Crystal structure of tetraammonium bis[(pyridine-2,6-dicarboxylato-*O,N,O'*)cadmate(II)] pentahydrate, $(NH_4)_4[Cd(C_7H_3NO_4)_2]_2 \cdot 5H_2O$

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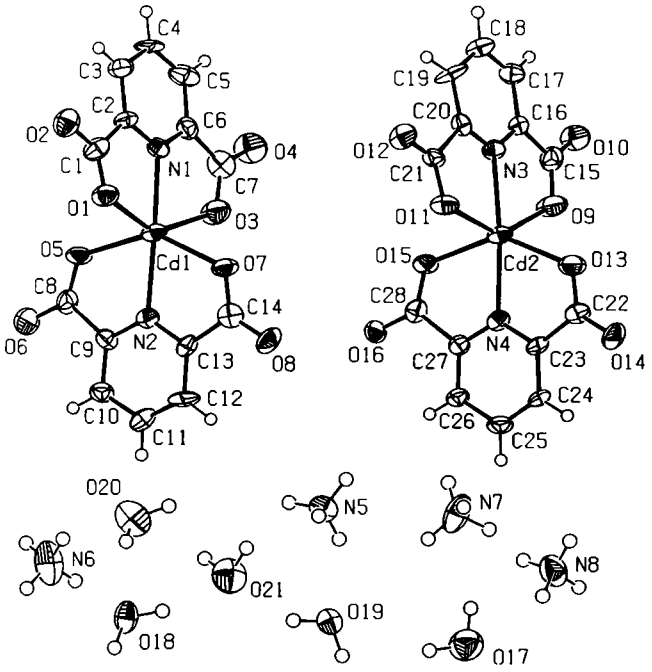

Abstract

$C_{28}H_{38}Cd_2N_8O_{21}$, monoclinic, $P12_1/c1$ (no. 14), $a = 16.440(5)$ Å, $b = 16.586(5)$ Å, $c = 15.332(5)$ Å, $\beta = 115.695(5)°$, $V = 3767.3$ Å^3, $Z = 4$, $R_{gt}(F) = 0.051$, $wR_{ref}(F^2) = 0.139$, $T = 299$ K.

Source of material

A solution (10 ml) of distillation water containing $Cd(NO_3)_2 \cdot 6H_2O$ (0.5 mmol) was added slowly to an aqueous mixture (10 ml) of pyridine-2,6-dicarboxylic acid (2 mmol) and ammonia (2 mmol). The mixture was stirred for 2 h and left to stand at room temperature for about three weeks. Colorless block-shaped crystals were obtained.

Elemental analysis: found – C, 31.97 %; H, 3.55 %; N, 10.63 %; calc. for $C_{28}H_{38}CdN_8O_{21}$ – C, 32.10 %; H, 3.66 %; N, 10.70 %.

Experimental details

The water and ammonium H atoms were localized from Fourier difference maps and their positions were fixed at idealized positions. The H atoms bonded to the C atoms were localized geometrically and treated as riding with C—H distances of 0.93 Å. All U_{iso}(H) values were fixed at 1.2 U_{eq} of the parent atoms. The low N_{gt}/N_{param} ratio is caused by crystal decomposition due to a water loss during measurement.

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Discussion

Recently, some transition metal complexes with the 2,6-pyridinedicarboxylic acid ligand (H_2DPC) have been reported, such as $(C_2H_{10}N_2)[Zn(C_2H_8N_2)_2(H_2O)_2][Zn(C_7H_3NO_4)_2]_2 \cdot 2H_2O$ [1], $(C_2H_{10}N_2)_2[Cd(C_2H_8N_2)_3] \cdot 6H_2O$ [2].

The title complex crystallizes with an asymmetric unit consisting of two similar $[Cd(C_7H_3NO_4)_2]^{2-}$ anions, four ammonium cations and five uncoordinated water molecules. For each anion, the Cd(II) atom in a distorted octahedral environment with four O atoms and two N atoms from two pyridine-2,6-dicarboxylate ligands. The averaged bond lengths of Cd(II)—N(imine) (2.228(6) Å) and Cd(II)—O(carboxyl) (2.338(6) Å) in the Cd1 octahedron are similar to the corresponding values in the Cd2 octahedron (Cd(II)—O 2.348(6) Å, Cd(II)—N 2.285(6) Å). All Cd—N and Cd—O bond distances are in normal range compared to those in the similar compounds. Four oxygen atoms each constitute the equatorial planes of the distorted Cd octahedra. Three diagonal angles of the Cd1 polyhedron are 172.5(2)°, 142.0(2)°, 141.9(2)°, and of the Cd2 polyhedron 173.6(2)°, 141.8(2)°, 141.4(2)°. There is also hydrogen bonding between the water oxygen, ammonium nitrogen and carboxyl oxygen atoms from pyridine-2,6-dicarboxylate ligands, which connect the anions, cations and water molecules into a 3D network.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.10 × 0.10 × 0.25 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	12.26 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	46.84°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	16725, 5426
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3130
$N(param)_{refined}$:	532
Programs:	SHELXTL [3], SHELXTL-plus [4]

