Crystal structure of bis[2-((2-(4-(1*H*-imidazol-1-yl)benzylamino)-cyclohexylamino)methyl)benzoato]nickel(II) dihydrate, $Ni(C_{24}H_{27}N_4O_2)_2 \cdot 2H_2O$

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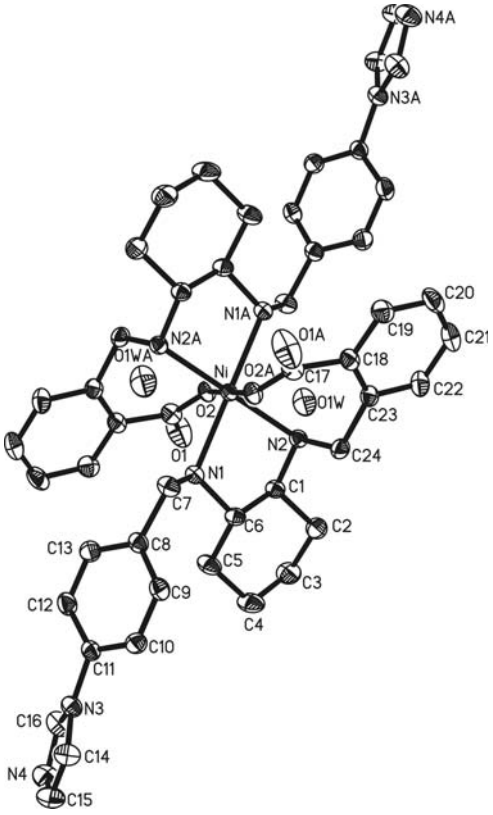

Abstract

$C_{48}H_{58}N_8NiO_6$, triclinic, $P\bar{1}$ (no. 2), a = 9.1579(4) Å, b = 10.8716(5) Å, c = 12.4645(6) Å, α = 74.678(4)°, β = 83.415(4)°, γ = 67.395(4)°, V = 1104.8 Å^3, Z = 1, $R_{gt}(F)$ = 0.035, $wR_{ref}(F^2)$ = 0.081, T = 293 K.

Source of material

4-(1*H*-imidazol-1-yl)benzaldehyde was prepared according to the literature [1]. A mixture of 2-formylbenzoic acid (1.51 g, 0.01 mol), cyclohexane-1,2-diamine (1.14 g, 0.01 mol) and methanol (100 mL) was stirred and refluxed for an hour, then the mixture was cooled to room temperature and sodium borohydride (0.76 g, 0.02 mol) was added. After stirring for one hour, 50 mL water was added, and the mixture was stirred for another one hour. Then the solvent was evaporated. The mixture was dissolved by 100 mL methanol, then 4-(1*H*-imidazol-1-yl)benzaldehyde (1.73, 0.01 mol) was added. After stirring for an hour, sodium borohydride (0.76 g, 0.02 mol) was added and the mixture was stirred for another one hour. Then methanol was evaporated and 100 mL water was added. After adding concentrated HCl drop by drop, a colorless precipitate of 2-((2-(4-(1*H*-imidazol-1-yl)benzylamino)cyclohexylamino)methyl)benzoic acid (HL) was obtained (yield 72 %). The title compound was prepared from a mixture of $NiCO_3$ (0.012 g, 0.1 mmol) and HL (0.04 g, 0.1 mmol) in 4 mL methanol and 4 mL water. This mixture was heated in a sealed Teflon-lined steel autoclave at 140 °C for three days. After the mixture was slowly cooled to room temperature at a rate of 10 °C/h, green crystals of the title compoud were obtained (yield 58 %).

Experimental details

H atoms on C and N atoms were placed in calculated positions with d(C—H) = 0.93 - 0.97 Å, d(N—H) = 0.91 Å and U_{iso}(H) = 1.2 U_{eq}(C,N). The H atoms on water molecules were located from difference Fourier maps and refined using a riding model with d(O—H) constrained to 0.85 - 0.87 Å and U_{iso}(H) = 1.5 U_{eq}(O).

Discussion

In the title crystal structure, the Ni(II) adopts a octahedral coordination, supplied by four nitrogen atoms from two different L^- ligands with d(Ni—N) = 2.107 - 2.180 Å, ∠N–Ni–N = 180°, and two oxygen atoms from two carboxylate groups of L^- in the monodentate mode with d(Ni—O) = 2.059 Å, ∠O–Ni–O = 180°. The discrete molecules are interlinked by the hydrogen bonds between the lattice water and N2, N4, O1 and O2 of L^- to form a double-chain with d(N2···O1W) = 3.242 Å, d(N4···O1W) = 2.916 Å, d(O1···O1W) = 3.332 Å, d(O2···O1W) = 3.015 Å and ∠N2–H2N···O1W = 140.7°, ∠O1W–H1A···N4 = 173°, ∠O1W–H1B···O1 = 164° and ∠O1W–H1B···O2 = 134°. In the crystal structure, the N atoms of the imidazole groups did not participate in coordination, so we can let the compound react with another kind of metal salt to form bimetal compounds.

