Crystal structure of diaqua-cinnamate-κO-(1,12,15-triaza-3,4:9,10-dibenzo-5,8-dioxacycloheptadecane-$\kappa^3 N,N',N''$)nickel(II) cinnamate hydrate, $[Ni(H_2O)_2(C_9H_7O_2)(C_{20}H_{27}N_3O_2)](C_9H_7O_2) \cdot H_2O$

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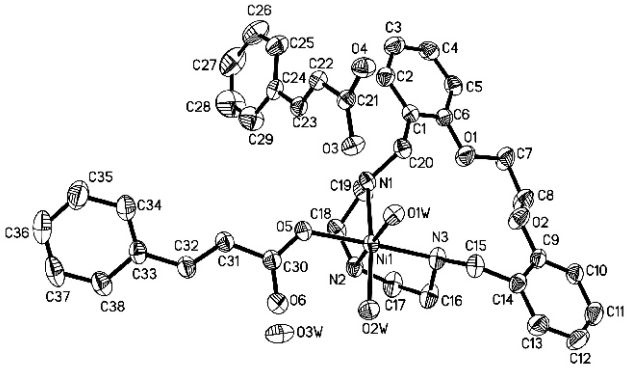

Abstract

$C_{38}H_{47}N_3NiO_9$, monoclinic, $P2_1/n$ (no. 14), a = 10.267(2) Å, b = 12.421(4) Å, c = 30.146(9) Å, β = 97.08(1)°, V = 3815.1 Å^3, Z = 4, $R_{gt}(F)$ = 0.055, $wR_{ref}(F^2)$ = 0.143, T = 293 K.

Source of material

A mixture of $NiCO_3$ (11.9 mg, 0.10 mmol) and cinnamic acid (29.6 mg, 0.20 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 64 %, 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 difference Fourier map and refined with U_{iso}(H) = 1.5 U_{eq}(O/N).

Discussion

In the asymmetric unit of the crystal structure of the title compound, there are one Ni(II) cation, one L ligand, two cinnamate anions, and three water molecules. The Ni(II) cation is six-coordinated by three nitrogen atoms from the L ligand, one oxygen atom from one cinnamate anion and two water molecules, forming a distorted octahedral coordination. Adjacent molecules are further linked by hydrogen bonds to form a dimer.

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Table 1. Data collection and handling.

Crystal:	green block, size 0.150 × 0.304 × 0.314 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	5.65 cm^{-1}
Diffractometer, scan mode:	Rigaku RAXIS-RAPID, ω
$2\theta_{max}$:	54.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	34507, 8729
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 5443
$N(param)_{refined}$:	487
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*	0.5500	–0.0525	0.3424	0.074
H(3)	4*e*	0.3555	–0.1184	0.3066	0.086
H(4)	4*e*	0.1619	–0.0841	0.3360	0.082
H(5)	4*e*	0.1626	0.0221	0.3981	0.072
H(7A)	4*e*	0.2547	0.0568	0.4806	0.096
H(7B)	4*e*	0.1857	0.1493	0.4511	0.096
H(8A)	4*e*	0.2845	0.2764	0.4943	0.096
H(8B)	4*e*	0.2376	0.1915	0.5275	0.096
H(10)	4*e*	0.2976	0.2765	0.5875	0.079
H(11)	4*e*	0.3863	0.3630	0.6517	0.096
H(12)	4*e*	0.6070	0.3852	0.6673	0.104
H(13)	4*e*	0.7446	0.3326	0.6155	0.091
H(15A)	4*e*	0.7833	0.2453	0.5493	0.067
H(15B)	4*e*	0.6862	0.1506	0.5355	0.067
H(16A)	4*e*	0.7333	0.4252	0.5057	0.087
H(16B)	4*e*	0.5822	0.4142	0.5090	0.087
H(17A)	4*e*	0.5327	0.4126	0.4331	0.095
H(17B)	4*e*	0.6307	0.5089	0.4436	0.095
H(18A)	4*e*	0.7077	0.3361	0.3508	0.078
H(18B)	4*e*	0.5823	0.4010	0.3598	0.078
H(19A)	4*e*	0.5024	0.2541	0.3954	0.077
H(19B)	4*e*	0.5346	0.2158	0.3483	0.077
H(20A)	4*e*	0.5895	0.0797	0.4472	0.063
H(20B)	4*e*	0.6666	0.0060	0.4175	0.063
H(22)	4*e*	0.8625	–0.2041	0.3569	0.060
H(23)	4*e*	0.9020	0.0108	0.3697	0.059
H(25)	4*e*	0.7217	–0.1751	0.2967	0.084
H(26)	4*e*	0.6212	–0.1422	0.2254	0.113
H(27)	4*e*	0.6427	0.0232	0.1937	0.131
H(28)	4*e*	0.7621	0.1542	0.2330	0.149
H(29)	4*e*	0.8590	0.1234	0.3044	0.103
H(31)	4*e*	1.0760	0.1925	0.3382	0.066
H(32)	4*e*	1.2011	0.3844	0.3517	0.075
H(34)	4*e*	1.2142	0.1396	0.2912	0.094
H(35)	4*e*	1.3508	0.105	0.2374	0.118
H(36)	4*e*	1.4844	0.2375	0.2155	0.118
H(37)	4*e*	1.4853	0.4032	0.248	0.109
H(38)	4*e*	1.3495	0.4405	0.3010	0.089
H(1C)	4*e*	0.907(3)	0.057(2)	0.460(1)	0.077