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

Atom	Site	x	y	z	U_{iso}
H(29)	4e	0.5404	0.4573	0.2896	0.050
H(30)	4e	0.4673	0.4075	0.2788	0.050
H(31)	4e	0.5313	0.4279	0.3705	0.050
H(32)	4e	0.4719	0.4866	0.3123	0.050
H(33)	4e	0.7464	1.0337	0.4223	0.071
H(34)	4e	0.7179	0.9629	0.4491	0.071
H(35)	4e	0.6987	0.9768	0.3515	0.071
H(36)	4e	0.6549	1.0211	0.3955	0.071
H(37)	4e	0.8833	−0.0240	0.8547	0.062
H(38)	4e	0.8915	0.0121	0.7769	0.062

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(39)	4e	0.8894	0.0595	0.8505	0.062
H(40)	4e	0.8108	0.0197	0.7870	0.062
H(41)	4e	0.7531	0.5698	0.5380	0.059
H(42)	4e	0.7961	0.6258	0.5034	0.059
H(43)	4e	0.7079	0.6392	0.4903	0.059
H(44)	4e	0.7262	0.5775	0.4386	0.059
H(45)	4e	0.8434	0.4557	0.5277	0.069
H(46)	4e	0.8956	0.5205	0.5721	0.069
H(47)	4e	0.8359	0.9876	0.6327	0.045
H(48)	4e	0.7951	0.9163	0.5964	0.045
H(49)	4e	0.5986	0.5286	0.5104	0.034
H(50)	4e	0.6598	0.4720	0.5626	0.034
H(51)	4e	0.7551	1.0053	0.2299	0.077
H(52)	4e	0.7028	0.9389	0.2057	0.077

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(53)	4e	0.6454	0.5156	0.2255	0.069
H(54)	4e	0.6135	0.5680	0.2707	0.069
H(3)	4e	0.7673	0.7455	0.8687	0.054
H(4)	4e	0.8275	0.6409	0.9746	0.053
H(5)	4e	0.9375	0.5586	0.9629	0.057
H(10)	4e	1.1916	0.8976	0.6225	0.045
H(11)	4e	1.1809	0.8225	0.4923	0.042
H(12)	4e	1.0885	0.7179	0.4355	0.048
H(17)	4e	0.4988	0.0777	0.6439	0.045
H(18)	4e	0.3803	0.1546	0.6350	0.050
H(19)	4e	0.3330	0.2654	0.5274	0.051
H(24)	4e	0.6487	0.1893	0.0996	0.043
H(25)	4e	0.7553	0.2920	0.1517	0.047
H(26)	4e	0.7693	0.3697	0.2863	0.038

