Crystal structure of catena-poly[diaqua-3-(4-fluoro-phenyl)-acrylate-κ^2*O,O′*-bis(κ^2*O,O′*)-1,2-di(4-pyridyl)ethylene-κ^2*N:N′*nickel(II)] 3-(4-fluoro-phenyl)-acrylate, $C_{30}H_{26}F_2N_2NiO_6$

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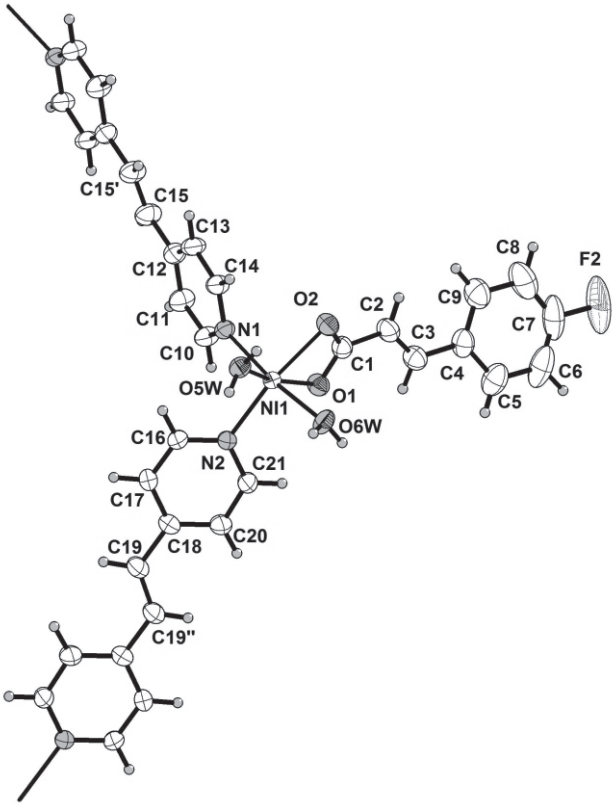

Abstract

$C_{30}H_{26}F_2N_2NiO_6$, monoclinic, $P2_1/c$ (no. 14), a = 16.6375(6) Å, b = 6.7702(3) Å, c = 25.352(1) Å, β = 105.390(1)°, V = 2753.2 Å^3, Z = 4, $R_{gt}(F)$ = 0.0325, $wR_{ref}(F^2)$ = 0.0781, T = 296 K.

Source of material

The mixture of 3-(4-fluoro-phenyl)-acrylic acid (0.1 mmol, 16.6 mg), 1,2-di(4-pyridyl)ethylene (0.1 mmol, 18.4 mg), $NiSO_4{\cdot}6H_2O$ (0.1 mmol, 26.2 mg), NaOH (0.1 mmol, 4.0 mg), and H_2O (7 ml) was sealed in a 23 ml Teflon-lined autoclave, heated to 393K for 3 days, and then slowly cooled down to room temperature for crystallization. Green needle-shaped crystals of the title compound were obtained.

