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Crystal structure of bis(acetylacetonato)-bis(3-methylpyridine)-nickel(II) dihydrate, $Ni(C_6H_7N)_2(C_5H_7O_2)_2 \cdot 2H_2O$

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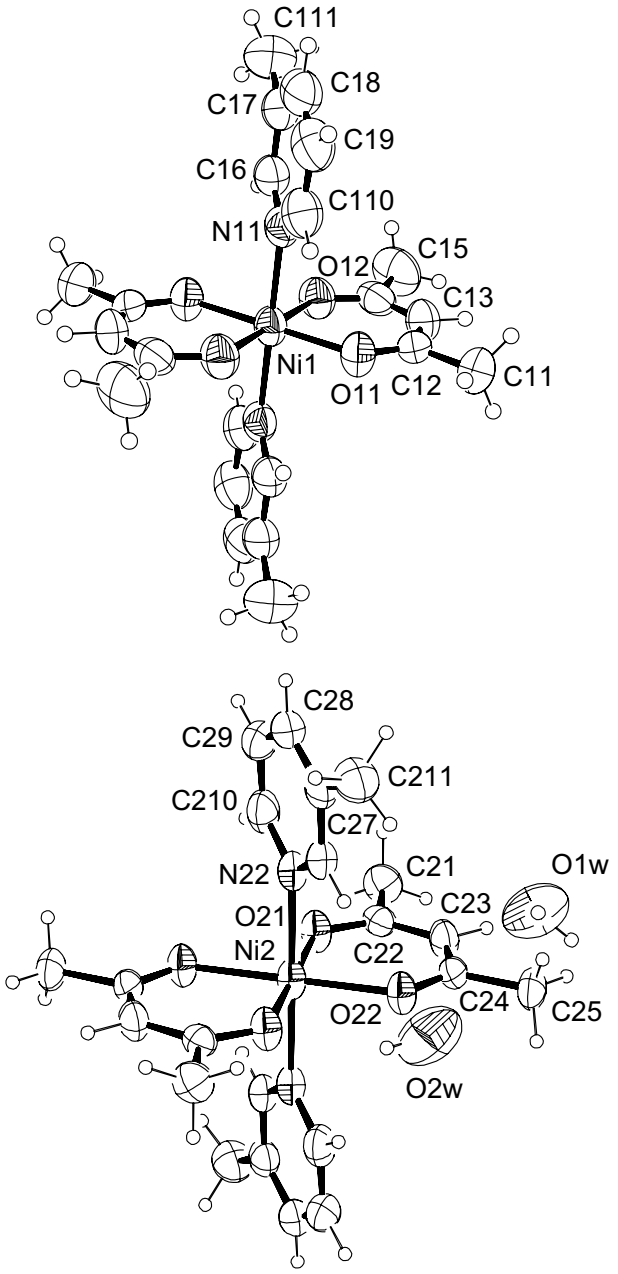

Abstract

$C_{22}H_{32}N_2NiO_6$, triclinic, $P\bar{1}$ (no. 2), a = 8.335(1) Å, b = 9.314(1) Å, c = 17.045(2) Å, α = 88.45(1)°, β = 82.12(1)°, γ = 70.296(9)°, V = 1233.7 Å^3, Z = 2, $R_{gt}(F)$ = 0.050, $wR_{ref}(F^2)$ = 0.177, T = 293 K.

Source of material

The synthesis has been described elsewhere [1]. Crystals were obtained by slow evaporization from ethanol at 277 K.

Experimental details

H atoms, except those of the water molecules, were located on stereochemical grounds and refined riding. Water H atom positions were found, refined using restraints and finally fixed.

Discussion

Adducts of Ni(II) acetylacetonate (acac) chelate with heterocyclic bases have been synthesized with the aim of establishing correlations between the bond energies and other thermodynamic parameters [1]. The title compound belongs to this group with the general formula $Ni(acac)_2L_2$ with L = 3-methylpyridine. The crystal structures of the compounds with L = 3- and 4-cyanopyridine and quinoline have already been published [2,3].

The title compound crystallizes with two independent formula units in the asymmetric unit, whereas the Ni(II) atoms are situated on centers of symmetry. Both Ni(II) atoms are bonded octahedrally to two equatorial acac groups and two 3-methylpyridine groups axially coordinated in a *trans* configuration. The Ni1—O_{acac} distances of 2.014(3) Å and 2.033(3) Å as well as Ni2—O_{acac} of 2.014(3) Å and 2.025(3) Å give rise in both independent moieties to the so-called tetragonal distortion. The acac moiety is not planar, in fact the r.m.s. deviation of On1, On2, Cn1-Cn5 being 0.017 Å and 0.008 Å, for n = 1 and 2 respectively and with Ni1 and Ni2 being 0.0878(5) Å and 0.013(5) Å, respectively, out of the plane. The water molecules are involved in hydrogen bonds with d(O1W···O11^{i}) = 2.902(7) Å, ∠O1W–H1O1···O11^{i} = 121°, d(O1W···O12^{ii}) = 2.960(6) Å, ∠O1W–H2O1···O12^{ii} = 168°; d(O2W···O22) = 2.910(7) Å, ∠O2W–H1O2···O22 = 170° (symmetry operations: i = 1–x,–y,–z; ii = 1+x,y,z).