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(1D)	4*e*	0.911(3)	0.105(3)	0.5020(7)	0.077
H(2C)	4*e*	0.999(3)	0.349(3)	0.469(1)	0.091
H(2D)	4*e*	0.979(4)	0.303(3)	0.5092(9)	0.091
H(3D)	4*e*	0.917(5)	0.613(3)	0.409(2)	0.155

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(3C)	4*e*	0.993(4)	0.572(4)	0.376(2)	0.155
H(1A)	4*e*	0.719(3)	0.142(3)	0.381(1)	0.071
H(2A)	4*e*	0.770(3)	0.428(3)	0.411(1)	0.082
H(3A)	4*e*	0.590(2)	0.248(3)	0.481(1)	0.082

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*	0.80211(3)	0.24425(3)	0.44555(1)	0.0396(2)	0.0457(2)	0.0440(2)	0.0055(2)	0.0074(1)	0.0043(2)
C(1)	4*e*	0.4759(3)	0.0270(2)	0.3913(1)	0.044(2)	0.047(2)	0.053(2)	0.002(1)	0.007(1)	0.006(1)
C(2)	4*e*	0.4720(3)	–0.0365(3)	0.3537(1)	0.056(2)	0.065(2)	0.064(2)	0.000(2)	0.014(2)	–0.008(2)
C(3)	4*e*	0.3559(4)	–0.0770(3)	0.3323(1)	0.077(2)	0.073(3)	0.064(2)	–0.006(2)	0.004(2)	–0.015(2)
C(4)	4*e*	0.2404(3)	–0.0553(3)	0.3496(1)	0.058(2)	0.072(2)	0.073(2)	–0.014(2)	–0.003(2)	–0.003(2)
C(5)	4*e*	0.2406(3)	0.0081(3)	0.3865(1)	0.040(2)	0.072(2)	0.068(2)	–0.006(2)	0.005(2)	0.005(2)
C(6)	4*e*	0.3568(3)	0.0515(3)	0.4065(1)	0.046(2)	0.055(2)	0.055(2)	–0.002(1)	0.009(1)	–0.004(2)
C(7)	4*e*	0.2672(3)	0.1280(4)	0.4686(1)	0.050(2)	0.120(4)	0.073(2)	–0.005(2)	0.022(2)	–0.019(2)
C(8)	4*e*	0.2975(3)	0.2034(4)	0.5055(1)	0.056(2)	0.110(3)	0.076(3)	0.018(2)	0.020(2)	–0.018(2)
C(9)	4*e*	0.4692(3)	0.2501(3)	0.5636(1)	0.066(2)	0.052(2)	0.048(2)	0.006(2)	0.020(1)	0.002(2)
C(10)	4*e*	0.3878(4)	0.2866(3)	0.5933(1)	0.082(2)	0.057(2)	0.066(2)	0.002(2)	0.035(2)	–0.001(2)
C(11)	4*e*	0.4412(5)	0.3380(3)	0.6316(1)	0.118(4)	0.061(2)	0.067(3)	0.012(2)	0.038(2)	0.001(2)
C(12)	4*e*	0.5721(5)	0.3528(3)	0.6406(1)	0.135(4)	0.072(3)	0.054(2)	–0.002(3)	0.014(2)	–0.016(2)
C(13)	4*e*	0.6549(4)	0.3195(3)	0.6098(1)	0.092(3)	0.073(3)	0.061(2)	–0.004(2)	0.005(2)	–0.002(2)
