2i	0.7938(9)	0.2714(7)	0.3529(6)	0.049(4)	0.035(4)	0.061(5)	0.020(3)	−0.001(3)	0.013(3)
C(10)	2i	0.8006(8)	0.3670(7)	0.2887(5)	0.041(4)	0.037(3)	0.049(4)	0.019(3)	0.005(3)	0.014(3)
C(11)	2i	0.7541(7)	0.6712(6)	0.4596(4)	0.023(3)	0.039(3)	0.032(3)	0.009(2)	0.006(2)	0.016(3)
C(12)	2i	0.7628(7)	0.5303(6)	0.4218(4)	0.022(3)	0.038(3)	0.034(3)	0.009(2)	0.005(2)	0.020(3)
C(13)	2i	0.7466(8)	0.8594(7)	0.1019(5)	0.041(3)	0.035(3)	0.041(4)	0.018(3)	0.017(3)	0.025(3)
C(14)	2i	0.7534(8)	0.9596(6)	0.0305(4)	0.040(3)	0.032(3)	0.029(3)	0.019(3)	0.014(2)	0.016(2)
C(15)	2i	0.6231(8)	0.9069(7)	−0.0719(5)	0.042(3)	0.029(3)	0.041(3)	0.004(3)	0.003(3)	0.015(3)
C(16)	2i	0.6327(8)	0.9994(6)	−0.1378(5)	0.041(3)	0.035(3)	0.032(3)	0.010(3)	0.002(3)	0.016(3)
C(17)	2i	0.7754(7)	1.1444(6)	−0.1018(4)	0.030(3)	0.030(3)	0.034(3)	0.014(2)	0.013(2)	0.017(2)
C(18)	2i	0.9062(8)	1.1988(6)	0.0009(5)	0.039(3)	0.026(3)	0.039(3)	0.008(3)	0.009(3)	0.013(3)
C(19)	2i	0.8941(8)	1.1071(6)	0.0665(5)	0.037(3)	0.034(3)	0.030(3)	0.015(3)	0.000(2)	0.010(2)

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