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

Hong-Ye Bai and Jian-Fang Ma*

Department of Chemistry, Northeast Normal University, Changchun 130024, P. R. China

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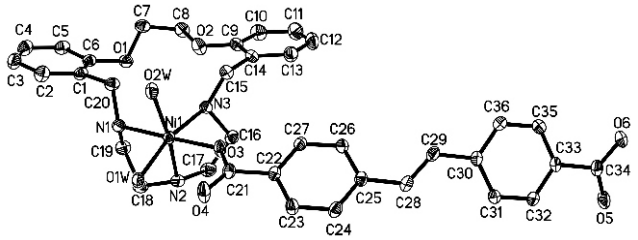

Abstract

$C_{36}H_{41}N_3NiO_8$, monoclinic, $P12_1/c1$ (no. 14), a = 15.193(3) Å, b = 19.503(4) Å, c = 11.983(3) Å, β = 111.088(9)°, V = 3312.9 Å^3, Z = 4, $R_{gt}(F)$ = 0.047, $wR_{ref}(F^2)$ = 0.100, T = 293 K.

Source of material

A mixture of $NiCO_3$ (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. Green single crystals of the title compound were obtained by slow evaporation of the solution at ambient temperature (yield 71 %, based on $NiCO_3$).

Experimental details

All H atoms on C atoms were generated geometrically and refined as riding atoms with d(C—H) = 0.97 (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 a difference Fourier map and refined with U_{iso}(H) = 1.5 U_{eq}(O/N).

Discussion

The asymmetric unit of the title compound consists of one Ni(II) atom, one L ligand, one biphenylethene-4,4′-dicarboxylate ligand, and two coordinated water molecules. The Ni(II) cation is six-coordinated by three nitrogen atoms from the L ligand, one oxygen atom from the biphenylethene-4,4′-dicarboxylate ligand, and two water molecule, showing a distorted octahedral coordination. The biphenylethene-4,4′-dicarboxylate ligand coordinates to the Ni(II) center in a mondentate mode. There are intermolecular N–H···O and O–H···O hydrogen bonding between carboxylate oxygen atoms and coordinated water molecules, which connect the molecules to form a two-dimensional supramolecular network.

* Correspondence author (e-mail: majf247nenu@yahoo.com.cn)

Table 1. Data collection and handling.

Crystal:	green block, size 0.153 × 0.180 × 0.202 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	6.43 cm^{-1}
Diffractometer, scan mode:	Rigaku RAXIS-RAPID, ω
$2\theta_{max}$:	54.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	31720, 7577
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4934
$N(param)_{refined}$:	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*	0.4332	0.0747	0.3033	0.059
H(3)	4*e*	0.3248	–0.0008	0.3264	0.063
H(4)	4*e*	0.3334	–0.1153	0.2858	0.062
H(5)	4*e*	0.4527	–0.1552	0.2263	0.054
H(7A)	4*e*	0.5249	–0.0679	0.0288	0.055
H(7B)	4*e*	0.5246	–0.1459	0.0611	0.055
H(8A)	4*e*	0.6892	–0.1492	0.0982	0.055
H(8B)	4*e*	0.6286	–0.1337	–0.0366	0.055
H(10)	4*e*	0.7750	–0.1431	–0.0428	0.066
H(11)	4*e*	0.8848	–0.1209	–0.1316	0.076
H(12)	4*e*	0.9321	–0.0109	–0.1469	0.071
H(13)	4*e*	0.8773	0.0783	–0.0635	0.058
H(15A)	4*e*	0.7823	0.1231	0.0375	0.044
H(15B)	4*e*	0.6884	0.0828	0.0159	0.044
H(16A)	4*e*	0.9178	0.1053	0.2383	0.048
H(16B)	4*e*	0.9084	0.0256	0.2172	0.048
H(17A)	4*e*	0.8838	0.0074	0.3923	0.058
H(17B)	4*e*	0.9695	0.0583	0.4262	0.058
H(18A)	4*e*	0.7840	0.1089	0.5446	0.059
H(18B)	4*e*	0.8405	0.0403	0.5538	0.059
H(19A)	4*e*	0.7258	–0.0001	0.3757	0.056
H(19B)	4*e*	0.6752	0.0198	0.4649	0.056
H(20A)	4*e*	0.6058	0.0344	0.1780	0.048
H(20B)	4*e*	0.5378	0.0940	0.1799	0.048
H(23)	4*e*	0.9966	0.3711	0.3135	0.046
H(24)	4*e*	1.1160	0.3956	0.2430	0.047
H(26)	4*e*	1.0235	0.2451	0.0007	0.043
H(27)	4*e*	0.9095	0.2177	0.0770	0.041
H(28)	4*e*	1.2140	0.3605	0.1252	0.045
H(29)	4*e*	1.1156	0.2937	–0.0816	0.049
H(31)	4*e*	1.3528	0.3379	0.0998	0.053
H(32)	4*e*	1.4823	0.3216	0.0474	0.049
H(35)	4*e*	1.3166	0.2704	–0.2840	0.045
H(36)	4*e*	1.1865	0.2799	–0.2299	0.047
H(1A)	4*e*	0.621(2)	0.115(1)	0.365(2)	0.053
H(2A)	4*e*	0.888(2)	0.136(1)	0.467(2)	0.060
H(3A)	4*e*	0.757(2)	0.034(1)	0.193(2)	0.049

