Crystal structure of [μ-11,23-dimethyl-3,7,15,19-tetraazatricyclo-[19.3.1.1^{9,13}]-3,7,15,19-tetracyanomethyltetracosa-1(24),-9(25),10,12,21,23-hexaene-25,26-diolato-$\kappa^8N^3,N^7,O^{25},O^{26}$:$N^{15},N^{19},O^{25},O^{26}$]-bis[(acetato-$\kappa^2O,O'$)cadmium(II)] hydrate, $Cd_2(C_{36}H_{46}N_8O_2)(CH_3COO)_2 \cdot 0.8H_2O$

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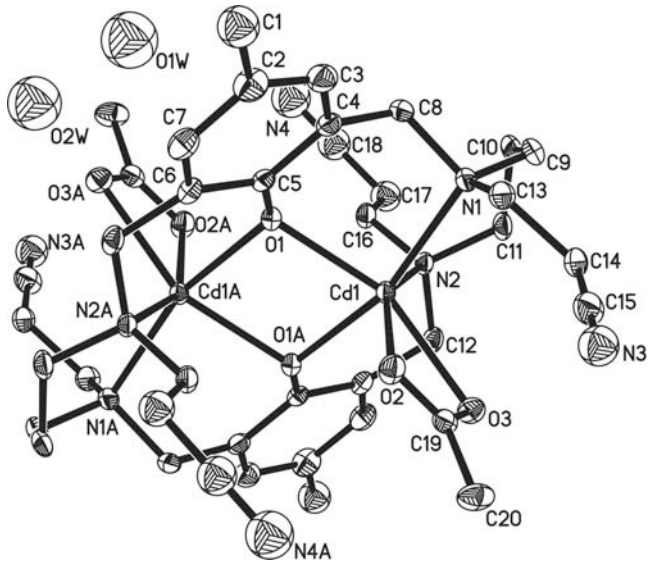

Abstract

$C_{40}H_{53.6}Cd_2N_8O_{6.80}$, trigonal, $R\bar{3}$ (no. 148), a = 31.46(1) Å, c = 12.33(3) Å, V = 10568 Å^3, Z = 9, $R_{gt}(F)$ = 0.045, $wR_{ref}(F^2)$ = 0.148, T = 293 K.

Source of material

Tetraaminodiphenol macrocyclic ligand was refluxed in acrylonitrile to give the H_2L [1]. A mixture of H_2L (0.0632 g, 0.1 mmol) and $Cd(CH_3COO)_2$ (0.05 g, 0.2 mmol) in methanol (10 ml) and $CHCl_3$ (10 ml) was stirred for 10 min and white precipitate was obtained. The precipitate was then dissolved in a minimum amount of ammonia solution. The resulting solution was filtered. Colorless single crystals of the title compound were obtained by slow evaporation of the filtrate at room temperature (yield 56 %). Chemical analysis — found: C, 49.11 %; H, 5.56 %; N, 11.51 %; calculated for $C_{40}H_{53.6}Cd_2N_8O_{6.8}$:C, 49.02 %; H, 5.51 %; N, 11.43 %.

Experimental details

All H atoms on C atoms were generated geometrically and refined as riding atoms with d(C—H) = 0.93 Å and U_{iso}(H) = 1.2 U_{eq}(C). The water H atoms were not locate from a difference Fourier map. The terminal group atoms C1, C17, C18, N3 and N4 were refined isotropically.

Discussion

Crown ether compounds have attracted much interest as a result of their significance in biological transport systems [2]. To further widen the scope of applications of crown ethers, a variety of armed crown ether deviatives were prepared.