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Table 1. Data collection and handling.

Crystal:	green, irregular, size 0.08 × 0.12 × 0.15 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	8.23 cm^{-1}
Diffractometer, scan mode:	Nonius CAD4, $\theta/2\theta$
$2\theta_{max}$:	50.94°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4911, 4574
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2277
$N(param)_{refined}$:	289
Programs:	SIR92 [4], SHELXL- 97 [5], PARST95 [6], PLATON [7], WinGX [8], ORTEP-2 [9]

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

Atom	Site	x	y	z	U_{iso}
H(120)	2*i*	0.2834	–0.3028	0.0229	0.092
H(19)	2*i*	0.4409	–0.4379	0.1183	0.105
H(18)	2*i*	0.3841	–0.3346	0.2455	0.109
H(26)	2*i*	0.7205	0.5081	0.3425	0.083
H(230)	2*i*	0.4333	0.8417	0.4854	0.107
H(28)	2*i*	0.7125	0.9286	0.2945	0.113
H(16)	2*i*	0.0212	0.0230	0.1752	0.082
H(12A)	2*i*	0.0430	–0.0012	0.3127	0.181
H(12B)	2*i*	0.2375	–0.0187	0.3099	0.181
H(12C)	2*i*	0.1712	–0.1495	0.3441	0.181
H(29)	2*i*	0.5201	1.0139	0.4085	0.119
H(11A)	2*i*	0.5809	–0.0348	–0.1140	0.141
H(11B)	2*i*	0.4746	0.0341	–0.1834	0.141
H(11C)	2*i*	0.4959	–0.1327	–0.1561	0.141
H(23A)	2*i*	0.8253	0.7121	0.1877	0.152
H(23B)	2*i*	0.8606	0.5459	0.2190	0.152
H(23C)	2*i*	0.9743	0.6404	0.2389	0.152
H(13)	2*i*	0.3805	0.2033	–0.0465	0.095
H(25A)	2*i*	0.3608	0.3702	0.2675	0.135
H(25B)	2*i*	0.1715	0.3847	0.3018	0.135
H(25C)	2*i*	0.3255	0.2460	0.3245	0.135
H(15A)	2*i*	0.0466	0.4507	0.0647	0.164
H(15B)	2*i*	0.2447	0.4085	0.0375	0.164
H(15C)	2*i*	0.1783	0.3462	0.1172	0.164
H(23)	2*i*	0.0561	0.5770	0.3924	0.090
H(21A)	2*i*	–0.1227	0.7461	0.4913	0.130
H(21B)	2*i*	–0.0329	0.8697	0.4775	0.130
H(21C)	2*i*	–0.0463	0.7958	0.5608	0.130
H(1O1)	2*i*	0.6574	0.2786	0.1495	0.201
H(2O1)	2*i*	0.8141	0.2966	0.1275	0.201
H(1O2)	2*i*	0.6623	0.2505	0.3439	0.226
H(2O2)	2*i*	0.8380	0.1876	0.3381	0.226

