Crystal structure of diaqua(1,10-phenanthroline)nickel(II) methanedisulfonate, $Ni(H_2O)_2(C_{12}H_8N_2)[CH_2(SO_3)_2]$

Wei-Qiu Kan and Ying-Ying Liu*

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

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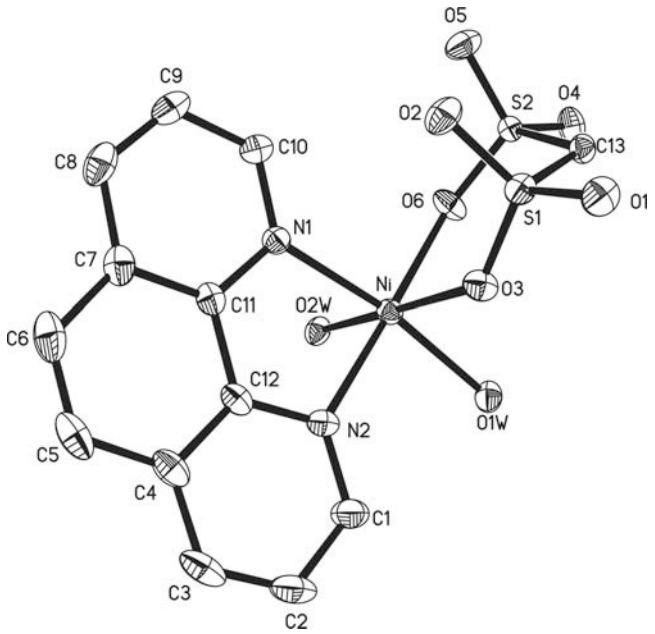

Abstract

$C_{13}H_{14}N_2NiO_8S_2$, triclinic, $P\bar{1}$ (no. 2), a = 7.6399(3) Å, b = 9.5157(4) Å, c = 11.9884(6) Å, α = 85.227(4)°, β = 78.052(4)°, γ = 87.500(3)°, V = 849.4 Å^3, Z = 2, $R_{gt}(F)$ = 0.026, $wR_{ref}(F^2)$ = 0.064, T = 293 K.

Source of material

All reagents and solvents were purchased from commercial sources and used as received. Methanedisulfonic acid (0.176 g, 1 mmol), $NiCO_3$ (0.119 g, 1 mmol), and 1,10-phenanthroline (0.180 g, 1 mmol) were added to 5 mL methanol with stirring at room temperature. After a few minutes, the solution was sealed in a 15 mL Teflon reacter, which was heated at 140 °C for 3 days and then cooled to room temperature at 10 °C/h. Green block-shaped crystals of the title compound were collected in 32 % yield.

Experimental details

All H atoms on C atoms were generated geometrically and refined as riding with d(C—H) = 0.93 - 0.97 Å and U_{iso}(H) = 1.2 U_{eq}(C). The water H atoms were located from difference Fourier map and refined with d(O—H) = 0.832 - 0.863 Å, U_{iso}(H) = 0.064.

Discussion

The crystal structure of the title compound is isomorphic to $Co(H_2O)_2(C_{12}H_8N_2)[CH_2(SO_3)_2]$ [1]. The asymmetric unit contains one Ni(II) ion, one methanedisulfonate anion, one 1,10-phenanthroline molecule and two coordinating water molecules. Ni(II) adopts slightly distorted octahedral environment, which is six-coordinated by two N atoms and four O atoms with d(Ni—N1) = 2.059 Å, d(Ni—N2) = 2.058 Å, d(Ni—O3) = 2.103 Å, d(Ni—O6) = 2.072 Å, d(Ni—O1W) = 2.067 Å, and d(Ni—O2W) = 2.063 Å, respectively.

Table 1. Data collection and handling.

Crystal:	green block, size 0.10 × 0.13 × 0.16 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	14.34 cm^{-1}
Diffractometer, scan mode:	Oxford Diffraction Gemini R Ultra, ω
$2\theta_{max}$:	58.2°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7980, 3923
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3130
$N(param)_{refined}$:	247
Programs:	SHELXS-97 [2], SHELXL-97 [3]

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

Atom	Site	x	y	z	U_{iso}
H(13A)	2*i*	0.7295	0.4925	0.4544	0.034
H(13B)	2*i*	0.8658	0.4199	0.5234	0.034
H(2)	2*i*	0.5653	0.3166	–0.1395	0.063
H(3)	2*i*	0.7071	0.1063	–0.1897	0.061
H(6)	2*i*	0.9323	–0.2688	0.0152	0.063
H(022)	2*i*	0.8999	–0.2266	0.4046	0.052
H(5)	2*i*	0.8492	–0.1262	–0.1263	0.061
H(8)	2*i*	0.9535	–0.3117	0.2270	0.055
H(025)	2*i*	0.7446	–0.0154	0.4352	0.041
H(1)	2*i*	0.5067	0.3696	0.0499	0.052
H(1B)	2*i*	0.311(1)	0.405(2)	0.278(2)	0.064
H(1A)	2*i*	0.460(3)	0.476(2)	0.299(2)	0.064
H(2A)	2*i*	0.343(3)	0.026(1)	0.345(2)	0.064
H(2B)	2*i*	0.235(2)	0.150(2)	0.332(2)	0.064

