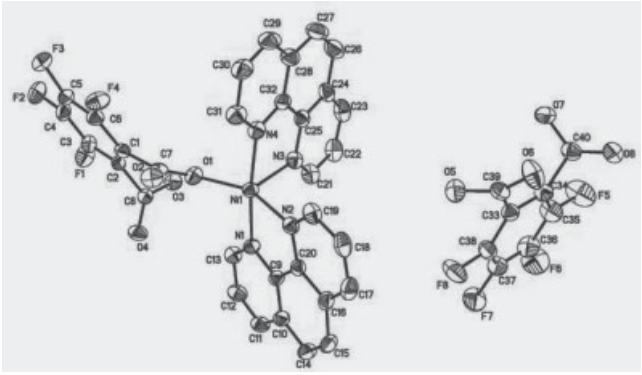
Crystal structure of bis(1,10-phenanthroline-κ^2N,N)(tetrafluorophthalato-κO)nickel (II)-tetrafluorophtalic acid (1:1), $[Ni(C_8F_4O_4)(C_{12}H_8N_2)_2](C_8H_2F_4O_4)$, $C_{40}H_{18}F_8N_4NiO_8$

Yuan-Ying Liu, Jin-Fu Guo*, Hui-Yun Li and Guo-Yan Zhao

College of Chemistry and Chemical Engineering, AnYang Normal University, Anyang 455002, Henan Province, P. R. China

Received November 19, 2012, accepted May 08, 2013, available online June 07, 2013, CCDC no. 1267/3950



Abstract

$C_{40}H_{18}F_8N_4NiO_8$, triclinic, $P\bar{1}$ (No. 2), a = 11.5061(2) Å, b = 11.8003(3) Å, c = 14.5181(3) Å, α = 111.888(2)°, β = 101.867(2)°, γ = 95.595(2)°, V = 1756.8 Å^3, Z = 2, $R_{gt}(F)$ = 0.0490, $wR_{ref}(F^2)$ = 0.1169, T = 296 K.

Table 1. Data collection and handling.

Crystal:	green blocks, size 0.20×0.20×0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	6.59 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	12767, 6501
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4350
$N(param)_{refined}$:	552

Source of material

The mixture of $Ni(OAc)_2{\cdot}4H_2O$ (24.9 mg, 0.1 mmol) and tetrafluorophthalic acid (23.6 mg, 0.1 mmol), 1,10-phenanthroline (19.8 mg, 0.1 mmol), were added to a H_2O solution (15 ml) in a Teflon-lined stainless steel reactor. The mixture was heated at 433 K for 3 days, and then slowly cooled to room temperature over 24 hours. Green, block-shaped crystals of the title compound were obtained.

Discussion

Inorganic-organic hybrid polymers have attracted considerable attention in recent years because of their interesting molecular topologies and crystal packing motifs along with potential applications in many areas including gas storage, separation, catalysis, magnetism, optics as well as electrical conductivity [1-3]. It is well know that organic ligands play a crucial role in the design and construction of desirable frameworks. Phthalic acid and its derivatives are good candidates to produce unique structural motifs with useful functional properties [4, 5]. The asymmetric unit of the title crystal structure contains one Ni(II) ion, two 1,10-phenanthroline (*phen*) ligands, and one tetrafluorophthalato ligand, and one cocrystallized tetrafluorophthalic acid molecule. Each Ni(II) ion is trigonal biyramidal coordinated by four N atoms from two chelating *phen* ligands and one O atom of the twofold deprotonated tetrafluorophthalate ligand. The bond lengths of Ni–O are 1.963(2) Å. The bond lengths of Ni–N are 1.982(3) Å, 1.983(3) Å, 2.089(3) Å and 2.176(3) Å. In the complex, there exist intermolecular hydrogen bonds: d(O7–H7···O3) = 2.731(4) Å and d(O5–H5···O3) = 2.526(3) Å between both carboxyl groups of tetrafluorophthalic acid and carboxylate group of the anionic ligand. In the complex the hydrogen bonds play an important role in forming by self-assembly and stabilizing the supramolecular structure.

