Crystal structure of poly[μ_2-1,4-di(1*H*-imidazol-1-yl)butan-κ^2N:N'-μ_2-thiophene-2,3-dicarboxylato-κ^4O,O':O'',O'''nickel(II)], $C_{16}H_{16}N_4NiO_4S$

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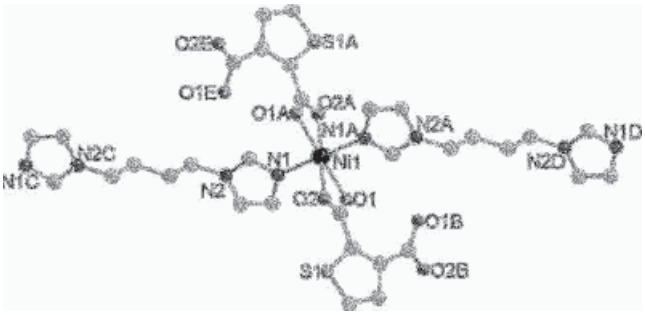
Received April 03, 2013, accepted June 18, 2013, available online August 02, 2013, CCDC no. 1267/3996



Abstract

$C_{16}H_{16}N_4NiO_4S$, monoclinic, *C*2/*c* (no. 15), a = 15.691(3) Å, b = 8.744(2) Å, c = 12.853(2) Å, β = 106.309(2)°, V = 1692.5 Å^3, Z = 4, $R_{gt}(F)$ = 0.0326, $wR_{ref}(F^2)$ = 0.0740, T = 296 K.

Table 1. Data collection and handling.

Crystal:	yellow blocks, size 0.28×0.35×0.41 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	13.01 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6135, 1577
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1260
$N(param)_{refined}$:	123
Programs:	SHELX [9]

Source of material

A mixture of *H*$_2$*tpda* (2,3-thiophene dicarboxylic acid, 0.1 mmol), Ni(OAc)$_2$·4H$_2$O (0.1 mmol), *bib* (1,4-bis(imidazol-1-yl)butane, 0.1 mmol) and distilled water (10 mL) was sealed in a 25 mL Teflon lined stainless-steel reactor, heated to 433 K and kept at constant temperature for 5 days. Slow cooling to room temperature gave yellow block-like crystals of the title compound.

Discussion

The design and construction of metal-organic frameworks (MOFs) with well-regulated network structures has been provoked significant interest on these functional materials with properties, such as electrochemistry, photophysics, catalysis, adsorption and separation [1-4]. The organic ligands, which have their differences in the size, the flexibility, the coordination ability, the number of the carboxylate groups, the positions of the carboxylate groups and so on, play crucial role in the design and construction of desirable frameworks with interesting properties [5-8]. In addition, *N*-containing auxiliary ligands are known to be ideal connectors between metal atoms for the propagation of coordination networks. With these considerations in mind, we have chosen the *tpda* and *bib* as bridging ligands to construct a new complex. The asymmetric unit contains one Ni(II) ion, one completely deprotonated *tpda*$^{2-}$ anion and one *bib* molecule, as shown in the Fig. The nickel atom is six-coordinated [NiN_2O_4] in a distorted octahedral manner. The equatorial plane is consisted of four carboxyl oxygen atoms from two *tpda*$^{2-}$ ligands, while two nitrogen atoms from two *bib* molecules locate at the axial positions. All the Ni–O bond lengths are in the range of 2.1331(17) - 2.1443(18) Å and the Ni–N bond distance is 2.055(2) Å. Along the crystallographic *c* axis, the *tpda*$^{2-}$ ligands bridge the Ni(II) centers to form 1D carboxylate chains, which are interconnected to each other through the *bib* molecules to result in a 3D metal-organic framework.

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

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(9)	8*f*	0.5	–0.0077	0.5381	–0.0980	0.045
H(3)	8*f*		0.1829	0.2049	0.0239	0.046
H(4)	8*f*		0.2963	0.2462	0.1958	0.050
H(5)	8*f*		0.0995	0.0095	0.2501	0.034
H(6A)	8*f*		0.3202	0.1236	0.3947	0.043
H(6B)	8*f*		0.2414	0.0254	0.4112	0.043
H(7A)	8*f*		0.1640	0.2494	0.4260	0.047
H(7B)	8*f*		0.2431	0.3473	0.4100	0.047
H(8)	4*e*		0	0.7139	–0.2500	0.059

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1)	8*f*	0.5	–0.0075(3)	0.5230(2)	–0.1449(2)	0.0594(9)	0.0270(8)	0.028(1)	–0.0032(7)	0.016(1)	–0.0080(7)
C(9)	8*f*	0.5	–0.004(1)	0.5064(9)	–0.1657(9)	0.0594(9)	0.0270(8)	0.028(1)	–0.0032(7)	0.016(1)	–0.0080(7)
Ni(1)	4*a*		0	0	0	0.0233(3)	0.0237(3)	0.0139(2)	–0.0008(2)	0.0033(2)	–0.0006(2)
O(1)	8*f*		0.0429(1)	0.0965(2)	–0.1292(1)	0.031(1)	0.027(1)	0.0173(9)	0.0036(8)	0.0058(8)	–0.0002(8)
O(2)	8*f*		–0.0428(1)	0.2270(2)	–0.0529(1)	0.035(1)	0.031(1)	0.024(1)	0.0045(9)	0.0137(9)	0.0014(8)
N(1)	8*f*		0.1113(1)	0.0877(2)	0.1088(2)	0.028(1)	0.031(1)	0.017(1)	–0.005(1)	0.003(1)	–0.0009(9)
N(2)	8*f*		0.2137(2)	0.1234(3)	0.2642(2)	0.028(1)	0.037(1)	0.019(1)	–0.007(1)	–0.001(1)	–0.004(1)
C(1)	8*f*		–0.0024(2)	0.3474(3)	–0.1972(2)	0.027(2)	0.024(1)	0.021(1)	0.001(1)	0.004(1)	–0.001(1)
C(2)	8*f*		–0.0012(2)	0.2160(3)	–0.1230(2)	0.025(2)	0.027(2)	0.015(1)	–0.004(1)	0.003(1)	–0.003(1)
C(3)	8*f*		0.1801(2)	0.1700(4)	0.0912(2)	0.039(2)	0.051(2)	0.024(2)	–0.014(2)	0.006(1)	0.006(1)
C(4)	8*f*		0.2432(2)	0.1932(4)	0.1859(2)	0.036(2)	0.053(2)	0.035(2)	–0.020(2)	0.007(1)	0.002(2)
C(5)	8*f*		0.1343(2)	0.0628(3)	0.2145(2)	0.029(2)	0.034(2)	0.021(2)	–0.006(1)	0.004(1)	–0.002(1)
C(6)	8*f*		0.2562(2)	0.1205(3)	0.3816(2)	0.035(2)	0.044(2)	0.021(2)	–0.004(1)	–0.004(1)	–0.004(1)
C(7)	8*f*		0.2280(2)	0.2521(4)	0.4392(2)	0.040(2)	0.047(2)	0.024(2)	0.001(2)	–0.003(1)	–0.007(1)
C(8)	4*e*		0	0.6076(5)	–0.25	0.061(3)	0.024(2)	0.065(3)	0	0.020(3)	0

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