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

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	2*i*	0.56945(3)	0.22024(2)	0.28507(2)	0.0229(1)	0.0268(1)	0.0240(1)	0.0013(1)	–0.00605(9)	–0.00341(9)
S(1)	2*i*	0.92116(6)	0.34585(5)	0.34333(4)	0.0190(2)	0.0296(3)	0.0374(3)	–0.0007(2)	–0.0055(2)	–0.0003(2)
S(2)	2*i*	0.61916(6)	0.28404(5)	0.54551(4)	0.0266(2)	0.0247(2)	0.0256(2)	–0.0017(2)	–0.0067(2)	–0.0031(2)
O(1)	2*i*	1.0537(2)	0.4541(2)	0.3057(1)	0.0249(7)	0.0406(8)	0.0530(9)	–0.0089(6)	–0.0066(6)	0.0045(7)
O(5)	2*i*	0.7087(2)	0.1698(2)	0.5991(1)	0.0445(8)	0.0370(8)	0.0484(9)	0.0003(7)	–0.0144(7)	0.0129(7)
O(3)	2*i*	0.7993(2)	0.3415(1)	0.2639(1)	0.0264(7)	0.0413(8)	0.0321(7)	–0.0070(6)	–0.0063(5)	0.0009(6)
O(4)	2*i*	0.5012(2)	0.3681(2)	0.6257(1)	0.0380(8)	0.0386(8)	0.0420(8)	–0.0053(7)	0.0021(6)	–0.0156(6)
O(2)	2*i*	0.9975(2)	0.2095(2)	0.3712(1)	0.0281(7)	0.0339(8)	0.0562(9)	0.0078(6)	–0.0059(6)	0.0010(7)
C(4)	2*i*	0.7330(3)	0.0329(3)	–0.0291(2)	0.031(1)	0.056(1)	0.032(1)	–0.011(1)	–0.0024(8)	–0.012(1)
O(2W)	2*i*	0.3370(2)	0.1082(1)	0.3152(1)	0.0254(7)	0.0259(7)	0.0416(8)	0.0008(6)	–0.0090(6)	–0.0026(6)
O(6)	2*i*	0.5288(2)	0.2379(2)	0.4597(1)	0.0337(7)	0.0523(9)	0.0256(7)	–0.0169(7)	–0.0053(6)	–0.0055(6)
O(1W)	2*i*	0.4258(2)	0.4068(1)	0.2662(1)	0.0277(7)	0.0285(7)	0.0465(8)	0.0025(6)	–0.0126(6)	–0.0069(6)
C(12)	2*i*	0.6941(2)	0.0738(2)	0.0832(2)	0.0230(9)	0.040(1)	0.027(1)	–0.0057(9)	–0.0030(7)	–0.0076(8)
N(1)	2*i*	0.7111(2)	0.0310(2)	0.2792(1)	0.0244(7)	0.0279(8)	0.0280(8)	0.0006(7)	–0.0057(6)	–0.0025(6)
C(7)	2*i*	0.8381(3)	–0.1448(2)	0.1480(2)	0.026(1)	0.038(1)	0.049(1)	–0.0005(9)	–0.0020(9)	–0.012(1)
C(11)	2*i*	0.7476(2)	–0.0167(2)	0.1727(2)	0.0209(8)	0.032(1)	0.033(1)	–0.0028(8)	–0.0038(7)	–0.0069(8)
C(13)	2*i*	0.7875(2)	0.4031(2)	0.4720(2)	0.0270(9)	0.0248(9)	0.036(1)	–0.0023(8)	–0.0095(8)	–0.0033(8)
N(2)	2*i*	0.6107(2)	0.1973(2)	0.1123(1)	0.0303(8)	0.0356(9)	0.0258(8)	–0.0028(7)	–0.0076(6)	0.0013(7)
C(2)	2*i*	0.5992(3)	0.2529(3)	–0.0841(2)	0.060(2)	0.068(2)	0.032(1)	–0.012(1)	–0.019(1)	0.011(1)
C(3)	2*i*	0.6830(3)	0.1283(3)	–0.1138(2)	0.048(1)	0.081(2)	0.024(1)	–0.017(1)	–0.0054(9)	–0.009(1)
C(6)	2*i*	0.8735(3)	–0.1832(3)	0.0325(2)	0.041(1)	0.049(2)	0.067(2)	0.001(1)	–0.001(1)	–0.029(1)
C(9)	2*i*	0.8608(3)	–0.1754(2)	0.3444(2)	0.036(1)	0.037(1)	0.053(1)	0.004(1)	–0.011(1)	0.010(1)
C(5)	2*i*	0.8238(3)	–0.0984(3)	–0.0520(2)	0.042(1)	0.068(2)	0.044(1)	–0.008(1)	–0.000(1)	–0.030(1)
C(8)	2*i*	0.8941(3)	–0.2252(2)	0.2391(2)	0.033(1)	0.033(1)	0.070(2)	0.006(1)	–0.004(1)	–0.005(1)
C(10)	2*i*	0.7678(3)	–0.0473(2)	0.3622(2)	0.033(1)	0.035(1)	0.034(1)	–0.0003(9)	–0.0090(8)	0.0029(9)
C(1)	2*i*	0.5645(3)	0.2844(2)	0.0304(2)	0.050(1)	0.047(1)	0.034(1)	–0.001(1)	–0.014(1)	0.004(1)

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References

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