Table 1. Data collection and handling.

Crystal:	green block, size 0.18 × 0.2 × 0.22 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	4.99 cm^{-1}
Diffractometer, scan mode:	Oxford Diffraction Gemini R Ultra, ω
$2\theta_{max}$:	58.4°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8040, 5038
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3830
$N(param)_{refined}$:	292
Programs:	SHELXS-97, SHELXL-97 [2]

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

Atom	Site	x	y	z	U_{iso}
H(1N)	2*i*	–0.0108	–0.1289	0.3564	0.038
H(2N)	2*i*	0.0637	0.2021	0.3809	0.038
H(19)	2*i*	0.3341	0.0277	0.7831	0.058
H(9)	2*i*	0.4455	–0.4199	0.3445	0.050
H(10)	2*i*	0.4955	–0.5318	0.2043	0.050
H(5A)	2*i*	0.1676	–0.1861	0.1759	0.058
H(5B)	2*i*	0.0104	–0.0565	0.1642	0.058
H(1)	2*i*	–0.0132	0.1237	0.2615	0.042
H(7A)	2*i*	0.1129	–0.3108	0.4959	0.051
H(7B)	2*i*	0.2704	–0.2851	0.4609	0.051
H(12)	2*i*	0.0373	–0.4735	0.2023	0.056
H(24A)	2*i*	0.3336	0.1242	0.3494	0.047
H(24B)	2*i*	0.3421	–0.0198	0.4227	0.047
H(6)	2*i*	0.2711	–0.1076	0.2949	0.043
H(22)	2*i*	0.3096	0.3024	0.4294	0.062
H(13)	2*i*	–0.0106	–0.3643	0.3444	0.057
H(4A)	2*i*	0.3084	–0.0527	0.0780	0.071
H(4B)	2*i*	0.1651	–0.0269	0.0058	0.071
H(2A)	2*i*	0.1357	0.2562	0.1727	0.060
H(2B)	2*i*	0.2889	0.1227	0.1840	0.060
H(20)	2*i*	0.3505	0.2409	0.7554	0.070
H(16)	2*i*	0.1066	–0.4362	–0.0077	0.063
H(14)	2*i*	0.5168	–0.7176	0.1068	0.059
H(21)	2*i*	0.3357	0.3787	0.5781	0.072
H(3A)	2*i*	0.0219	0.1782	0.0579	0.070
H(3B)	2*i*	0.1850	0.1825	0.0058	0.070
H(15)	2*i*	0.4740	–0.7334	–0.0778	0.069
H(1A)	2*i*	–0.211(3)	0.443(3)	0.237(2)	0.105
H(1B)	2*i*	–0.234(3)	0.342(3)	0.308(2)	0.105

