Crystal structure of diaqua(biphenylethene-4,4′-dicarboxylato-κO)-(1,12,15-triaza-3,4:9,10-dibenzo-5,8-dioxacycloheptadecane-$\kappa^3 N,N',N''$)cobalt(II), $[Co(H_2O)_2(C_{20}H_{27}N_3O_2)][(C_{16}H_{10}O_4)]$

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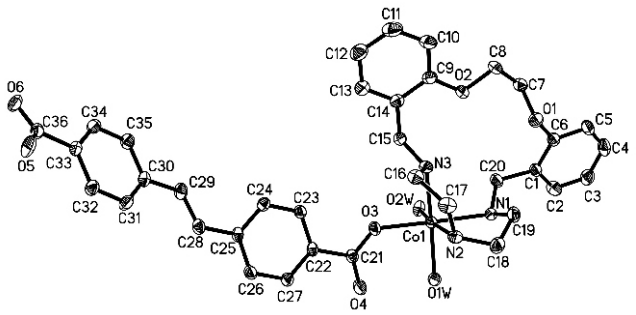

Abstract

$C_{36}H_{41}CoN_3O_8$, monoclinic, $P12_1/c1$ (no. 14), a = 15.200(5) Å, b = 19.496(7) Å, c = 12.008(3) Å, β = 111.21(1)°, V = 3317.4 Å^3, Z = 4, $R_{gt}(F)$ = 0.050, $wR_{ref}(F^2)$ = 0.103, T = 293 K.

Source of material

A mixture of $CoCO_3$ (11.9 mg, 0.10 mmol) and biphenylethene-4,4′-dicarboxylic acid (26.8 mg, 0.10 mmol) in water was stirred for 10 min at 60 °C, then 1,12,15-triaza-3,4:9,10-dibenzo-5,8-dioxacycloheptadecane (L, 34.1 mg, 0.10 mmol) was added. After stirring for 30 min, a necessary amount of ammonia (14 mL) was added to get a clear solution. Red single crystals of the title compound were obtained by slow evaporation of the solution at ambient temperature (yield 57%, based on $CoCO_3$).

Experimental details

All H atoms on C atoms were generated geometrically and refined as riding atoms with d(C—H) = 0.97 Å (CH_2) or 0.93 Å (CH), and U_{iso}(H) = 1.2 U_{eq}(C). The H atoms bonded to nitrogen and water were located from difference Fourier map and refined with U_{iso}(H) = 1.5 U_{eq}(O/N).

Discussion

In the asymmetric unit of the title compound, there are one Co(II) atom, one L ligand, one biphenylethene-4,4′-dicarboxylate ligand, and two coordinated water molecules. The Co(II) cation is six-coordinated by three nitrogen atoms from the L ligand, one oxygen atom from the biphenylethene-4,4′-dicarboxylate ligand, and one water molecule, forming a distorted octahedral coordination. The adjacent molecules are linked by the N–H···O and O–H···O hydrogen bonds to form a 2D supramolecular layer.

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Table 1. Data collection and handling.

Crystal:	red block, size 0.202 × 0.278 × 0.304 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	5.75 cm^{-1}
Diffractometer, scan mode:	Rigaku RAXIS-RAPID, ω
$2\theta_{max}$:	54.92°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	30984, 7542
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4827
$N(param)_{refined}$:	454
Programs:	SHELXS-97, SHELXL-97 [1]