C(14)	4*e*	0.6048(3)	0.2672(3)	0.5707(1)	0.069(2)	0.050(2)	0.045(2)	0.008(2)	0.010(1)	0.004(1)
C(15)	4*e*	0.6933(3)	0.2283(3)	0.5378(1)	0.050(2)	0.069(2)	0.050(2)	0.014(2)	0.010(1)	0.012(2)
C(16)	4*e*	0.6523(4)	0.3928(3)	0.4920(1)	0.096(3)	0.061(2)	0.064(2)	0.036(2)	0.029(2)	0.015(2)
C(17)	4*e*	0.6225(4)	0.4312(3)	0.4444(1)	0.103(3)	0.062(2)	0.078(3)	0.037(2)	0.033(2)	0.024(2)
C(18)	4*e*	0.6443(3)	0.3463(3)	0.3718(1)	0.061(2)	0.073(2)	0.058(2)	–0.003(2)	–0.003(2)	0.023(2)
C(19)	4*e*	0.5728(3)	0.2421(3)	0.3773(1)	0.052(2)	0.070(2)	0.067(2)	0.001(2)	–0.006(2)	0.022(2)
C(20)	4*e*	0.6042(3)	0.0649(3)	0.4167(1)	0.042(2)	0.061(2)	0.055(2)	–0.000(1)	0.004(1)	0.007(2)
C(21)	4*e*	0.9534(3)	–0.1584(3)	0.4200(1)	0.038(1)	0.051(2)	0.045(2)	0.006(1)	0.007(1)	–0.003(1)
C(22)	4*e*	0.8903(3)	–0.1429(3)	0.3732(1)	0.052(2)	0.053(2)	0.046(2)	–0.001(1)	0.011(1)	–0.004(1)
C(23)	4*e*	0.8710(3)	–0.0495(3)	0.3534(1)	0.043(2)	0.056(2)	0.050(2)	0.001(1)	0.013(1)	–0.004(1)
C(24)	4*e*	0.8051(3)	–0.0305(3)	0.3079(1)	0.049(2)	0.063(2)	0.043(2)	0.009(2)	0.011(1)	–0.000(2)
C(25)	4*e*	0.7310(4)	–0.1079(3)	0.2838(1)	0.093(3)	0.061(2)	0.055(2)	0.010(2)	0.004(2)	–0.003(2)
C(26)	4*e*	0.6702(5)	–0.0885(4)	0.2412(1)	0.109(3)	0.108(4)	0.061(3)	0.023(3)	–0.011(2)	–0.019(3)
C(27)	4*e*	0.6827(6)	0.0094(5)	0.2225(2)	0.143(5)	0.127(5)	0.051(3)	0.046(4)	–0.007(3)	0.013(3)
C(28)	4*e*	0.7535(6)	0.0870(5)	0.2459(2)	0.183(6)	0.094(4)	0.089(4)	0.000(4)	–0.009(4)	0.042(3)
C(29)	4*e*	0.8129(4)	0.0683(4)	0.2885(1)	0.104(3)	0.076(3)	0.074(3)	–0.003(2)	–0.003(2)	0.023(2)
C(30)	4*e*	1.0229(3)	0.2867(3)	0.3903(1)	0.052(2)	0.057(2)	0.058(2)	–0.001(2)	0.011(2)	0.002(2)
C(31)	4*e*	1.0957(3)	0.2579(3)	0.3525(1)	0.049(2)	0.058(2)	0.061(2)	–0.001(2)	0.011(1)	0.002(2)
C(32)	4*e*	1.1859(3)	0.3180(3)	0.3377(1)	0.063(2)	0.065(2)	0.062(2)	–0.005(2)	0.017(2)	0.006(2)