The crystal structure of the title compound contains a dinuclear cadmium complex. Each of the two Cd(II) atoms has a distorted octahedral environment, defined by two N atoms and two O atoms from the macrocyclic ligand L^{2-} and two O atoms from a chelating acetate anion. Atoms Cd1, O1, O1A, Cd1A form a distorted square plane in which the two metal centres are separated by 3.51(1) Å.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.17 × 0.20 × 0.25 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	9.57 cm^{-1}
Diffractometer, scan mode:	Oxford Diffraction Gemini R Ultra, ω
$2\theta_{max}$:	58.76°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	27271, 5850
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4178
$N(param)_{refined}$:	236
Programs:	SHELXS-97 [3], SHELXL-97 [4]

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

Atom	Site	x	y	z	U_{iso}
C(1)	18*f*	0.5918(3)	0.2517(3)	0.5365(6)	0.112(2)
H(1A)	18*f*	0.5775	0.2568	0.6005	0.168
H(1B)	18*f*	0.5844	0.2656	0.4749	0.168
H(1C)	18*f*	0.6267	0.2672	0.5455	0.168
H(3)	18*f*	0.6064	0.1996	0.3855	0.081
H(8A)	18*f*	0.5922	0.1371	0.2668	0.065
H(8B)	18*f*	0.5463	0.0851	0.2838	0.065
H(9A)	18*f*	0.6503	0.0722	0.2423	0.078
H(9B)	18*f*	0.6340	0.1059	0.1847	0.078
H(10A)	18*f*	0.5531	0.0356	0.1694	0.073
H(10B)	18*f*	0.5929	0.0337	0.0969	0.073
H(11A)	18*f*	0.5579	–0.0401	0.1606	0.068
H(11B)	18*f*	0.5988	–0.0141	0.2481	0.068
H(12A)	18*f*	0.5263	–0.1015	0.2868	0.063
H(12B)	18*f*	0.5588	–0.0722	0.3855	0.063
H(13A)	18*f*	0.6449	0.1340	0.4616	0.071
H(13B)	18*f*	0.6682	0.1565	0.3486	0.071

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Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(01A)	18*f*	0.6681	0.0611	0.7281	0.116
H(01B)	18*f*	0.6947	0.0446	0.6448	0.116
H(01C)	18*f*	0.6526	0.0055	0.7183	0.116
H(14A)	18*f*	0.6758	0.0759	0.4218	0.073
H(14B)	18*f*	0.7117	0.1176	0.3428	0.073
H(16A)	18*f*	0.4874	–0.0122	0.2460	0.061
H(16B)	18*f*	0.4606	–0.0563	0.3262	0.061

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
C(17)	18*f*	0.4624(2)	–0.0787(2)	0.1708(5)	0.089(2)
H(17A)	18*f*	0.4869	–0.0688	0.1140	0.107
H(17B)	18*f*	0.4534	–0.1116	0.1937	0.107
C(18)	18*f*	0.4199(3)	–0.0783(3)	0.1298(6)	0.106(2)
N(3)	18*f*	0.7497(2)	0.1567(2)	0.5681(5)	0.107(2)
N(4)	18*f*	0.3873(3)	–0.0725(3)	0.1003(7)	0.154(3)
H(7)	18*f*	0.5264	0.1754	0.6495	0.079