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

Atom	Site	x	y	z	U_{iso}
H(2)	4*e*	–0.0710	0.0736	0.3044	0.063
H(3)	4*e*	–0.1793	–0.0031	0.3244	0.064
H(4)	4*e*	–0.1681	–0.1171	0.2836	0.065
H(5)	4*e*	–0.0470	–0.1554	0.2264	0.057
H(7A)	4*e*	0.0230	–0.0671	0.0287	0.057
H(7B)	4*e*	0.0227	–0.1449	0.0622	0.057
H(8A)	4*e*	0.1876	–0.1484	0.0993	0.056
H(8B)	4*e*	0.1268	–0.1335	–0.0355	0.056
H(10)	4*e*	0.2727	–0.1440	–0.0408	0.067
H(11)	4*e*	0.3821	–0.1239	–0.1298	0.079
H(12)	4*e*	0.4329	–0.0141	–0.1437	0.075
H(13)	4*e*	0.3787	0.0757	–0.0618	0.060
H(15A)	4*e*	0.2852	0.1223	0.0375	0.046
H(15B)	4*e*	0.1899	0.0835	0.0143	0.046
H(16A)	4*e*	0.4170	0.1064	0.2423	0.052
H(16B)	4*e*	0.4090	0.0268	0.2188	0.052
H(17A)	4*e*	0.3811	0.0062	0.3910	0.059
H(17B)	4*e*	0.4672	0.0567	0.4283	0.059
H(18A)	4*e*	0.2821	0.1065	0.5451	0.060
H(18B)	4*e*	0.3372	0.0375	0.5522	0.060
H(19A)	4*e*	0.2218	–0.0002	0.3735	0.057
H(19B)	4*e*	0.1727	0.0184	0.4646	0.057
H(20A)	4*e*	0.1021	0.0360	0.1792	0.050
H(20B)	4*e*	0.0339	0.0951	0.1831	0.050
H(23)	4*e*	0.4084	0.2197	0.0783	0.042
H(24)	4*e*	0.5219	0.2466	0.0018	0.043
H(26)	4*e*	0.6161	0.3964	0.2438	0.048
H(27)	4*e*	0.4972	0.3726	0.3147	0.047
H(28)	4*e*	0.7132	0.3615	0.1257	0.047
H(29)	4*e*	0.6151	0.2931	–0.0802	0.050
H(31)	4*e*	0.8516	0.3389	0.0997	0.052
H(32)	4*e*	0.9816	0.3234	0.0477	0.050
H(34)	4*e*	0.8151	0.2689	–0.2813	0.047
H(35)	4*e*	0.6854	0.2792	–0.2276	0.047
H(1A)	4*e*	0.117(2)	0.114(1)	0.366(2)	0.055
H(2A)	4*e*	0.384(2)	0.136(1)	0.464(2)	0.062
H(3A)	4*e*	0.255(2)	0.034(1)	0.191(2)	0.053
H(1C)	4*e*	0.183(1)	0.237(2)	0.405(3)	0.062
H(1D)	4*e*	0.259(2)	0.266(1)	0.372(3)	0.062
H(2C)	4*e*	0.076(2)	0.207(2)	0.167(3)	0.064
H(2D)	4*e*	0.119(2)	0.224(1)	0.087(2)	0.064