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

Atom	Site	x	y	z	U_{iso}
H(11)	2*i*	0.7078	0.3662	0.6197	0.062
H(12)	2*i*	0.8610	0.5337	0.6753	0.065
H(13)	2*i*	0.8177	0.7297	0.7077	0.057
H(14)	2*i*	0.4794	0.2821	0.5664	0.067
H(15)	2*i*	0.2895	0.3216	0.5495	0.071
H(17)	2*i*	0.1474	0.4800	0.5695	0.070
H(18)	2*i*	0.1288	0.6829	0.6088	0.072
H(19)	2*i*	0.2979	0.8353	0.6563	0.061
H(21)	2*i*	0.7238	0.7775	0.8969	0.065
H(22)	2*i*	0.7796	0.8742	1.0735	0.079
H(23)	2*i*	0.7323	1.0659	1.1528	0.079
H(26)	2*i*	0.6326	1.2453	1.1346	0.087
H(27)	2*i*	0.5353	1.3286	1.0308	0.089
H(29)	2*i*	0.4409	1.3094	0.8481	0.083
H(30)	2*i*	0.3998	1.1962	0.6749	0.075
H(31)	2*i*	0.4574	1.0067	0.6119	0.065
H(5)	2*i*	0.0410	0.7935	0.8032	0.089

* Correspondence author (e-mail: aysygjf@163.com)