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}
Cd(1)	4e	0.96946(4)	0.72283(4)	0.69564(5)	0.0464(4)	0.0465(5)	0.0318(4)	−0.0019(4)	0.0271(3)	0.0013(3)
Cd(2)	4e	0.54362(4)	0.21155(4)	0.36756(4)	0.0478(4)	0.0446(4)	0.0308(4)	−0.0021(4)	0.0273(3)	0.0010(3)
N(1)	4e	0.9074(4)	0.6828(4)	0.7914(5)	0.030(4)	0.030(4)	0.026(4)	−0.001(3)	0.010(3)	0.006(3)
N(2)	4e	1.0490(4)	0.7637(4)	0.6168(5)	0.029(4)	0.033(4)	0.027(4)	0.006(4)	0.013(3)	0.006(3)
N(3)	4e	0.4776(5)	0.1871(4)	0.4653(4)	0.039(4)	0.036(4)	0.019(4)	0.001(4)	0.013(3)	0.005(3)
N(4)	4e	0.6189(4)	0.2459(4)	0.2820(4)	0.032(4)	0.037(4)	0.015(4)	0.003(4)	0.007(3)	0.002(3)
N(5)	4e	0.5027(4)	0.4450(4)	0.3104(5)	0.045(5)	0.040(5)	0.036(5)	−0.004(4)	0.013(4)	−0.006(4)
N(6)	4e	0.7054(5)	0.9973(4)	0.4047(5)	0.083(6)	0.042(5)	0.085(7)	0.004(5)	0.067(6)	−0.003(5)
N(7)	4e	0.8672(5)	0.0171(4)	0.8152(5)	0.047(5)	0.049(5)	0.075(6)	0.025(4)	0.040(5)	0.022(4)
N(8)	4e	0.7455(4)	0.6034(4)	0.4916(5)	0.038(5)	0.039(5)	0.070(6)	−0.001(4)	0.023(4)	−0.012(4)
O(1)	4e	0.8431(4)	0.8019(3)	0.6633(4)	0.052(4)	0.049(4)	0.044(4)	0.013(3)	0.030(3)	0.014(3)
O(2)	4e	0.7496(4)	0.8395(4)	0.7252(5)	0.048(4)	0.052(4)	0.061(5)	0.010(4)	0.026(4)	−0.001(3)
O(3)	4e	1.0437(4)	0.6047(4)	0.7784(5)	0.066(5)	0.055(4)	0.055(5)	0.018(4)	0.038(4)	0.015(4)
O(4)	4e	1.0493(4)	0.5157(4)	0.8884(5)	0.067(5)	0.062(5)	0.059(5)	0.027(4)	0.026(4)	0.030(4)
O(5)	4e	1.0632(4)	0.8302(4)	0.7802(4)	0.072(5)	0.055(4)	0.034(4)	−0.025(4)	0.039(4)	−0.011(3)
O(6)	4e	1.1516(5)	0.9276(4)	0.7692(4)	0.075(5)	0.055(4)	0.045(4)	−0.018(4)	0.031(4)	−0.016(3)
O(7)	4e	0.9275(4)	0.6451(3)	0.5553(4)	0.042(4)	0.056(4)	0.032(4)	−0.012(3)	0.024(3)	−0.007(3)
O(8)	4e	0.9666(4)	0.6120(4)	0.4362(4)	0.052(4)	0.061(4)	0.043(4)	−0.014(4)	0.024(3)	−0.030(4)
O(9)	4e	0.6168(4)	0.1028(4)	0.4694(5)	0.065(5)	0.061(5)	0.057(5)	0.021(4)	0.045(4)	0.020(4)
O(10)	4e	0.6223(4)	0.0282(4)	0.5925(4)	0.050(4)	0.053(4)	0.047(4)	0.018(4)	0.024(3)	0.018(4)
O(11)	4e	0.4238(4)	0.3019(3)	0.3311(4)	0.047(4)	0.054(4)	0.037(4)	0.001(3)	0.027(3)	0.009(3)
O(12)	4e	0.3240(4)	0.3473(4)	0.3803(4)	0.045(4)	0.046(4)	0.042(4)	0.021(3)	0.020(3)	0.013(3)
O(13)	4e	0.4955(4)	0.1296(4)	0.2287(4)	0.055(4)	0.058(4)	0.042(4)	−0.017(3)	0.031(3)	−0.007(3)
O(14)	4e	0.5168(4)	0.1024(3)	0.0976(4)	0.057(4)	0.051(4)	0.034(4)	−0.009(3)	0.020(3)	−0.021(3)
O(15)	4e	0.6462(4)	0.3174(3)	0.4484(4)	0.065(4)	0.049(4)	0.022(3)	−0.016(3)	0.027(3)	−0.005(3)
O(16)	4e	0.7270(4)	0.4142(3)	0.4248(4)	0.050(4)	0.035(4)	0.032(4)	−0.013(3)	0.020(3)	−0.008(3)
O(17)	4e	0.8919(4)	0.4698(4)	0.5758(4)	0.052(4)	0.058(5)	0.057(5)	−0.005(4)	0.017(4)	0.006(4)
O(18)	4e	0.7861(3)	0.9641(3)	0.6061(4)	0.042(4)	0.028(3)	0.052(4)	0.003(3)	0.030(3)	0.006(3)
O(19)	4e	0.6162(3)	0.4820(3)	0.5093(3)	0.039(3)	0.032(3)	0.022(3)	0.001(3)	0.019(3)	−0.003(2)
O(20)	4e	0.7032(4)	0.9882(4)	0.2244(5)	0.070(5)	0.060(5)	0.066(5)	−0.011(4)	0.033(4)	−0.012(4)