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}
Co(1)	4*e*	0.24081(2)	0.15524(2)	0.27738(3)	0.0330(2)	0.0302(2)	0.0360(2)	–0.0032(2)	0.0164(1)	–0.0031(2)
C(1)	4*e*	0.0078(2)	0.0063(1)	0.2530(2)	0.039(2)	0.036(2)	0.036(1)	–0.006(1)	0.010(1)	0.002(1)
C(2)	4*e*	–0.0657(2)	0.0274(2)	0.2884(3)	0.048(2)	0.044(2)	0.065(2)	–0.002(2)	0.021(2)	–0.005(2)
C(3)	4*e*	–0.1309(2)	–0.0183(2)	0.3004(3)	0.046(2)	0.064(2)	0.058(2)	–0.004(2)	0.027(2)	0.001(2)
C(4)	4*e*	–0.1238(2)	–0.0862(2)	0.2766(3)	0.044(2)	0.059(2)	0.058(2)	–0.014(2)	0.015(2)	0.012(2)
C(5)	4*e*	–0.0516(2)	–0.1092(2)	0.2424(2)	0.052(2)	0.036(2)	0.053(2)	–0.008(1)	0.016(2)	0.005(1)
C(6)	4*e*	0.0141(2)	–0.0627(1)	0.2322(2)	0.038(2)	0.037(2)	0.031(1)	–0.003(1)	0.008(1)	0.003(1)
C(7)	4*e*	0.0609(2)	–0.1036(2)	0.0776(2)	0.057(2)	0.042(2)	0.041(2)	–0.011(1)	0.015(1)	–0.006(1)
C(8)	4*e*	0.1457(2)	–0.1155(1)	0.0451(3)	0.061(2)	0.029(2)	0.052(2)	–0.006(1)	0.022(2)	–0.005(1)
C(9)	4*e*	0.2596(2)	–0.0451(1)	0.0007(2)	0.051(2)	0.040(2)	0.038(2)	0.000(1)	0.018(1)	–0.005(1)
C(10)	4*e*	0.2936(2)	–0.0996(2)	–0.0455(3)	0.071(2)	0.039(2)	0.059(2)	–0.000(2)	0.026(2)	–0.012(2)
C(11)	4*e*	0.3590(2)	–0.0874(2)	–0.0988(3)	0.065(2)	0.068(2)	0.071(2)	0.007(2)	0.031(2)	–0.023(2)
C(12)	4*e*	0.3900(2)	–0.0221(2)	–0.1063(3)	0.058(2)	0.074(3)	0.066(2)	–0.002(2)	0.035(2)	–0.014(2)
C(13)	4*e*	0.3568(2)	0.0315(2)	–0.0577(2)	0.053(2)	0.052(2)	0.050(2)	–0.004(2)	0.024(2)	–0.004(1)
C(14)	4*e*	0.2921(2)	0.0214(1)	–0.0032(2)	0.043(2)	0.040(2)	0.034(1)	0.003(1)	0.014(1)	–0.000(1)
C(15)	4*e*	0.2579(2)	0.0800(1)	0.0526(2)	0.047(2)	0.033(1)	0.038(1)	–0.002(1)	0.018(1)	0.001(1)
C(16)	4*e*	0.3844(2)	0.0650(1)	0.2504(2)	0.036(2)	0.041(2)	0.052(2)	0.007(1)	0.016(1)	–0.003(1)
C(17)	4*e*	0.4005(2)	0.0525(2)	0.3812(2)	0.043(2)	0.046(2)	0.048(2)	0.009(1)	0.006(1)	–0.002(1)
C(18)	4*e*	0.2970(2)	0.0712(2)	0.4979(2)	0.055(2)	0.057(2)	0.035(2)	–0.003(2)	0.012(1)	0.007(1)
C(19)	4*e*	0.2069(2)	0.0371(2)	0.4171(2)	0.052(2)	0.046(2)	0.041(2)	–0.009(1)	0.014(1)	0.008(1)
C(20)	4*e*	0.0726(2)	0.0579(1)	0.2293(2)	0.043(2)	0.037(2)	0.042(2)	–0.010(1)	0.012(1)	–0.001(1)
C(21)	4*e*	0.3663(2)	0.2733(1)	0.2554(2)	0.032(2)	0.032(1)	0.044(2)	0.001(1)	0.018(1)	0.001(1)
C(22)	4*e*	0.4409(2)	0.2932(1)	0.2055(2)	0.027(1)	0.032(1)	0.040(1)	0.002(1)	0.016(1)	0.006(1)
C(23)	4*e*	0.4497(2)	0.2558(1)	0.1112(2)	0.033(2)	0.034(1)	0.040(1)	–0.004(1)	0.015(1)	–0.001(1)