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*b*	0	0	½	0.0289(2)	0.0286(2)	0.0287(2)	–0.0097(1)	–0.0040(1)	–0.0076(1)
O(2)	2*i*	–0.2068(1)	0.1210(1)	0.41536(9)	0.0350(6)	0.0334(6)	0.0410(6)	–0.0113(5)	–0.0108(5)	–0.0071(5)
N(3)	2*i*	0.2976(2)	–0.5595(1)	0.0877(1)	0.0451(8)	0.0353(7)	0.0369(7)	–0.0126(7)	–0.0063(6)	–0.0088(6)
N(1)	2*i*	0.0790(2)	–0.1302(1)	0.3822(1)	0.0358(7)	0.0287(7)	0.0295(6)	–0.0096(6)	–0.0049(6)	–0.0058(5)
N(2)	2*i*	0.1179(2)	0.1114(1)	0.3843(1)	0.0308(7)	0.0313(7)	0.0338(7)	–0.0101(6)	–0.0015(5)	–0.0094(5)
O(1)	2*i*	–0.3552(2)	0.1873(2)	0.2699(1)	0.112(1)	0.0536(9)	0.082(1)	–0.0274(9)	–0.066(1)	0.0142(8)
C(18)	2*i*	–0.3053(2)	–0.0379(2)	0.3779(1)	0.0275(8)	0.0423(9)	0.0462(9)	–0.0101(7)	–0.0073(7)	–0.0149(8)
N(4)	2*i*	0.2636(2)	–0.5738(2)	–0.0806(1)	0.075(1)	0.062(1)	0.0408(9)	–0.020(1)	–0.0137(8)	–0.0108(8)
C(19)	2*i*	0.3269(2)	0.0835(2)	0.7116(2)	0.041(1)	0.062(1)	0.046(1)	–0.0184(9)	–0.0094(8)	–0.0160(9)
C(9)	2*i*	0.3619(2)	–0.4272(2)	0.3139(1)	0.041(1)	0.0409(9)	0.046(1)	–0.0157(8)	–0.0073(8)	–0.0125(8)
C(10)	2*i*	0.3924(2)	–0.4941(2)	0.2300(1)	0.0353(9)	0.044(1)	0.046(1)	–0.0130(8)	0.0017(8)	–0.0141(8)
C(5)	2*i*	0.1239(2)	–0.0887(2)	0.1732(1)	0.072(1)	0.039(1)	0.0320(9)	–0.0156(9)	–0.0071(9)	–0.0093(7)
C(11)	2*i*	0.2691(2)	–0.5051(2)	0.1837(1)	0.0414(9)	0.0305(8)	0.0375(9)	–0.0137(7)	–0.0019(7)	–0.0086(7)
C(1)	2*i*	0.1006(2)	0.0863(2)	0.2754(1)	0.0392(9)	0.0344(9)	0.0299(8)	–0.0118(7)	–0.0023(7)	–0.0064(7)
C(8)	2*i*	0.2107(2)	–0.3706(2)	0.3543(1)	0.046(1)	0.0280(8)	0.0375(9)	–0.0123(7)	–0.0031(7)	–0.0065(7)
C(7)	2*i*	0.1723(2)	–0.2783(2)	0.4329(1)	0.055(1)	0.0306(9)	0.0357(9)	–0.0093(8)	–0.0058(8)	–0.0062(7)
C(23)	2*i*	0.2979(2)	0.1210(2)	0.5144(1)	0.0291(8)	0.046(1)	0.0439(9)	–0.0162(8)	–0.0039(7)	–0.0132(8)
C(12)	2*i*	0.1190(2)	–0.4597(2)	0.2290(2)	0.039(1)	0.049(1)	0.063(1)	–0.0200(9)	–0.0024(9)	–0.0224(9)
C(24)	2*i*	0.2846(2)	0.0783(2)	0.4117(1)	0.0335(8)	0.049(1)	0.0399(9)	–0.0198(8)	0.0011(7)	–0.0114(8)
C(6)	2*i*	0.1566(2)	–0.0673(2)	0.2835(1)	0.0389(9)	0.0352(9)	0.0295(8)	–0.0099(7)	–0.0021(7)	–0.0078(7)
C(17)	2*i*	–0.2897(2)	0.1011(2)	0.3526(1)	0.0358(9)	0.042(1)	0.0431(9)	–0.0080(8)	–0.0113(8)	–0.0076(8)
C(22)	2*i*	0.3120(2)	0.2472(2)	0.5008(2)	0.052(1)	0.054(1)	0.058(1)	–0.031(1)	–0.0082(9)	–0.0089(9)
C(13)	2*i*	0.0906(2)	–0.3939(2)	0.3136(2)	0.0380(9)	0.048(1)	0.061(1)	–0.0160(8)	0.0089(9)	–0.0237(9)
C(4)	2*i*	0.1939(3)	–0.0140(2)	0.0734(1)	0.082(2)	0.059(1)	0.0312(9)	–0.020(1)	0.0007(9)	–0.0118(9)
C(2)	2*i*	0.1750(2)	0.1588(2)	0.1757(1)	0.066(1)	0.047(1)	0.0393(9)	–0.028(1)	0.0027(9)	–0.0056(8)
C(20)	2*i*	0.3377(2)	0.2109(2)	0.6951(2)	0.052(1)	0.070(1)	0.070(1)	–0.025(1)	–0.011(1)	–0.037(1)
C(16)	2*i*	0.2035(2)	–0.5091(2)	–0.0024(2)	0.057(1)	0.043(1)	0.050(1)	–0.0091(9)	–0.021(1)	–0.0061(9)
C(14)	2*i*	0.4289(2)	–0.6634(2)	0.0626(2)	0.048(1)	0.049(1)	0.043(1)	–0.0078(9)	–0.0043(8)	–0.0122(8)
C(21)	2*i*	0.3295(2)	0.2927(2)	0.5895(2)	0.060(1)	0.054(1)	0.080(2)	–0.030(1)	–0.014(1)	–0.020(1)
C(3)	2*i*	0.1355(3)	0.1376(2)	0.0684(1)	0.084(2)	0.054(1)	0.0332(9)	–0.029(1)	0.001(1)	–0.0009(8)
C(15)	2*i*	0.4040(3)	–0.6705(2)	–0.0399(2)	0.065(1)	0.063(1)	0.041(1)	–0.014(1)	–0.0006(9)	–0.0194(9)
O(1W)	2*i*	–0.1816(2)	0.3939(2)	0.3021(1)	0.092(1)	0.065(1)	0.0564(9)	–0.0389(9)	–0.0223(8)	0.0047(7)

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