Discussion

Weak intermolecular interactions such as hydrogen bonding, π-π stacking interactions, hydrophobic interaction, and van derWaals force play an important role in the self-assembly of molecules in crystal lattices [1–5]. However the research of halogen–hydrogen bonding and halogen–halogen bonding in organic supramolecular systems has attracted intensive attention in recent years [6–9]. Halogen–halogen contacts are described as the interhalogen distance less than the sum of van der Waals radii (r_{vdw}) of the corresponding halogen atoms [10]. As for the halogen–hydrogen bonding and halogen–halogen bonding, the C–F···F–C interactions have been less attractive to researchers. But topological analysis and experimental charge density have demonstrated that F···F intermolecular interactions are significantly detectable and reinforce the crystal packing [11–12]. In order to study the F···F interactions and build novel coordination compounds, 3-(4-fluoro-phenyl)-acrylic acid is used for these reasons: (1) 3-(4-fluoro-phenyl)-acrylic acid has the coordination modes of carboxylato group; (2) The F···F interactions may be observed and contribute to construct the novel coordination compound. The asymmetric unit contains one Ni(II) cation, one coordination 3-(4-fluoro-phenyl)-acrylic acid ligand, one free deprotonated 3-(4-fluoro-phenyl)-acrylic acid, two coordination water molecules, two half 1,2-di(4-pyridyl)ethylene (*bpe*) molecules. The Ni(II) ion is six-coordinated by two nitrogen atoms from two *bpe* ligands, two oxygen atoms from one deprotonated carboxylate ligand and two oxygen atoms from two water molecules with a distorted octahedral coordination geometry. All the Ni–O bond lengths are between 2.0483(13) and 2.1260(14) Å. The Ni–N bond lengths are 2.0614(16) and 2.0788(16) Å. Two adjacent Ni(II) ions are connected by one *bpe* ligand to develop one-dimensional zigzag chain along the *b*-axis. There are uncoordinated 4-(3-fluoro-phenyl)-acrylic acid anions in the compound. The anions acting as the acceptor atom groups play an important role in forming the H-bonds. Meanwhile the coordinated water molecules are the donors. There are various O–H···O hydrogen bonds. Through these hydrogen bonds the 1D chains are assembled to give rise to a 3D supramolecular structure.

Table 1. Data collection and handling.

Crystal:	green needles, size 0.18×0.23×0.29 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	7.65 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{max}$:	50.9°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	17459, 5073
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4269
$N(param)_{refined}$:	370
Programs:	SHELX [13]

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

Atom	Site	x	y	z	U_{iso}
H(2)	4*e*	0.1389	0.1002	0.2598	0.052
H(3)	4*e*	0.1853	0.4651	0.2328	0.053
H(5)	4*e*	0.1641	0.7315	0.2926	0.075
H(6)	4*e*	0.0928	0.8654	0.3518	0.100
H(8)	4*e*	–0.0340	0.3633	0.3472	0.097
H(9)	4*e*	0.0380	0.2270	0.2892	0.074
H(10)	4*e*	0.1966	0.0742	0.0586	0.045
H(11)	4*e*	0.0804	–0.0600	0.0009	0.049
H(13)	4*e*	0.1242	–0.5563	0.0896	0.048
H(14)	4*e*	0.2398	–0.4090	0.1447	0.042
H(15)	4*e*	–0.0226	–0.3214	–0.0233	0.053
H(16)	4*e*	0.3699	–0.1048	0.0604	0.041
H(17)	4*e*	0.4230	0.0527	–0.0025	0.044
H(19)	4*e*	0.4778	0.3477	–0.0362	0.046
H(20)	4*e*	0.4522	0.5367	0.0909	0.051
H(21)	4*e*	0.3929	0.3699	0.1492	0.048
H(23)	4*e*	0.3526	0.7467	0.8000	0.057
H(24)	4*e*	0.3786	0.9355	0.8974	0.057
H(26)	4*e*	0.3248	0.4547	0.8406	0.063
H(27)	4*e*	0.2573	0.2104	0.8766	0.075
H(29)	4*e*	0.2289	0.5835	0.9920	0.078
H(30)	4*e*	0.2933	0.8288	0.9553	0.066
H(1W)	4*e*	0.4465	–0.2465	0.1704	0.049
H(2W)	4*e*	0.4051	–0.3333	0.2028	0.049
H(3W)	4*e*	0.4151	0.2128	0.2412	0.060
H(4W)	4*e*	0.4609	0.0415	0.2374	0.060