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}
C(1)	2*i*	0.8452(3)	1.0222(3)	0.6069(2)	0.044(2)	0.036(2)	0.036(2)	0.013(2)	0.011(1)	0.013(2)
C(2)	2*i*	0.9569(3)	0.9924(3)	0.6407(2)	0.042(2)	0.039(2)	0.035(2)	0.013(2)	0.014(1)	0.017(2)
C(3)	2*i*	1.0609(3)	1.0670(4)	0.6497(3)	0.040(2)	0.056(3)	0.049(2)	0.015(2)	0.015(2)	0.028(2)
C(4)	2*i*	1.0608(3)	1.1726(4)	0.6311(3)	0.049(2)	0.056(3)	0.053(2)	0.002(2)	0.020(2)	0.025(2)
C(5)	2*i*	0.9517(3)	1.2041(4)	0.6002(3)	0.068(2)	0.040(2)	0.047(2)	0.015(2)	0.020(2)	0.025(2)
C(6)	2*i*	0.8466(3)	1.1271(3)	0.5862(3)	0.051(2)	0.041(2)	0.044(2)	0.017(2)	0.012(2)	0.019(2)
C(7)	2*i*	0.7275(3)	0.9477(3)	0.6018(3)	0.043(2)	0.032(2)	0.046(2)	0.013(2)	0.010(2)	0.011(2)
C(8)	2*i*	0.9672(3)	0.8814(3)	0.6676(3)	0.034(2)	0.044(2)	0.052(2)	0.016(2)	0.013(2)	0.024(2)
C(9)	2*i*	0.5495(3)	0.5833(3)	0.6424(2)	0.046(2)	0.034(2)	0.034(2)	0.005(2)	0.008(1)	0.015(2)
C(10)	2*i*	0.5686(3)	0.4622(3)	0.6202(2)	0.060(2)	0.038(2)	0.039(2)	0.009(2)	0.013(2)	0.018(2)
C(11)	2*i*	0.6904(3)	0.4453(4)	0.6334(3)	0.071(2)	0.041(2)	0.048(2)	0.025(2)	0.015(2)	0.020(2)
C(12)	2*i*	0.7810(3)	0.5444(4)	0.6660(3)	0.058(2)	0.052(3)	0.054(2)	0.026(2)	0.012(2)	0.019(2)
C(13)	2*i*	0.7545(3)	0.6624(4)	0.6857(3)	0.041(2)	0.051(2)	0.053(2)	0.013(2)	0.011(2)	0.023(2)
C(14)	2*i*	0.4677(3)	0.3635(4)	0.5836(3)	0.073(3)	0.039(2)	0.054(2)	0.003(2)	0.017(2)	0.018(2)
C(15)	2*i*	0.3545(4)	0.3877(4)	0.5737(3)	0.071(3)	0.048(3)	0.056(2)	–0.010(2)	0.019(2)	0.022(2)
C(16)	2*i*	0.3307(3)	0.5105(4)	0.5989(3)	0.051(2)	0.052(3)	0.041(2)	0.002(2)	0.015(2)	0.023(2)
C(17)	2*i*	0.2159(3)	0.5415(4)	0.5905(3)	0.046(2)	0.073(3)	0.056(2)	–0.003(2)	0.013(2)	0.029(2)
C(18)	2*i*	0.2049(3)	0.6617(5)	0.6132(3)	0.041(2)	0.091(4)	0.055(2)	0.017(2)	0.019(2)	0.033(2)
C(19)	2*i*	0.3077(3)	0.7539(4)	0.6431(3)	0.043(2)	0.066(3)	0.050(2)	0.020(2)	0.014(2)	0.027(2)
C(20)	2*i*	0.4299(3)	0.6091(3)	0.6314(2)	0.043(2)	0.041(2)	0.035(2)	0.009(2)	0.012(1)	0.019(2)
C(21)	2*i*	0.7043(3)	0.8549(4)	0.9283(3)	0.045(2)	0.071(3)	0.056(2)	0.021(2)	0.011(2)	0.033(2)
C(22)	2*i*	0.7385(3)	0.9124(5)	1.0347(3)	0.050(2)	0.099(4)	0.056(3)	0.014(2)	0.007(2)	0.042(3)
C(23)	2*i*	0.7106(3)	1.0261(5)	1.0815(3)	0.050(2)	0.096(4)	0.044(2)	0.002(2)	0.008(2)	0.026(3)
C(24)	2*i*	0.6490(3)	1.0833(4)	1.0220(3)	0.043(2)	0.062(3)	0.047(2)	0.002(2)	0.017(2)	0.012(2)
C(25)	2*i*	0.6192(3)	1.0187(3)	0.9151(3)	0.033(2)	0.049(2)	0.049(2)	0.003(2)	0.013(2)	0.018(2)
C(26)	2*i*	0.6145(4)	1.2018(4)	1.0637(3)	0.065(3)	0.065(3)	0.065(3)	0.002(2)	0.025(2)	0.001(3)
C(27)	2*i*	0.5563(4)	1.2515(4)	1.0017(4)	0.066(3)	0.049(3)	0.095(4)	0.015(2)	0.035(2)	0.009(3)
C(28)	2*i*	0.5265(3)	1.1877(4)	0.8924(3)	0.049(2)	0.048(3)	0.072(3)	0.015(2)	0.026(2)	0.018(2)
C(29)	2*i*	0.4648(3)	1.2331(4)	0.8232(4)	0.061(2)	0.048(3)	0.107(4)	0.026(2)	0.038(2)	0.028(3)
C(30)	2*i*	0.4401(3)	1.1656(4)	0.7203(4)	0.061(2)	0.057(3)	0.086(3)	0.026(2)	0.024(2)	0.040(3)
C(31)	2*i*	0.4746(3)	1.0515(4)	0.6827(3)	0.047(2)	0.054(3)	0.067(3)	0.018(2)	0.014(2)	0.029(2)
C(32)	2*i*	0.5574(3)	1.0711(3)	0.8488(3)	0.036(2)	0.039(2)	0.054(2)	0.004(2)	0.016(2)	0.017(2)
C(33)	2*i*	0.0936(3)	0.6543(3)	0.9429(3)	0.048(2)	0.042(2)	0.045(2)	0.010(2)	0.012(2)	0.018(2)
C(34)	2*i*	0.0975(3)	0.6864(4)	1.0465(3)	0.054(2)	0.047(2)	0.049(2)	0.014(2)	0.011(2)	0.022(2)
C(35)	2*i*	0.1598(4)	0.6254(4)	1.0978(3)	0.090(3)	0.079(3)	0.054(3)	0.034(3)	0.017(2)	0.039(3)
C(36)	2*i*	0.2192(4)	0.5334(4)	1.0509(4)	0.073(3)	0.071(3)	0.087(3)	0.032(2)	0.018(2)	0.051(3)
C(37)	2*i*	0.2151(3)	0.5010(4)	0.9499(4)	0.065(2)	0.050(3)	0.098(3)	0.035(2)	0.038(2)	0.039(3)
C(38)	2*i*	0.1519(3)	0.5607(4)	0.8959(3)	0.065(2)	0.055(3)	0.054(2)	0.020(2)	0.026(2)	0.023(2)
C(39)	2*i*	0.0256(3)	0.7220(3)	0.8869(3)	0.044(2)	0.048(2)	0.038(2)	0.013(2)	0.007(2)	0.012(2)
C(40)	2*i*	0.0348(4)	0.7867(4)	1.1034(3)	0.066(2)	0.052(3)	0.038(2)	0.015(2)	0.014(2)	0.015(2)
F(1)	2*i*	1.1694(2)	1.0387(2)	0.6806(2)	0.042(1)	0.090(2)	0.097(2)	0.019(1)	0.019(1)	0.055(2)
F(2)	2*i*	1.1645(2)	1.2477(2)	0.6462(2)	0.064(1)	0.079(2)	0.096(2)	–0.003(1)	0.029(1)	0.048(2)
F(3)	2*i*	0.9494(2)	1.3114(2)	0.5876(2)	0.088(2)	0.051(2)	0.082(2)	0.016(1)	0.030(1)	0.039(1)
F(4)	2*i*	0.7423(2)	1.1627(2)	0.5557(2)	0.061(1)	0.052(1)	0.086(2)	0.028(1)	0.010(1)	0.036(1)
F(5)	2*i*	0.1669(3)	0.6560(3)	1.1981(2)	0.173(3)	0.125(3)	0.064(2)	0.077(2)	0.029(2)	0.057(2)
F(6)	2*i*	0.2846(3)	0.4790(3)	1.1040(2)	0.132(2)	0.116(3)	0.128(2)	0.072(2)	0.034(2)	0.084(2)
F(7)	2*i*	0.2753(2)	0.4148(3)	0.9033(2)	0.105(2)	0.085(2)	0.146(2)	0.065(2)	0.071(2)	0.064(2)
F(8)	2*i*	0.1499(2)	0.5249(3)	0.7965(2)	0.131(2)	0.086(2)	0.072(2)	0.059(2)	0.055(2)	0.034(2)
N(1)	2*i*	0.6415(2)	0.6819(3)	0.6739(2)	0.039(1)	0.035(2)	0.045(2)	0.010(1)	0.008(1)	0.018(1)
N(2)	2*i*	0.4196(2)	0.7292(3)	0.6535(2)	0.044(2)	0.045(2)	0.039(2)	0.013(1)	0.012(1)	0.018(1)
N(3)	2*i*	0.6455(2)	0.9043(3)	0.8689(2)	0.041(2)	0.051(2)	0.050(2)	0.013(1)	0.013(1)	0.023(2)
N(4)	2*i*	0.5319(2)	1.0042(3)	0.7452(2)	0.042(2)	0.048(2)	0.051(2)	0.013(1)	0.011(1)	0.027(2)
Ni(1)	2*i*	0.58955(3)	0.84418(4)	0.70264(3)	0.0352(2)	0.0343(3)	0.0437(3)	0.0090(2)	0.0104(2)	0.0151(2)
O(1)	2*i*	0.7342(2)	0.9134(2)	0.6750(2)	0.041(1)	0.050(2)	0.063(2)	0.013(1)	0.014(1)	0.031(1)
O(2)	2*i*	0.6365(2)	0.9286(2)	0.5319(2)	0.048(1)	0.057(2)	0.058(2)	0.007(1)	–0.003(1)	0.019(1)
O(3)	2*i*	0.9996(2)	0.9082(2)	0.7647(2)	0.061(1)	0.048(2)	0.045(2)	0.015(1)	0.005(1)	0.023(1)
O(4)	2*i*	0.9481(2)	0.7777(2)	0.5986(2)	0.065(2)	0.044(2)	0.057(2)	0.024(1)	0.019(1)	0.017(1)
O(5)	2*i*	0.0750(2)	0.7450(3)	0.8220(2)	0.059(2)	0.071(2)	0.071(2)	0.029(1)	0.025(1)	0.046(2)
O(6)	2*i*	–0.0678(2)	0.7512(3)	0.9048(2)	0.061(2)	0.126(3)	0.067(2)	0.050(2)	0.025(1)	0.051(2)
O(7)	2*i*	0.0878(2)	0.8966(3)	1.1176(2)	0.078(2)	0.043(2)	0.060(2)	0.013(1)	0.023(1)	0.014(2)
O(8)	2*i*	–0.0502(3)	0.7627(3)	1.1341(2)	0.094(2)	0.064(2)	0.074(2)	0.020(2)	0.045(2)	0.024(2)

