Crystal structure of bis(4,4,5,5-tetramethyl-2-(thiophenyl-2-yl)-imidazoline-1-oxyl-3-oxide)-bis(hexafluoroacetylacetonato)nickel(II), $\text{Ni}(\text{C}_{11}\text{H}_{15}\text{N}_2\text{O}_2\text{S})_2[\text{C}_3\text{HO}_2(\text{CF}_3)_2]_2$

Xiao-Song Wang^I, Xia Gao^I, Xiao-Lei Hou^{*II} and Xiao-Qiang Wang^{*II}

^I Henan Institute of Education, Department of Chemistry, Zhengzhou 450046, P. R. China

^{II} Henan Provincial Nonferrous Metals Geological Exploration, Total Institute, Zhengzhou 450052, P. R. China

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Abstract

$\text{C}_{32}\text{H}_{32}\text{F}_{12}\text{N}_4\text{NiO}_8\text{S}_2$, monoclinic, $P121/c1$ (no. 14), $a = 12.1886(9)$ Å, $b = 15.137(1)$ Å, $c = 22.367(2)$ Å, $\beta = 101.895(1)^\circ$, $V = 4038.1$ Å³, $Z = 4$, $R_g(F) = 0.073$, $wR_{\text{ref}}(F^2) = 0.239$, $T = 291$ K.

Source of material

NIT2-thp was prepared according to a literature method [1]. The title complex was synthesized as follows: a mixture of $\text{Ni}(\text{hfac})_2 \cdot 2\text{H}_2\text{O}$ (77 mg, 0.15 mmol) and NIT2-thp (36 mg, 0.15 mmol) was dissolved with heating to 75 °C in heptane/acetone (10 ml / 10 ml) mixture, the resulting solution was stirred for 1 h and then cooled to room temperature. After filtration, the filtrate was allowed to stand at room temperature for 72 h to produce well-shaped dark-green crystals. Elemental analysis — found: C, 40.32 %; H, 3.36 %; N, 5.91 %; calculated for $\text{C}_{32}\text{H}_{32}\text{F}_{12}\text{N}_4\text{NiO}_8\text{S}_2$: C, 40.39 %; H, 3.39 %; N, 5.89 %.

Experimental details

The CF_3 -groups were handled according to the disorder in the crystal structure, which caused larger residual values.

Discussion

During the past decade, nitronyl nitroxide radicals (NITR) have been widely employed as molecular units in the design and synthesis of molecular magnetic materials [2–4]. The nitroxide derivatives can be bound to the metal through the oxygen atom of O–N groups, resulting in a variety of transition metal-radical complexes have [5–9]. Although the family of nitronyl nitroxides has

been widely studied for a long time, the NITR (R = methyl, ethyl, phenyl, pyridyl etc.), thiophenyl substituents have rarely been reported so far. Therefore, in order to extend our knowledge of the extremely rich chemistry of such systems, we are devoted to the explorations of new building block for molecule-based magnetic materials. In this paper, we have synthesized a novel nickel(II) complex involving thiophene-substituted nitronyl nitroxide radical (NIT2-thp).

In the title crystal structure the nickel atom is hexa-coordinated in a distorted octahedral environment. The apical positions are occupied by two oxygen atoms (O1 and O3) of the nitronyl nitroxide radicals, and the equatorial positions are occupied by four oxygen atoms (O5, O6, O7, O8) of two hfac molecules. The Ni1—O1 and Ni1—O3 bond length (2.105(3) Å and 2.099(3) Å, respectively) are larger than the Ni1—O5, Ni1—O6, Ni1—O7 and Ni1—O8 bond lengths (2.021(3) Å, 2.007(3) Å, 2.011(3) Å and 2.023(3) Å, respectively). The coordinated N—O bond lengths of N1—O1 and N3—O3 are 1.284(5) Å and 1.295(5) Å, respectively, which is similar to the values observed in nitroxide (NIT2-thp, the N—O bond lengths are 1.285(5) Å and 1.282(5) Å), which indicates the nitroxide oxygen atoms are only very loosely bonded to the central Ni(II) ion in the apical direction. The uncoordinated N—O bond lengths of N2—O2 and N4—O4 are 1.267(6) Å and 1.285(6) Å, respectively. Bond distances and angles in the hfac molecules are in the normal range for this kind of compound [10–12]. The dihedral angle between the thiophene ring (C1, C2, C3, C4, S1) and the mean plane of the nitroxide radical (O2, N2, C5, N1, O1) is 15.9°. From the unit cell packing, two adjacent molecules of the title complex are linked by pairs of inversion-related O···S weak intermolecular interactions involving O4B···S2A and S2B···O4A, resulting in a dimeric structure, the distance of O···S is 3.118 Å.