C(33)	4*e*	1.2657(3)	0.2939(3)	0.3020(1)	0.049(2)	0.075(2)	0.050(2)	0.002(2)	0.009(1)	0.014(2)
C(34)	4*e*	1.2681(4)	0.1939(4)	0.2826(1)	0.077(3)	0.086(3)	0.079(3)	–0.006(2)	0.032(2)	0.004(2)
C(35)	4*e*	1.3504(5)	0.1730(4)	0.2503(2)	0.101(3)	0.108(4)	0.093(3)	0.010(3)	0.044(3)	–0.005(3)
C(36)	4*e*	1.4301(4)	0.2515(5)	0.2374(2)	0.077(3)	0.150(5)	0.075(3)	0.012(3)	0.035(2)	0.021(3)
C(37)	4*e*	1.4298(4)	0.3499(5)	0.2565(2)	0.067(2)	0.130(4)	0.079(3)	–0.015(3)	0.022(2)	0.035(3)
C(38)	4*e*	1.3487(4)	0.3721(3)	0.2884(1)	0.068(2)	0.085(3)	0.070(2)	–0.013(2)	0.012(2)	0.017(2)
O(1)	4*e*	0.3644(2)	0.1239(2)	0.44143(9)	0.057(1)	0.108(2)	0.080(2)	–0.015(1)	0.024(1)	–0.032(2)
O(2)	4*e*	0.4255(2)	0.1922(2)	0.52574(8)	0.054(1)	0.103(2)	0.066(2)	0.008(1)	0.011(1)	–0.025(2)
O(3)	4*e*	0.9887(2)	–0.0774(2)	0.44345(7)	0.066(1)	0.050(1)	0.050(1)	0.009(1)	–0.003(1)	–0.006(1)
O(4)	4*e*	0.9661(2)	–0.2538(2)	0.43318(7)	0.069(1)	0.048(1)	0.057(1)	0.002(1)	–0.002(1)	–0.001(1)
O(6)	4*e*	1.0640(3)	0.3651(2)	0.41485(9)	0.092(2)	0.073(2)	0.090(2)	–0.023(2)	0.036(2)	–0.016(2)
O(5)	4*e*	0.9296(2)	0.2254(2)	0.39708(7)	0.044(1)	0.061(1)	0.061(1)	–0.006(1)	0.017(1)	–0.004(1)
O(1W)	4*e*	0.8720(2)	0.1021(2)	0.47560(7)	0.053(1)	0.052(1)	0.049(1)	0.015(1)	–0.001(1)	–0.000(1)
O(2W)	4*e*	0.9394(2)	0.3346(2)	0.48611(8)	0.061(1)	0.064(2)	0.056(1)	–0.000(1)	0.003(1)	0.000(1)
O(3W)	4*e*	0.9140(4)	0.5505(3)	0.3893(1)	0.125(3)	0.080(2)	0.099(2)	–0.011(2)	–0.009(2)	–0.025(2)
N(1)	4*e*	0.6658(2)	0.1613(2)	0.39909(8)	0.038(1)	0.052(2)	0.053(2)	–0.001(1)	0.007(1)	0.010(1)
N(2)	4*e*	0.7127(3)	0.3822(2)	0.41532(9)	0.064(2)	0.049(2)	0.054(2)	0.006(1)	0.016(1)	0.012(1)
N(3)	4*e*	0.6650(3)	0.2746(2)	0.49250(9)	0.058(2)	0.056(2)	0.052(2)	0.020(1)	0.016(1)	0.012(1)

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References

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