C(24)	4*e*	0.5185(2)	0.2714(1)	0.0662(2)	0.034(2)	0.041(2)	0.036(1)	0.001(1)	0.017(1)	0.000(1)
C(25)	4*e*	0.5835(2)	0.3238(1)	0.1155(2)	0.028(1)	0.033(1)	0.042(1)	0.004(1)	0.016(1)	0.007(1)
C(26)	4*e*	0.5740(2)	0.3610(1)	0.2095(2)	0.037(2)	0.036(2)	0.051(2)	–0.008(1)	0.022(1)	–0.005(1)
C(27)	4*e*	0.5029(2)	0.3464(1)	0.2530(2)	0.043(2)	0.035(2)	0.048(2)	–0.003(1)	0.027(1)	–0.004(1)
C(28)	4*e*	0.6632(2)	0.3353(1)	0.0763(2)	0.030(2)	0.041(2)	0.050(2)	–0.003(1)	0.019(1)	0.000(1)
C(29)	4*e*	0.6700(2)	0.3113(1)	–0.0246(2)	0.027(2)	0.053(2)	0.044(2)	–0.001(1)	0.013(1)	0.006(1)
C(30)	4*e*	0.7537(2)	0.3102(1)	–0.0576(2)	0.030(2)	0.039(2)	0.040(1)	0.000(1)	0.017(1)	0.004(1)
C(31)	4*e*	0.8437(2)	0.3237(2)	0.0233(2)	0.036(2)	0.066(2)	0.035(1)	–0.006(1)	0.019(1)	–0.009(1)
C(32)	4*e*	0.9217(2)	0.3148(2)	–0.0082(2)	0.029(2)	0.060(2)	0.040(2)	–0.006(1)	0.016(1)	–0.007(1)
C(33)	4*e*	0.9121(2)	0.2932(1)	–0.1216(2)	0.033(2)	0.032(1)	0.037(1)	0.002(1)	0.017(1)	0.004(1)
C(34)	4*e*	0.8229(2)	0.2817(1)	–0.2037(2)	0.038(2)	0.045(2)	0.036(1)	–0.002(1)	0.016(1)	–0.002(1)
C(35)	4*e*	0.7449(2)	0.2888(1)	–0.1718(2)	0.029(2)	0.050(2)	0.038(1)	–0.003(1)	0.011(1)	–0.000(1)
C(36)	4*e*	0.9986(2)	0.2826(1)	–0.1535(2)	0.041(2)	0.033(2)	0.048(2)	–0.001(1)	0.026(1)	0.001(1)
O(1)	4*e*	0.0889(1)	–0.0856(1)	0.2006(2)	0.045(1)	0.048(1)	0.039(1)	0.0017(9)	0.0129(9)	0.0018(9)
O(2)	4*e*	0.1929(2)	–0.05109(9)	0.0530(2)	0.073(2)	0.032(1)	0.060(1)	–0.012(1)	0.039(1)	–0.0096(9)
O(3)	4*e*	0.3327(1)	0.21374(9)	0.2273(2)	0.050(1)	0.038(1)	0.060(1)	–0.0122(9)	0.037(1)	–0.0087(9)
O(4)	4*e*	0.3449(2)	0.3141(1)	0.3206(2)	0.068(2)	0.039(1)	0.090(2)	–0.013(1)	0.058(1)	–0.018(1)
O(5)	4*e*	1.0761(1)	0.2730(1)	–0.0689(2)	0.035(1)	0.085(2)	0.049(1)	0.011(1)	0.025(1)	0.014(1)
O(6)	4*e*	0.9881(1)	0.2849(1)	–0.2615(2)	0.048(1)	0.064(1)	0.042(1)	–0.003(1)	0.0291(9)	–0.0039(9)
N(1)	4*e*	0.1481(2)	0.0885(1)	0.3327(2)	0.040(1)	0.035(1)	0.037(1)	–0.006(1)	0.017(1)	–0.003(1)
N(2)	4*e*	0.3469(2)	0.1018(1)	0.4252(2)	0.041(2)	0.042(1)	0.037(1)	–0.006(1)	0.010(1)	–0.006(1)
N(3)	4*e*	0.2823(2)	0.0723(1)	0.1836(2)	0.036(1)	0.032(1)	0.038(1)	–0.000(1)	0.013(1)	–0.005(1)
O(1W)	4*e*	0.2391(1)	0.2306(1)	0.4004(2)	0.038(1)	0.044(1)	0.050(1)	–0.0067(9)	0.0256(9)	–0.0090(9)
O(2W)	4*e*	0.1226(1)	0.1976(1)	0.1448(2)	0.040(1)	0.049(1)	0.045(1)	0.0062(9)	0.0225(9)	0.0124(9)

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References

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