Table 1. Data collection and handling.

Crystal:	dark-green block, size 0.29 × 0.34 × 0.45 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	6.91 cm ^{−1}
Diffractionmeter, scan mode:	Bruker SMART CCD, ϕ/ω
$2\theta_{\text{max}}$:	51°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	21447, 7472
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 5698
$N(\text{param})_{\text{refined}}$:	580
Programs:	SHELXS-97 [13], SHELXL-97 [14], SHELXTL [15]

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

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U _{iso}
H(1)	4e	-0.1550	-0.0072	0.9126	0.127
H(2)	4e	-0.1504	-0.0024	0.8110	0.128
H(3)	4e	-0.0096	0.1085	0.7798	0.064
H(8A)	4e	0.4098	0.2463	0.8734	0.089
H(8B)	4e	0.4123	0.3498	0.8718	0.089
H(8C)	4e	0.3610	0.2967	0.8125	0.089
H(9A)	4e	0.1798	0.3671	0.7953	0.104
H(9B)	4e	0.2226	0.4313	0.8502	0.104
H(9C)	4e	0.1129	0.3761	0.8478	0.104
H(10A)	4e	0.3509	0.2427	1.0209	0.135
H(10B)	4e	0.4270	0.2782	0.9776	0.135
H(10C)	4e	0.3607	0.1895	0.9621	0.135
H(11A)	4e	0.1820	0.4191	0.9470	0.134
H(11B)	4e	0.3119	0.4229	0.9721	0.134
H(11C)	4e	0.2336	0.3802	1.0116	0.134
H(12)	4e	0.6821	0.1385	0.6021	0.086

Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(13)	4e	0.6991	0.1395	0.7062	0.095
H(14)	4e	0.5452	0.0513	0.7491	0.024
H(19A)	4e	0.1454	-0.0263	0.5833	0.115
H(19B)	4e	0.0769	-0.1129	0.5636	0.115
H(19C)	4e	0.1504	-0.0716	0.5209	0.115
H(20A)	4e	0.2486	-0.2176	0.5212	0.127
H(20B)	4e	0.1793	-0.2581	0.5663	0.127
H(20C)	4e	0.3107	-0.2590	0.5832	0.127
H(21A)	4e	0.1695	-0.1368	0.7324	0.100
H(21B)	4e	0.1064	-0.1853	0.6733	0.100
H(21C)	4e	0.1135	-0.0819	0.6747	0.100
H(22A)	4e	0.4032	-0.2166	0.6784	0.106
H(22B)	4e	0.2964	-0.2705	0.6847	0.106
H(22C)	4e	0.3541	-0.2076	0.7376	0.106
H(25)	4e	0.4010	-0.0756	0.9221	0.067
H(30)	4e	0.1407	0.2455	0.6168	0.068

