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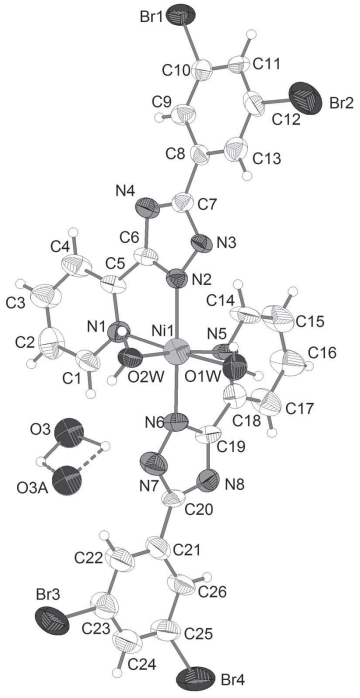
Crystal structure of diaquabis(3-(3,5-dibromophenyl)-5-(pyridin-2-yl)-1,2,4-triazol-4-ido-κ^2*N*,*N*′)nickel(II) mono hydrate, $C_{26}H_{20}Br_4N_8NiO_3$



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Abstract

$C_{26}H_{20}Br_4N_8NiO_3$, monoclinic, $P2/c$ (no. 13), $a = 13.760(4)$ Å, $b = 14.079(4)$ Å, $c = 16.317(5)$ Å, $\beta = 98.604(6)°$, $V = 3125.5(16)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0719$, $wR_{ref}(F^2) = 0.1376$, $T = 293$ K.

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Table 1: Data collection and handling.

Crystal:	Light-blue, block, size 0.17×0.21×0.26 mm
Wavelength:	MoK_α radiation (0.71073 Å)
μ:	57.76 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω scans
$2\theta_{max}$:	49.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	15584, 5497
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2546
$N(param)_{refined}$:	391
Programs:	Bruker programs [1], SHELX [2]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1A)	4*g*	0.2233	0.2455	−0.0055	0.056
H(2A)	4*g*	0.1303	0.3697	−0.0679	0.079
H(3A)	4*g*	0.1842	0.5198	−0.0451	0.079
H(4A)	4*g*	0.3378	0.5476	0.0280	0.067
H(9A)	4*g*	0.6189	0.6460	0.1621	0.051
H(11A)	4*g*	0.8841	0.6790	0.3015	0.064
H(13A)	4*g*	0.7508	0.4270	0.2891	0.060
H(14A)	4*g*	0.5883	0.3156	0.0018	0.072
H(15A)	4*g*	0.6614	0.2870	−0.1146	0.086
H(16A)	4*g*	0.6105	0.1586	−0.1958	0.088
H(17A)	4*g*	0.4928	0.0581	−0.1584	0.073
H(22A)	4*g*	0.1386	−0.0471	0.0724	0.080
H(24A)	4*g*	0.0231	−0.2803	−0.0367	0.080
H(26A)	4*g*	0.2565	−0.1625	−0.1132	0.064
H(1WB)	4*g*	0.568(6)	0.095(5)	0.126(4)	0.060
H(1WA)	4*g*	0.562(6)	0.149(5)	0.204(3)	0.060
H(2WB)	4*g*	0.310(3)	0.195(5)	0.199(5)	0.052
H(2WA)	4*g*	0.378(6)	0.267(4)	0.234(4)	0.052
O(3)[a]	4*g*	0.1780(7)	0.1497(7)	0.1510(6)	0.072(3)
O(3A)[b]	4*g*	0.147(1)	0.042(1)	0.232(1)	0.072(3)
H(31)	4*g*	0.1070	0.1127	0.1828	0.080
H(32)	4*g*	0.2417	0.0784	0.2053	0.080

[a]Disordered, occupancy factor: 0.641(7); [b]Disordered, occupancy factor: 0.359(7).

Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Table 3: Fractional coordinates and atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Br(1)	4*g*	0.75332(8)	0.80596(7)	0.19690(7)	0.0694(8)	0.0442(6)	0.0855(8)	−0.0175(6)	0.0252(6)	−0.0059(6)
Br(2)	4*g*	0.93874(9)	0.49585(9)	0.36935(9)	0.0562(8)	0.098(1)	0.131(1)	−0.0052(7)	−0.0230(8)	0.0186(9)
Br(3)	4*g*	−0.02612(9)	−0.16490(9)	0.09625(8)	0.0777(9)	0.105(1)	0.113(1)	−0.0399(8)	0.0447(8)	−0.0274(8)
Br(4)	4*g*	0.1380(1)	−0.32357(8)	−0.16805(8)	0.119(1)	0.0718(9)	0.100(1)	−0.0369(8)	0.0348(8)	−0.0349(8)
C(1)	4*g*	0.2478(6)	0.3071(7)	0.0007(5)	0.037(6)	0.057(7)	0.045(6)	−0.013(6)	0.006(5)	−0.011(5)
C(2)	4*g*	0.1907(7)	0.3815(9)	−0.0356(6)	0.049(7)	0.078(9)	0.062(8)	0.015(7)	−0.016(6)	0.012(7)
C(3)	4*g*	0.2232(8)	0.4691(8)	−0.0234(6)	0.065(8)	0.043(7)	0.082(9)	−0.006(6)	−0.011(7)	0.021(6)
C(4)	4*g*	0.3140(8)	0.4859(7)	0.0208(6)	0.059(8)	0.049(7)	0.057(7)	−0.018(6)	0.005(6)	0.016(6)
C(5)	4*g*	0.3703(7)	0.4100(7)	0.0551(5)	0.041(6)	0.039(6)	0.046(6)	−0.015(5)	0.002(5)	−0.003(5)
C(6)	4*g*	0.4635(6)	0.4182(7)	0.1080(5)	0.040(6)	0.037(6)	0.034(6)	−0.014(5)	0.008(5)	−0.005(5)
C(7)	4*g*	0.5908(7)	0.4650(6)	0.1797(5)	0.043(7)	0.031(6)	0.039(6)	−0.002(5)	0.014(5)	0.001(5)
C(8)	4*g*	0.6708(6)	0.5269(6)	0.2186(5)	0.028(6)	0.034(6)	0.049(6)	−0.001(5)	0.010(5)	0.007(5)
C(9)	4*g*	0.6716(7)	0.6204(6)	0.1975(5)	0.049(7)	0.034(6)	0.045(6)	−0.006(5)	0.011(5)	0.006(5)
C(10)	4*g*	0.7497(7)	0.6769(6)	0.2281(6)	0.045(6)	0.045(6)	0.058(7)	−0.008(6)	0.031(5)	−0.021(5)
C(11)	4*g*	0.8309(7)	0.6407(7)	0.2811(6)	0.046(7)	0.029(6)	0.092(9)	−0.011(5)	0.032(6)	−0.016(6)
C(12)	4*g*	0.8283(7)	0.5502(8)	0.3006(5)	0.030(6)	0.070(8)	0.050(7)	−0.006(6)	0.008(5)	−0.017(6)
C(13)	4*g*	0.7502(7)	0.4902(7)	0.2724(5)	0.054(7)	0.045(6)	0.055(7)	0.002(6)	0.019(6)	−0.003(5)
C(14)	4*g*	0.5698(7)	0.2621(7)	−0.0301(6)	0.066(7)	0.052(7)	0.067(7)	−0.038(6)	0.031(6)	−0.021(6)
C(15)	4*g*	0.6133(8)	0.2463(8)	−0.1006(7)	0.076(8)	0.079(9)	0.071(8)	−0.031(7)	0.045(7)	0.002(7)
C(16)	4*g*	0.5834(8)	0.1703(8)	−0.1478(6)	0.093(9)	0.086(9)	0.052(7)	−0.037(7)	0.047(6)	−0.020(7)
C(17)	4*g*	0.5139(7)	0.1105(7)	−0.1258(6)	0.061(7)	0.083(8)	0.044(7)	−0.022(6)	0.020(6)	−0.001(6)
C(18)	4*g*	0.4754(7)	0.1292(7)	−0.0540(6)	0.045(6)	0.046(6)	0.034(6)	0.003(5)	0.007(5)	0.011(5)
C(19)	4*g*	0.3984(7)	0.0738(6)	−0.0263(5)	0.055(7)	0.028(6)	0.031(6)	−0.009(5)	0.009(5)	−0.002(5)
C(20)	4*g*	0.2821(7)	−0.0164(6)	−0.0115(6)	0.046(7)	0.038(6)	0.051(7)	−0.012(5)	0.013(5)	−0.024(5)
C(21)	4*g*	0.2099(7)	−0.0925(7)	−0.0171(6)	0.039(7)	0.061(8)	0.063(8)	−0.012(6)	0.009(6)	−0.010(6)
C(22)	4*g*	0.1392(8)	−0.0943(7)	0.0327(6)	0.056(8)	0.075(8)	0.070(8)	−0.025(7)	0.015(6)	−0.019(6)
C(23)	4*g*	0.0698(7)	−0.1629(8)	0.0261(6)	0.058(8)	0.077(8)	0.060(8)	−0.013(7)	0.019(6)	−0.016(6)
C(24)	4*g*	0.0701(8)	−0.2323(7)	−0.0322(7)	0.058(8)	0.063(8)	0.078(8)	−0.019(6)	0.012(7)	−0.006(7)
C(25)	4*g*	0.1403(8)	−0.2311(7)	−0.0844(6)	0.064(8)	0.057(7)	0.066(8)	−0.023(6)	0.015(6)	−0.016(6)
C(26)	4*g*	0.2095(7)	−0.1629(7)	−0.0778(6)	0.052(7)	0.056(7)	0.056(7)	−0.025(6)	0.019(5)	−0.008(6)
N(1)	4*g*	0.3374(5)	0.3212(5)	0.0443(4)	0.041(5)	0.039(5)	0.035(4)	−0.001(4)	0.001(4)	−0.008(4)
N(2)	4*g*	0.5072(5)	0.3413(5)	0.1412(4)	0.034(5)	0.031(5)	0.043(5)	−0.002(4)	0.001(4)	0.004(4)
N(3)	4*g*	0.5914(5)	0.3707(5)	0.1884(4)	0.028(4)	0.035(5)	0.036(4)	−0.007(4)	−0.002(4)	0.000(4)
N(4)	4*g*	0.5127(5)	0.4993(5)	0.1305(4)	0.039(5)	0.041(5)	0.048(5)	−0.008(4)	0.001(4)	0.001(4)
N(5)	4*g*	0.5046(5)	0.2057(5)	−0.0069(4)	0.051(5)	0.032(5)	0.045(5)	−0.017(4)	0.016(4)	−0.012(4)
N(6)	4*g*	0.3618(5)	0.1068(5)	0.0401(4)	0.051(5)	0.033(4)	0.041(5)	−0.011(4)	0.010(4)	−0.005(4)
N(7)	4*g*	0.2872(6)	0.0473(6)	0.0499(4)	0.050(6)	0.064(6)	0.049(5)	−0.017(5)	0.009(4)	−0.014(5)
N(8)	4*g*	0.3523(5)	−0.0012(5)	−0.0588(4)	0.049(5)	0.038(5)	0.047(5)	0.000(4)	0.020(4)	0.001(4)
Ni(1)	4*g*	0.43595(8)	0.21898(7)	0.09952(7)	0.0416(7)	0.0308(7)	0.0390(7)	−0.0088(6)	0.0096(6)	−0.0043(6)
O(1W)	4*g*	0.5390(5)	0.1227(5)	0.1599(4)	0.053(5)	0.059(5)	0.037(5)	0.004(4)	0.006(3)	−0.010(4)
O(2W)	4*g*	0.3644(4)	0.2195(4)	0.2050(4)	0.047(4)	0.041(4)	0.045(4)	−0.011(4)	0.012(4)	−0.004(3)