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1C)	4*e*	0.684(1)	0.236(2)	0.399(3)	0.059
H(1D)	4*e*	0.765(2)	0.263(1)	0.376(3)	0.059
H(2C)	4*e*	0.585(2)	0.206(2)	0.175(3)	0.061
H(2D)	4*e*	0.622(2)	0.224(1)	0.088(2)	0.061

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}
Ni(1)	4*e*	0.74449(2)	0.15278(2)	0.27901(3)	0.0301(2)	0.0284(2)	0.0342(2)	–0.0036(1)	0.0151(1)	–0.0033(1)
C(1)	4*e*	0.5106(2)	0.0062(1)	0.2528(2)	0.035(1)	0.038(2)	0.038(2)	–0.009(1)	0.009(1)	0.003(1)
C(2)	4*e*	0.4379(2)	0.0283(2)	0.2882(3)	0.049(2)	0.040(2)	0.061(2)	–0.004(1)	0.022(2)	–0.003(1)
C(3)	4*e*	0.3725(2)	–0.0167(2)	0.3015(3)	0.042(2)	0.063(2)	0.057(2)	–0.004(2)	0.022(2)	0.004(2)
C(4)	4*e*	0.3781(2)	–0.0849(2)	0.2780(3)	0.045(2)	0.056(2)	0.055(2)	–0.014(2)	0.017(2)	0.012(2)
C(5)	4*e*	0.4494(2)	–0.1089(1)	0.2429(2)	0.047(2)	0.036(2)	0.047(2)	–0.009(1)	0.011(1)	0.004(1)
C(6)	4*e*	0.5163(2)	–0.0633(1)	0.2325(2)	0.037(1)	0.037(2)	0.032(2)	–0.004(1)	0.008(1)	0.004(1)
C(7)	4*e*	0.5628(2)	–0.1047(2)	0.0771(2)	0.053(2)	0.039(2)	0.042(2)	–0.009(1)	0.012(1)	–0.006(1)
C(8)	4*e*	0.6474(2)	–0.1161(1)	0.0444(3)	0.060(2)	0.032(2)	0.049(2)	–0.008(1)	0.023(2)	–0.005(1)
C(9)	4*e*	0.7602(2)	–0.0444(1)	–0.0002(2)	0.047(2)	0.039(2)	0.035(2)	–0.001(1)	0.016(1)	–0.003(1)
C(10)	4*e*	0.7951(2)	–0.0984(2)	–0.0471(3)	0.066(2)	0.041(2)	0.061(2)	0.001(2)	0.026(2)	–0.011(2)
C(11)	4*e*	0.8605(2)	–0.0849(2)	–0.1007(3)	0.063(2)	0.065(2)	0.071(2)	0.008(2)	0.034(2)	–0.022(2)
C(12)	4*e*	0.8897(2)	–0.0195(2)	–0.1086(3)	0.056(2)	0.069(2)	0.063(2)	–0.002(2)	0.036(2)	–0.014(2)
C(13)	4*e*	0.8561(2)	0.0339(2)	–0.0595(3)	0.049(2)	0.048(2)	0.053(2)	–0.004(1)	0.024(2)	–0.005(1)
C(14)	4*e*	0.7914(2)	0.0225(1)	–0.0045(2)	0.043(2)	0.037(2)	0.030(1)	0.002(1)	0.013(1)	–0.002(1)
C(15)	4*e*	0.7565(2)	0.0802(1)	0.0530(2)	0.044(2)	0.029(1)	0.040(2)	–0.001(1)	0.018(1)	0.000(1)
C(16)	4*e*	0.8850(2)	0.0645(1)	0.2486(2)	0.033(1)	0.040(2)	0.046(2)	0.005(1)	0.013(1)	–0.004(1)
C(17)	4*e*	0.9026(2)	0.0536(2)	0.3804(3)	0.042(2)	0.048(2)	0.048(2)	0.010(1)	0.006(1)	–0.000(1)
C(18)	4*e*	0.7998(2)	0.0733(2)	0.4985(2)	0.050(2)	0.057(2)	0.035(2)	–0.004(2)	0.009(1)	0.004(1)
C(19)	4*e*	0.7099(2)	0.0378(2)	0.4174(2)	0.051(2)	0.048(2)	0.040(2)	–0.009(1)	0.014(1)	0.011(1)
C(20)	4*e*	0.5761(2)	0.0571(1)	0.2273(2)	0.043(2)	0.037(2)	0.038(2)	–0.010(1)	0.011(1)	0.000(1)
C(21)	4*e*	0.8667(2)	0.2710(1)	0.2544(2)	0.032(1)	0.031(1)	0.041(2)	0.003(1)	0.017(1)	0.003(1)
C(22)	4*e*	0.9408(2)	0.2912(1)	0.2041(2)	0.029(1)	0.028(1)	0.037(1)	0.001(1)	0.013(1)	0.005(1)