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	Occ.	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
F(1)	4e	0.670(6)	0.2510(6)	-0.1931(4)	0.9041(4)	0.105(4)	0.059(3)	0.151(5)	0.003(3)	0.044(4)	0.046(4)
F(2)	4e	0.670	0.1003(6)	-0.1437(6)	0.8481(3)	0.093(4)	0.102(5)	0.119(6)	-0.042(3)	0.016(4)	0.030(4)
F(3)	4e	0.670	0.1607(8)	-0.0942(6)	0.9363(4)	0.138(6)	0.134(6)	0.132(5)	-0.020(4)	0.081(5)	0.029(4)
F(4)	4e	0.61(3)	0.595(1)	-0.0308(6)	0.9447(6)	0.075(4)	0.073(3)	0.071(4)	0.026(3)	-0.008(3)	0.009(3)
F(5)	4e	0.61	0.557(1)	0.1071(7)	0.9532(6)	0.063(4)	0.064(3)	0.060(5)	-0.013(3)	0.001(3)	0.000(3)
F(6)	4e	0.61	0.628(1)	0.063(1)	0.8780(5)	0.050(2)	0.094(6)	0.095(3)	0.000(4)	0.015(2)	0.004(4)
F(7)	4e	0.69(1)	-0.0885(8)	0.1346(9)	0.6643(4)	0.057(3)	0.131(6)	0.118(4)	-0.003(4)	0.013(3)	0.015(4)
F(8)	4e	0.69	-0.0341(7)	0.0562(5)	0.5960(6)	0.096(4)	0.079(3)	0.099(5)	-0.002(3)	-0.040(4)	-0.021(3)
F(9)	4e	0.69	-0.0442(6)	0.1969(5)	0.5870(3)	0.069(3)	0.063(3)	0.074(3)	0.010(3)	-0.020(3)	0.001(3)
F(10)	4e	0.75(1)	0.4340(6)	0.2709(5)	0.6395(4)	0.090(4)	0.106(4)	0.100(5)	-0.014(3)	0.043(3)	0.027(3)
F(11)	4e	0.75	0.2850(5)	0.3476(4)	0.6181(3)	0.093(4)	0.065(3)	0.088(3)	0.001(3)	0.025(3)	0.034(3)
F(12)	4e	0.75	0.3883(9)	0.3490(5)	0.7086(3)	0.147(6)	0.091(4)	0.081(4)	-0.061(4)	0.011(3)	-0.001(3)
F(1')	4e	0.33	0.210(1)	-0.1934(7)	0.8526(6)	0.105(4)	0.059(3)	0.151(5)	0.003(3)	0.044(4)	0.046(4)
F(2')	4e	0.33	0.0861(7)	-0.112(1)	0.8769(7)	0.093(4)	0.102(5)	0.119(6)	-0.042(3)	0.016(4)	0.030(4)
F(3')	4e	0.33	0.231(1)	-0.147(1)	0.9423(4)	0.138(6)	0.134(6)	0.132(5)	-0.020(4)	0.081(5)	0.029(4)
F(4')	4e	0.39	0.582(2)	-0.019(1)	0.9570(8)	0.075(4)	0.073(3)	0.071(4)	0.026(3)	-0.008(3)	0.009(3)
F(5')	4e	0.39	0.573(2)	0.1212(7)	0.9381(9)	0.063(4)	0.064(3)	0.060(5)	-0.013(3)	0.001(3)	0.000(3)
F(6')	4e	0.39	0.623(2)	0.031(2)	0.8737(8)	0.050(2)	0.094(6)	0.095(3)	0.000(4)	0.015(2)	0.004(4)
F(7')	4e	0.31(2)	-0.072(2)	0.172(1)	0.6655(8)	0.057(3)	0.131(6)	0.118(4)	-0.003(4)	0.013(3)	0.015(4)
F(8')	4e	0.31	-0.046(2)	0.171(1)	0.5742(5)	0.096(4)	0.079(3)	0.099(5)	-0.002(3)	-0.040(4)	-0.021(3)
F(9')	4e	0.31	-0.050(1)	0.0489(5)	0.6234(9)	0.069(3)	0.063(3)	0.074(3)	0.010(3)	-0.020(3)	0.001(3)
F(10')	4e	0.25(1)	0.314(2)	0.330(2)	0.6049(4)	0.090(4)	0.106(4)	0.100(5)	-0.014(3)	0.043(3)	0.027(3)