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)	4*e*	0.32391(2)	–0.01734(3)	0.16701(1)	0.0261(1)	0.0240(1)	0.0257(1)	–0.0025(1)	0.0077(1)	–0.0015(1)
F(1)	4*e*	0.2001(1)	0.2329(3)	0.95905(9)	0.114(2)	0.093(2)	0.112(2)	–0.024(1)	0.047(1)	0.042(1)
F(2)	4*e*	–0.0191(2)	0.7049(4)	0.38694(9)	0.131(2)	0.210(3)	0.094(2)	0.069(2)	0.053(1)	–0.051(2)
N(1)	4*e*	0.2297(1)	–0.1527(2)	0.10740(7)	0.0290(9)	0.0310(9)	0.0296(9)	–0.0056(7)	0.0051(7)	–0.0016(8)
N(2)	4*e*	0.3757(1)	0.1167(2)	0.11077(7)	0.0325(9)	0.0268(9)	0.0309(9)	–0.0023(7)	0.0122(7)	–0.0010(7)
O(1)	4*e*	0.24663(9)	0.2125(2)	0.17734(6)	0.0401(9)	0.0333(8)	0.0409(9)	0.0042(7)	0.0166(7)	0.0024(7)
O(2)	4*e*	0.24712(9)	–0.0692(2)	0.22056(6)	0.0407(9)	0.0371(9)	0.0387(9)	0.0023(7)	0.0165(7)	0.0033(7)
O(3)	4*e*	0.4247(1)	1.0124(2)	0.76064(7)	0.0445(9)	0.0407(9)	0.044(1)	–0.0021(7)	0.0145(7)	–0.0029(7)
O(4)	4*e*	0.4550(1)	1.1402(3)	0.84379(7)	0.046(1)	0.056(1)	0.049(1)	–0.0058(8)	0.0172(8)	–0.0094(9)
C(1)	4*e*	0.2228(1)	0.1069(3)	0.21182(9)	0.030(1)	0.041(1)	0.034(1)	0.000(1)	0.0092(9)	–0.004(1)
C(2)	4*e*	0.1683(1)	0.1884(4)	0.2438(1)	0.041(1)	0.046(1)	0.049(1)	0.002(1)	0.022(1)	–0.002(1)
C(3)	4*e*	0.1589(1)	0.3779(4)	0.2511(1)	0.040(1)	0.048(2)	0.048(2)	0.002(1)	0.017(1)	0.000(1)
C(4)	4*e*	0.1099(2)	0.4632(4)	0.2860(1)	0.040(1)	0.054(2)	0.046(1)	0.012(1)	0.011(1)	–0.007(1)
C(5)	4*e*	0.1251(2)	0.6559(4)	0.3040(1)	0.056(2)	0.060(2)	0.067(2)	0.012(1)	0.007(1)	–0.018(2)
C(6)	4*e*	0.0821(2)	0.7370(6)	0.3388(1)	0.079(2)	0.085(2)	0.074(2)	0.031(2)	–0.001(2)	–0.039(2)
C(7)	4*e*	0.0240(2)	0.6242(7)	0.3535(1)	0.071(2)	0.128(3)	0.054(2)	0.043(2)	0.016(2)	–0.027(2)
C(8)	4*e*	0.0062(2)	0.4361(6)	0.3363(1)	0.063(2)	0.117(3)	0.074(2)	0.021(2)	0.037(2)	0.000(2)
C(9)	4*e*	0.0496(2)	0.3555(5)	0.3019(1)	0.057(2)	0.069(2)	0.068(2)	0.009(1)	0.029(1)	–0.008(2)
C(10)	4*e*	0.1818(1)	–0.0543(3)	0.06492(9)	0.037(1)	0.031(1)	0.040(1)	–0.0062(9)	0.004(1)	0.003(1)
C(11)	4*e*	0.1117(1)	–0.1341(3)	0.03013(9)	0.037(1)	0.041(1)	0.039(1)	–0.003(1)	–0.002(1)	0.005(1)