References

1. Furukawa, H.; Yaghi, O. M.: Storage of Hydrogen, Methane, and Carbon Dioxide in Highly Porous Covalent Organic Frameworks for Clean Energy Applications. J. Am. Chem. Soc. **131** (2009) 8875-8883.
2. Han, M. L.; Ling, X. L.; Wang, J. G.; Mi, L. W.: Crystal Structure of Cis-diaquabis(1,10-phenanthroline)Zinc(II)bis(3-amino-4-chlorobenzensulphonate)Dihydrate, $[(Zn(H_2O)_2(C_{12}H_8N_2)_2](H_2NC_6H_3ClSO_3)_2{\cdot}2H_2O$. Z. Kristallogr. NCS **225** (2010) 410-412.
3. Ma, L. F.; Wang, L. Y.; Wang, Y. Y.; Batten, S. R.; Wang, J. G.: Self-Assembly of a Series of Cobalt(II) Coordination Polymers Constructed from H_2tbip and Dipyridyl-Based Ligands. Inorg. Chem. **48** (2009) 915-924.
4. Liu, G. X.; Huang, Y. Q.; Chu, Q.; Okamura, T.; Sun, W. Y.: Effect of N-Donor Ancillary Ligands on Supramolecular Architectures of a Series of Zinc(II) and Cadmium(II) Complexes with Flexible Tricarboxylate. Cryst. Growth Des. **8** (2008) 3233-3245.
5. Braverman, M. A.; LaDuca, R. L.: Luminescent Two- and Three-Dimensional Zinc Coordination Polymers Containing Isomers of Phenylenediacetate and a Kinked Tethering Organodiimine. Cryst. Growth Des. **7** (2007) 2343-2351.
6. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.