F(11')	4e	0.25	0.322(2)	0.3647(8)	0.6974(7)	0.093(4)	0.065(3)	0.088(3)	0.001(3)	0.025(3)	0.034(3)
F(12')	4e	0.25	0.4558(8)	0.293(1)	0.673(1)	0.147(6)	0.091(4)	0.081(4)	-0.061(4)	0.011(3)	-0.001(3)
Ni(1)	4e		0.27005(5)	0.08209(4)	0.76829(3)	0.0386(4)	0.0380(4)	0.0319(4)	-0.0047(2)	0.0064(2)	0.0013(2)
S(1)	4e		-0.0170(2)	0.0928(2)	0.9422(1)	0.094(2)	0.091(2)	0.154(2)	-0.006(1)	0.069(2)	0.021(2)
S(2)	4e		0.5322(2)	0.0494(2)	0.5820(1)	0.086(1)	0.088(1)	0.111(2)	-0.001(1)	0.036(1)	0.019(1)
O(1)	4e		0.1813(3)	0.1865(2)	0.7986(2)	0.046(2)	0.043(2)	0.042(2)	0.000(2)	0.007(2)	-0.006(2)
O(2)	4e		0.1120(5)	0.2400(4)	0.9895(2)	0.122(5)	0.126(5)	0.076(3)	-0.029(4)	0.068(3)	-0.028(3)
O(3)	4e		0.3518(3)	-0.0243(2)	0.7360(2)	0.047(2)	0.049(2)	0.038(2)	0.004(2)	0.006(2)	-0.006(2)
O(4)	4e		0.3814(4)	-0.0839(3)	0.5378(2)	0.092(3)	0.079(3)	0.043(2)	-0.013(2)	0.025(2)	-0.014(2)
O(5)	4e		0.2038(3)	-0.0072(2)	0.8180(2)	0.046(2)	0.045(2)	0.049(2)	-0.004(2)	0.015(2)	0.006(2)
O(6)	4e		0.4081(3)	0.0877(2)	0.8349(2)	0.046(2)	0.042(2)	0.040(2)	-0.005(2)	0.003(2)	0.002(1)
O(7)	4e		0.1339(3)	0.0774(2)	0.7000(2)	0.047(2)	0.042(2)	0.044(2)	-0.005(2)	-0.001(2)	-0.001(2)
O(8)	4e		0.3373(3)	0.1747(2)	0.7214(2)	0.047(2)	0.048(2)	0.041(2)	-0.002(2)	0.011(2)	0.008(2)
N(1)	4e		0.1911(3)	0.2175(3)	0.8529(2)	0.042(2)	0.039(2)	0.041(2)	-0.002(2)	0.013(2)	-0.002(2)
N(2)	4e		0.1569(5)	0.2442(3)	0.9433(2)	0.074(3)	0.062(3)	0.059(3)	-0.009(3)	0.033(3)	-0.011(2)
N(3)	4e		0.3309(3)	-0.0572(3)	0.6814(2)	0.042(2)	0.039(2)	0.039(2)	-0.001(2)	0.009(2)	-0.004(2)
N(4)	4e		0.3460(4)	-0.0864(3)	0.5881(2)	0.058(3)	0.046(2)	0.039(2)	-0.002(2)	0.011(2)	-0.006(2)
C(1)	4e		-0.1055(7)	0.0294(6)	0.8977(6)	0.062(5)	0.086(6)	0.18(1)	-0.018(4)	0.058(6)	0.012(6)
C(2)	4e		-0.1030(8)	0.0329(6)	0.8392(6)	0.077(5)	0.075(5)	0.17(1)	-0.027(4)	0.034(6)	-0.015(6)
C(3)	4e		-0.0214(4)	0.0955(3)	0.8186(3)	0.029(2)	0.039(3)	0.099(5)	-0.016(2)	0.029(3)	-0.016(3)
C(4)	4e		0.0363(5)	0.1311(4)	0.8819(3)	0.051(3)	0.047(3)	0.086(4)	0.000(2)	0.033(3)	0.000(3)
C(5)	4e		0.1265(4)	0.1946(3)	0.8919(2)	0.050(3)	0.045(3)	0.054(3)	-0.002(2)	0.021(2)	-0.003(2)
C(6)	4e		0.2555(4)	0.3011(3)	0.8732(2)	0.049(3)	0.038(3)	0.049(3)	-0.007(2)	0.013(2)	-0.007(2)
C(7)	4e		0.2581(5)	0.2994(4)	0.9421(3)	0.062(3)	0.061(3)	0.050(3)	-0.010(3)	0.018(3)	-0.013(3)
C(8)	4e		0.3703(5)	0.2982(4)	0.8561(3)	0.057(3)	0.059(3)	0.067(4)	-0.020(3)	0.023(3)	-0.016(3)