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

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(2)	18*f*		0.5705(2)	0.1966(2)	0.5185(5)	0.072(3)	0.048(3)	0.100(4)	0.027(2)	–0.005(3)	–0.005(3)
C(3)	18*f*		0.5829(2)	0.1779(2)	0.4339(5)	0.060(3)	0.056(3)	0.085(4)	0.027(2)	0.004(2)	0.015(2)
C(4)	18*f*		0.5625(1)	0.1282(2)	0.4154(3)	0.042(2)	0.051(2)	0.058(2)	0.026(2)	–0.002(2)	0.008(2)
C(5)	18*f*		0.5266(1)	0.0944(1)	0.4871(3)	0.037(2)	0.048(2)	0.045(2)	0.025(2)	–0.006(2)	0.001(2)
C(8)	18*f*		0.5762(2)	0.1101(2)	0.3178(3)	0.049(2)	0.068(3)	0.048(2)	0.032(2)	0.007(2)	0.021(2)
C(9)	18*f*		0.6228(2)	0.0774(2)	0.2314(4)	0.062(3)	0.082(3)	0.056(3)	0.040(3)	0.026(2)	0.023(2)
C(10)	18*f*		0.5824(2)	0.0328(2)	0.1713(3)	0.060(3)	0.095(3)	0.035(2)	0.045(3)	0.016(2)	0.009(2)
C(11)	18*f*		0.5692(2)	–0.0163(2)	0.2190(3)	0.052(2)	0.086(3)	0.042(2)	0.041(2)	0.009(2)	–0.006(2)
C(19)	18*f*		0.6271(1)	0.0288(2)	0.5945(3)	0.035(2)	0.060(3)	0.044(2)	0.015(2)	–0.002(2)	0.009(2)
C(12)	18*f*		0.5282(2)	–0.0814(2)	0.3484(3)	0.056(2)	0.067(3)	0.048(2)	0.041(2)	–0.001(2)	–0.015(2)
C(13)	18*f*		0.6539(2)	0.1267(2)	0.3916(4)	0.038(2)	0.058(3)	0.070(3)	0.015(2)	0.003(2)	0.014(2)
C(20)	18*f*		0.6639(2)	0.0356(2)	0.6789(4)	0.064(3)	0.076(3)	0.075(3)	0.022(3)	–0.028(3)	0.008(3)
C(14)	18*f*		0.6924(2)	0.1110(2)	0.4085(4)	0.038(2)	0.068(3)	0.071(3)	0.022(2)	0.012(2)	0.016(2)
C(15)	18*f*		0.7245(2)	0.1362(2)	0.4976(4)	0.048(3)	0.091(4)	0.071(3)	0.021(3)	–0.001(2)	0.012(3)
C(16)	18*f*		0.4838(1)	–0.0435(2)	0.2667(3)	0.042(2)	0.082(3)	0.036(2)	0.036(2)	–0.003(2)	–0.007(2)
N(1)	18*f*		0.6093(1)	0.0891(1)	0.3372(3)	0.037(2)	0.060(2)	0.045(2)	0.023(2)	0.006(1)	0.012(2)
N(2)	18*f*		0.5315(1)	–0.0354(1)	0.3053(2)	0.039(2)	0.066(2)	0.032(2)	0.033(2)	0.001(1)	–0.004(1)
O(1)	18*f*		0.50646(9)	0.04667(9)	0.4673(2)	0.037(1)	0.044(1)	0.041(1)	0.021(1)	0.003(1)	–0.002(1)
O(2)	18*f*		0.6083(1)	0.0562(1)	0.5978(2)	0.053(2)	0.081(2)	0.052(2)	0.037(2)	–0.008(1)	–0.012(2)
O(3)	18*f*		0.6162(1)	–0.0022(1)	0.5225(2)	0.056(2)	0.061(2)	0.054(2)	0.030(2)	–0.010(1)	–0.005(1)
Cd(1)	18*f*		0.558896(9)	0.01850(1)	0.44930(2)	0.0313(2)	0.0494(2)	0.0326(2)	0.0204(1)	0.00082(9)	–0.0002(1)
C(6)	18*f*		0.5128(2)	0.1126(2)	0.5765(3)	0.047(2)	0.054(2)	0.053(2)	0.032(2)	–0.006(2)	–0.007(2)
C(7)	18*f*		0.5352(2)	0.1633(2)	0.5901(4)	0.069(3)	0.061(3)	0.077(3)	0.040(3)	–0.011(3)	–0.015(2)
O(1W)	18*f*	0.25	0.512(1)	0.341(1)	0.699(3)	0.18(1)	0.18(1)	0.18(1)	0.090(5)	–0.000(1)	–0.000(1)
O(2W)	18*f*	0.15	0.571(2)	0.346(2)	0.815(4)	0.15(1)	0.15(1)	0.15(1)	0.075(6)	0.000(1)	0.000(1)

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