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)	1*a*	0	0	0	0.0552(6)	0.0572(6)	0.0657(6)	–0.0277(5)	–0.0035(5)	–0.0042(5)
O(11)	2*i*	0.2213(4)	–0.0472(4)	–0.0747(2)	0.058(2)	0.073(2)	0.077(2)	–0.030(2)	0.003(2)	–0.006(2)
O(12)	2*i*	0.0283(5)	0.1920(4)	0.0407(2)	0.071(2)	0.064(2)	0.079(2)	–0.033(2)	–0.009(2)	–0.005(2)
N(11)	2*i*	0.1384(5)	–0.1269(5)	0.0903(3)	0.061(3)	0.065(3)	0.074(3)	–0.032(2)	–0.010(2)	0.005(2)
C(14)	2*i*	0.1578(8)	0.2312(6)	0.0197(3)	0.078(4)	0.067(3)	0.073(4)	–0.037(3)	–0.033(3)	0.015(3)
C(110)	2*i*	0.2612(7)	–0.2623(7)	0.0741(4)	0.075(4)	0.065(4)	0.097(4)	–0.033(3)	–0.009(3)	–0.003(3)
C(17)	2*i*	0.1964(8)	–0.1440(8)	0.2240(4)	0.084(4)	0.094(5)	0.081(4)	–0.054(4)	–0.018(4)	0.014(4)
C(19)	2*i*	0.3560(8)	–0.3438(7)	0.1307(5)	0.077(4)	0.065(4)	0.126(6)	–0.029(3)	–0.020(4)	0.015(4)
C(18)	2*i*	0.3215(9)	–0.2819(8)	0.2063(5)	0.091(5)	0.090(5)	0.115(6)	–0.054(4)	–0.034(4)	0.034(4)
Ni(2)	1*h*	½	½	½	0.0576(6)	0.0836(7)	0.0564(6)	–0.0204(5)	–0.0111(5)	–0.0153(5)
O(22)	2*i*	0.4442(5)	0.4022(4)	0.4075(2)	0.066(2)	0.089(3)	0.061(2)	–0.020(2)	–0.014(2)	–0.019(2)
O(21)	2*i*	0.2576(4)	0.6482(4)	0.5196(2)	0.061(2)	0.091(3)	0.065(2)	–0.017(2)	–0.013(2)	–0.017(2)
C(22)	2*i*	0.1387(7)	0.6542(6)	0.4801(3)	0.062(3)	0.065(3)	0.060(3)	–0.022(3)	–0.010(3)	0.011(3)
N(22)	2*i*	0.5719(6)	0.6575(5)	0.4229(3)	0.067(3)	0.084(3)	0.063(3)	–0.029(2)	–0.018(2)	–0.007(2)
C(24)	2*i*	0.2986(8)	0.4435(6)	0.3838(3)	0.085(4)	0.070(3)	0.048(3)	–0.038(3)	–0.018(3)	0.007(3)
C(26)	2*i*	0.6805(7)	0.6115(7)	0.3560(3)	0.063(3)	0.081(4)	0.068(4)	–0.027(3)	–0.019(3)	–0.003(3)
C(27)	2*i*	0.7365(7)	0.7086(7)	0.3058(3)	0.073(4)	0.093(5)	0.072(4)	–0.038(3)	–0.027(3)	0.010(4)
C(210)	2*i*	0.5123(8)	0.8070(8)	0.4402(4)	0.098(5)	0.083(5)	0.081(4)	–0.015(4)	–0.031(4)	–0.020(4)
C(28)	2*i*	0.676(1)	0.8598(8)	0.3264(4)	0.117(6)	0.094(5)	0.095(5)	–0.053(4)	–0.051(4)	0.021(4)
C(16)	2*i*	0.1075(7)	–0.0708(6)	0.1638(3)	0.064(3)	0.074(4)	0.076(4)	–0.034(3)	–0.012(3)	0.007(3)
C(111)	2*i*	0.159(1)	–0.0719(9)	0.3049(4)	0.129(6)	0.158(7)	0.089(5)	–0.061(6)	–0.034(5)	0.017(5)
C(12)	2*i*	0.3224(6)	0.0282(7)	–0.0800(3)	0.056(3)	0.093(4)	0.052(3)	–0.032(3)	–0.015(2)	0.022(3)
C(29)	2*i*	0.562(1)	0.9109(8)	0.3942(5)	0.131(6)	0.072(4)	0.101(5)	–0.028(4)	–0.048(5)	–0.005(4)
C(11)	2*i*	0.4830(7)	–0.0317(8)	–0.1387(3)	0.068(4)	0.138(6)	0.077(4)	–0.043(4)	0.004(3)	0.008(4)
C(211)	2*i*	0.8606(8)	0.6460(8)	0.2310(4)	0.098(5)	0.123(6)	0.089(5)	–0.050(4)	–0.005(4)	0.014(4)
C(13)	2*i*	0.2956(8)	0.1586(7)	–0.0366(4)	0.078(4)	0.096(4)	0.087(4)	–0.059(4)	–0.016(3)	0.013(4)
C(25)	2*i*	0.2881(8)	0.3527(7)	0.3128(3)	0.114(5)	0.087(4)	0.077(4)	–0.034(4)	–0.037(4)	–0.017(3)
C(15)	2*i*	0.1568(9)	0.3723(7)	0.0639(4)	0.131(6)	0.092(5)	0.131(6)	–0.062(5)	–0.039(5)	–0.008(4)
C(23)	2*i*	0.1546(7)	0.5599(7)	0.4163(3)	0.069(4)	0.087(4)	0.073(4)	–0.025(3)	–0.029(3)	–0.002(3)
C(21)	2*i*	–0.0312(7)	0.7776(7)	0.5047(4)	0.073(4)	0.086(4)	0.091(4)	–0.015(3)	–0.011(3)	0.000(3)
O(1W)	2*i*	0.7164(8)	0.3353(6)	0.1553(4)	0.143(5)	0.111(4)	0.217(7)	–0.020(4)	0.031(5)	–0.032(4)
O(2W)	2*i*	0.7526(9)	0.2020(6)	0.3131(4)	0.172(6)	0.116(4)	0.252(8)	–0.024(4)	0.005(6)	–0.055(5)

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