O(21)	4e	0.6614(4)	0.5418(4)	0.2785(4)	0.070(5)	0.063(5)	0.049(4)	0.003(4)	0.034(4)	−0.005(3)
C(1)	4e	0.8097(6)	0.7964(6)	0.7218(6)	0.026(5)	0.054(6)	0.038(6)	0.005(5)	0.014(5)	−0.004(5)
C(2)	4e	0.8429(5)	0.7279(5)	0.7953(6)	0.026(5)	0.043(6)	0.021(5)	−0.004(4)	0.013(4)	−0.003(4)
C(3)	4e	0.8119(6)	0.7131(6)	0.8650(7)	0.049(6)	0.055(6)	0.046(6)	−0.014(5)	0.034(5)	−0.011(5)
C(4)	4e	0.8474(6)	0.6509(6)	0.9273(6)	0.055(6)	0.060(7)	0.034(6)	−0.006(6)	0.036(5)	0.010(5)
C(5)	4e	0.9134(6)	0.6020(6)	0.9213(6)	0.047(6)	0.064(7)	0.030(6)	−0.012(5)	0.015(5)	0.010(5)
C(6)	4e	0.9424(6)	0.6209(5)	0.8494(6)	0.036(5)	0.031(5)	0.036(6)	−0.005(4)	0.015(4)	−0.002(4)
C(7)	4e	1.0171(6)	0.5753(6)	0.8393(7)	0.038(6)	0.050(7)	0.034(6)	0.005(5)	0.004(5)	0.010(5)
C(8)	4e	1.1069(6)	0.8647(5)	0.7414(6)	0.044(6)	0.036(6)	0.028(5)	−0.002(5)	0.013(5)	−0.006(4)
C(9)	4e	1.1038(5)	0.8260(5)	0.6495(6)	0.033(5)	0.033(5)	0.022(5)	−0.007(4)	0.009(4)	0.001(4)
C(10)	4e	1.1544(6)	0.8524(5)	0.6022(6)	0.042(6)	0.043(6)	0.029(5)	−0.008(5)	0.018(4)	0.000(4)
C(11)	4e	1.1460(6)	0.8077(5)	0.5239(6)	0.035(5)	0.052(6)	0.015(5)	0.006(5)	0.007(4)	−0.006(4)
C(12)	4e	1.0925(6)	0.7455(6)	0.4900(6)	0.042(6)	0.061(7)	0.026(5)	0.016(5)	0.024(5)	0.026(5)
C(13)	4e	1.0417(5)	0.7221(5)	0.5384(5)	0.036(5)	0.036(5)	0.011(4)	0.004(4)	0.007(4)	−0.003(4)
C(14)	4e	0.9734(5)	0.6542(5)	0.5076(6)	0.029(5)	0.042(6)	0.030(6)	0.009(5)	0.006(4)	0.010(5)
C(15)	4e	0.5892(6)	0.0823(5)	0.5312(7)	0.044(6)	0.040(6)	0.032(6)	−0.002(5)	0.014(5)	−0.005(5)
C(16)	4e	0.5094(5)	0.1295(5)	0.5304(6)	0.037(5)	0.028(5)	0.030(5)	−0.001(4)	0.020(4)	0.000(4)
C(17)	4e	0.4751(6)	0.1177(5)	0.5970(6)	0.047(6)	0.041(6)	0.030(5)	0.000(5)	0.020(5)	0.008(4)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(18)	4*e*	0.4068(6)	0.1645(5)	0.5935(6)	0.043(6)	0.059(6)	0.034(6)	0.008(5)	0.026(5)	0.004(5)
C(19)	4*e*	0.3755(5)	0.2288(6)	0.5267(6)	0.035(5)	0.081(7)	0.019(5)	0.029(5)	0.019(4)	0.012(5)
C(20)	4*e*	0.4108(5)	0.2345(5)	0.4609(6)	0.030(5)	0.025(5)	0.024(5)	0.001(4)	0.006(4)	0.004(4)
C(21)	4*e*	0.3838(6)	0.3006(5)	0.3846(6)	0.041(5)	0.032(5)	0.022(5)	−0.004(5)	0.016(4)	0.005(4)
C(22)	4*e*	0.5334(6)	0.1404(5)	0.1731(6)	0.034(5)	0.044(6)	0.020(5)	0.005(4)	0.010(4)	0.005(4)
C(23)	4*e*	0.6071(5)	0.2039(5)	0.2043(5)	0.031(5)	0.039(5)	0.011(4)	0.000(4)	0.009(4)	−0.003(4)
C(24)	4*e*	0.6572(6)	0.2198(5)	0.1538(6)	0.055(6)	0.038(5)	0.030(5)	−0.003(5)	0.033(5)	−0.006(4)
C(25)	4*e*	0.7200(6)	0.2810(5)	0.1841(6)	0.047(6)	0.047(6)	0.030(5)	−0.003(5)	0.025(5)	0.003(5)
C(26)	4*e*	0.7294(5)	0.3265(5)	0.2652(6)	0.039(5)	0.038(5)	0.024(5)	−0.004(4)	0.019(4)	−0.003(4)
C(27)	4*e*	0.6784(5)	0.3057(5)	0.3126(6)	0.033(5)	0.032(5)	0.024(5)	0.000(4)	0.013(4)	−0.001(4)
C(28)	4*e*	0.6852(6)	0.3498(5)	0.4035(6)	0.041(6)	0.033(5)	0.020(5)	0.007(5)	0.009(5)	0.003(4)

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