C(23)	4*e*	1.0028(2)	0.3449(1)	0.2519(2)	0.044(1)	0.033(1)	0.047(2)	–0.003(1)	0.027(1)	–0.004(1)
C(24)	4*e*	1.0742(2)	0.3600(1)	0.2086(3)	0.035(1)	0.033(1)	0.055(2)	–0.007(1)	0.021(1)	–0.006(1)
C(25)	4*e*	1.0839(2)	0.3227(1)	0.1145(2)	0.029(1)	0.035(1)	0.038(2)	0.004(1)	0.015(1)	0.006(1)
C(26)	4*e*	1.0196(2)	0.2699(1)	0.0651(2)	0.031(1)	0.042(2)	0.036(2)	0.001(1)	0.015(1)	–0.003(1)
C(27)	4*e*	0.9504(2)	0.2540(1)	0.1101(2)	0.030(1)	0.036(1)	0.038(2)	–0.005(1)	0.013(1)	–0.003(1)
C(28)	4*e*	1.1640(2)	0.3346(1)	0.0752(2)	0.030(1)	0.038(2)	0.049(2)	–0.004(1)	0.018(1)	0.000(1)
C(29)	4*e*	1.1707(2)	0.3113(1)	–0.0255(2)	0.025(1)	0.052(2)	0.044(2)	–0.002(1)	0.011(1)	0.004(1)
C(30)	4*e*	1.2547(2)	0.3101(1)	–0.0593(2)	0.032(1)	0.038(1)	0.038(2)	–0.000(1)	0.018(1)	0.003(1)
C(31)	4*e*	1.3449(2)	0.3230(2)	0.0230(2)	0.034(1)	0.066(2)	0.036(2)	–0.006(1)	0.018(1)	–0.009(1)
C(32)	4*e*	1.4224(2)	0.3139(1)	–0.0091(2)	0.029(1)	0.058(2)	0.040(2)	–0.006(1)	0.017(1)	–0.007(1)
C(33)	4*e*	1.4138(2)	0.2936(1)	–0.1229(2)	0.035(1)	0.031(1)	0.038(2)	–0.000(1)	0.021(1)	0.003(1)
C(34)	4*e*	1.5006(2)	0.2837(1)	–0.1547(3)	0.043(2)	0.031(1)	0.048(2)	–0.003(1)	0.027(1)	0.001(1)
C(35)	4*e*	1.3242(2)	0.2825(1)	–0.2060(2)	0.041(2)	0.041(2)	0.034(2)	0.001(1)	0.016(1)	–0.001(1)
C(36)	4*e*	1.2460(2)	0.2893(1)	–0.1739(2)	0.031(1)	0.046(2)	0.039(2)	–0.005(1)	0.011(1)	–0.001(1)
O(1)	4*e*	0.5907(1)	–0.08730(9)	0.2007(2)	0.042(1)	0.045(1)	0.037(1)	0.0017(9)	0.0109(9)	0.0004(9)
O(2)	4*e*	0.6944(1)	–0.05143(9)	0.0534(2)	0.076(1)	0.031(1)	0.060(1)	–0.015(1)	0.041(1)	–0.0112(9)
O(3)	4*e*	0.8345(1)	0.21103(9)	0.2285(2)	0.048(1)	0.034(1)	0.056(1)	–0.0109(9)	0.035(1)	–0.0067(9)
O(4)	4*e*	0.8440(1)	0.3124(1)	0.3186(2)	0.068(1)	0.039(1)	0.089(2)	–0.013(1)	0.060(1)	–0.018(1)
O(5)	4*e*	1.5777(1)	0.2747(1)	–0.0697(2)	0.036(1)	0.090(2)	0.050(1)	0.010(1)	0.025(1)	0.014(1)
O(6)	4*e*	1.4905(1)	0.2859(1)	–0.2629(2)	0.050(1)	0.061(1)	0.042(1)	–0.003(1)	0.030(1)	–0.003(1)
N(1)	4*e*	0.6517(1)	0.0883(1)	0.3306(2)	0.037(1)	0.035(1)	0.035(1)	–0.006(1)	0.014(1)	–0.001(1)
N(2)	4*e*	0.8494(2)	0.1037(1)	0.4240(2)	0.039(1)	0.042(1)	0.036(1)	–0.004(1)	0.011(1)	–0.007(1)
N(3)	4*e*	0.7822(1)	0.0723(1)	0.1839(2)	0.035(1)	0.029(1)	0.033(1)	–0.0006(9)	0.012(1)	–0.0016(9)
O(1W)	4*e*	0.7400(1)	0.22794(9)	0.3985(2)	0.037(1)	0.039(1)	0.050(1)	–0.0081(9)	0.0252(9)	–0.0105(9)
O(2W)	4*e*	0.6270(1)	0.1953(1)	0.1445(2)	0.039(1)	0.047(1)	0.044(1)	0.0049(9)	0.0225(9)	0.0110(9)

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References

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