C(12)	4*e*	0.0874(1)	–0.3247(3)	0.03847(9)	0.032(1)	0.043(1)	0.035(1)	–0.007(1)	0.0054(9)	–0.003(1)
C(13)	4*e*	0.1375(1)	–0.4274(3)	0.08246(9)	0.041(1)	0.035(1)	0.042(1)	–0.014(1)	0.009(1)	0.001(1)
C(14)	4*e*	0.2070(1)	–0.3378(3)	0.11543(9)	0.034(1)	0.035(1)	0.034(1)	–0.0068(9)	0.0048(9)	0.004(1)
C(15)	4*e*	0.0117(1)	–0.4082(4)	0.0011(1)	0.037(1)	0.049(1)	0.041(1)	–0.011(1)	–0.001(1)	0.003(1)
C(16)	4*e*	0.3857(1)	0.0268(3)	0.06626(9)	0.043(1)	0.026(1)	0.035(1)	–0.0068(9)	0.0138(9)	–0.0035(9)
C(17)	4*e*	0.4185(1)	0.1204(3)	0.02855(9)	0.044(1)	0.040(1)	0.031(1)	–0.006(1)	0.017(1)	–0.005(1)
C(18)	4*e*	0.4449(1)	0.3151(3)	0.03642(8)	0.033(1)	0.035(1)	0.034(1)	–0.0014(9)	0.0133(9)	0.0044(9)
C(19)	4*e*	0.4813(1)	0.4133(3)	–0.00338(9)	0.044(1)	0.039(1)	0.035(1)	–0.003(1)	0.017(1)	0.005(1)
C(20)	4*e*	0.4351(2)	0.4066(3)	0.0833(1)	0.060(2)	0.027(1)	0.048(1)	–0.009(1)	0.027(1)	–0.002(1)
C(21)	4*e*	0.4000(2)	0.3046(3)	0.11850(9)	0.059(2)	0.027(1)	0.041(1)	–0.007(1)	0.026(1)	–0.006(1)
C(22)	4*e*	0.4208(1)	1.0128(3)	0.8101(1)	0.028(1)	0.035(1)	0.050(1)	0.0050(9)	0.009(1)	0.006(1)
C(23)	4*e*	0.3731(2)	0.8451(4)	0.8257(1)	0.046(1)	0.048(2)	0.047(2)	–0.003(1)	0.010(1)	–0.002(1)
C(24)	4*e*	0.3594(1)	0.8317(4)	0.8733(1)	0.041(1)	0.053(2)	0.048(2)	0.001(1)	0.010(1)	–0.001(1)
C(25)	4*e*	0.3165(1)	0.6690(4)	0.89386(9)	0.037(1)	0.048(1)	0.041(1)	0.001(1)	0.008(1)	0.010(1)
C(26)	4*e*	0.3052(2)	0.4821(4)	0.8710(1)	0.059(2)	0.061(2)	0.040(1)	0.003(1)	0.017(1)	0.005(1)
C(27)	4*e*	0.2654(2)	0.3353(4)	0.8924(1)	0.077(2)	0.046(2)	0.063(2)	–0.003(1)	0.015(2)	0.005(1)
C(28)	4*e*	0.2382(2)	0.3782(5)	0.9373(1)	0.060(2)	0.065(2)	0.061(2)	–0.008(1)	0.019(1)	0.025(2)
C(29)	4*e*	0.2480(2)	0.5587(5)	0.9614(1)	0.066(2)	0.084(2)	0.054(2)	0.001(2)	0.031(2)	0.009(2)
C(30)	4*e*	0.2868(2)	0.7040(4)	0.9394(1)	0.057(2)	0.058(2)	0.052(2)	–0.004(1)	0.019(1)	–0.005(1)
O(5W)	4*e*	0.39762(8)	–0.2634(2)	0.17403(6)	0.0329(8)	0.0274(7)	0.0360(8)	0.0010(6)	0.0074(6)	–0.0010(6)
O(6W)	4*e*	0.41258(9)	0.0903(2)	0.23359(6)	0.0333(8)	0.0404(9)	0.0419(9)	–0.0013(7)	0.0038(7)	–0.0148(7)

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