Source of material

All of reagents were obtained commercially and used directly without further purification. In a typical reaction 0.005 g 2-(5-(3,5-dibromophenyl)-1*H*-1,2,4-triazol-3-yl)pyridine (*Hdbtp*) was dissolved in 4 mL absolute ethanol, 0.003 g $Ni(CH_3COO)_2 \cdot 4H_2O$ was added and this mixture was stirred for 1 h at room temperature. After filtered, the filtrate was kept in a 8 mL tube to allow evaporation at room temperature. After one week, light blue crystals suitable for X-ray diffraction analysis were obtained.

Experimental details

Single crystal X-ray data for the title complex was collected at room temperature yielding a completeness of 99.7%. The oxygen hydrogen distances of the coordinated water molecules were fixed at 0.83 Å with the DFIX command. The hydrogen atoms of the uncoordinated water were refined freely. All the other hydrogen atoms were placed in calculated positions using the AFIX 43 option of the SHELX program [2]. The oxygen atom of the uncoordinated water is disordered over two positions with occupancy factors of 0.641(7) (O3) and 0.359(7)

(O3A). One common isotropic displacement parameter was refined for both disordered sites.

Discussion

Ni(II) complexes play an important role due to their intriguing structures and properties [3]. It is well-known that triazole and pyridine moieties as important ligands are extensively used for coordination chemistry [4]. In the present work, we have used 2-(5-(3,5-dibromophenyl)-1*H*-1,2,4-triazol-3-yl)pyridine (*Hdbtp*) as a rigid ligand to react with $Ni(CH_3COO)_2 \cdot 4H_2O$, generating a new Ni(II) complex, formulated as $Ni(dbtp)_2(H_2O)_2 \cdot H_2O$. The asymmetric unit of the title structure contains two $dbtp^-$ ligands, one Ni(II) ion, two coordinated water molecules and one uncoordinated water molecule (figure). The Ni(II) exhibits a slightly distorted octahedral coordination geometry, defined by two pyridine nitrogen atoms and two triazole nitrogen atoms from two $dbtp^-$ ligands, and two oxygen atoms from two water molecules [Ni—N:2.039(8)–2.104(8) Å, Ni—O:2.103(8)–2.110(8) Å]. The bond angles around the Ni(II) atom vary from 78.3(3)° [N(6)—Ni(1)—N(5)] to 176.9(3)° [N(1)—Ni(1)—O(1W)].

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