Table 3. Continued.

Atom	Site	Occ.	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(9)	4e		0.1863(6)	0.3758(4)	0.8384(3)	0.078(4)	0.047(3)	0.082(4)	−0.003(3)	0.011(3)	0.001(3)
C(10)	4e		0.3587(7)	0.2475(6)	0.9792(3)	0.088(5)	0.128(7)	0.050(4)	−0.004(5)	0.003(4)	0.002(4)
C(11)	4e		0.2452(7)	0.3886(5)	0.9709(4)	0.102(6)	0.083(5)	0.089(5)	−0.018(4)	0.034(5)	−0.043(4)
C(12)	4e		0.6325(6)	0.1074(5)	0.6208(4)	0.069(4)	0.065(4)	0.093(5)	−0.017(3)	0.046(4)	−0.004(4)
C(13)	4e		0.6412(6)	0.1087(5)	0.6813(4)	0.063(4)	0.065(4)	0.113(6)	−0.018(3)	0.023(4)	−0.023(4)
C(14)	4e		0.5540(3)	0.0585(2)	0.7090(2)	0.012(2)	0.023(2)	0.029(2)	−0.009(1)	0.015(1)	−0.011(1)
C(15)	4e		0.4814(4)	0.0216(3)	0.6433(2)	0.045(3)	0.037(2)	0.049(3)	0.001(2)	0.011(2)	−0.006(2)
C(16)	4e		0.3877(4)	−0.0368(3)	0.6383(2)	0.045(3)	0.034(2)	0.039(2)	0.003(2)	0.008(2)	−0.004(2)
C(17)	4e		0.2452(5)	−0.1371(4)	0.5943(3)	0.053(3)	0.048(3)	0.052(3)	−0.007(2)	0.008(2)	−0.014(2)
C(18)	4e		0.2614(5)	−0.1397(3)	0.6649(3)	0.052(3)	0.038(3)	0.056(3)	−0.008(2)	0.013(2)	−0.007(2)
C(19)	4e		0.1452(6)	−0.0818(5)	0.5626(3)	0.062(4)	0.093(5)	0.066(4)	−0.003(4)	−0.008(3)	−0.003(4)
C(20)	4e		0.2460(8)	−0.2261(5)	0.5634(4)	0.115(6)	0.056(4)	0.087(5)	−0.025(4)	0.030(5)	−0.032(4)
C(21)	4e		0.1526(5)	−0.1355(4)	0.6885(3)	0.058(4)	0.062(4)	0.085(4)	−0.022(3)	0.029(3)	−0.016(3)
C(22)	4e		0.3356(6)	−0.2156(4)	0.6941(3)	0.084(5)	0.051(3)	0.076(4)	0.000(3)	0.013(4)	0.013(3)
C(23)	4e		0.1941(5)	−0.1222(4)	0.8856(3)	0.086(5)	0.063(4)	0.079(5)	−0.007(4)	0.026(4)	0.029(4)
C(24)	4e		0.2588(5)	−0.0487(3)	0.8620(2)	0.058(3)	0.040(3)	0.049(3)	−0.003(2)	0.019(3)	0.006(2)
C(25)	4e		0.3693(5)	−0.0370(4)	0.8910(3)	0.064(4)	0.051(3)	0.051(3)	0.000(3)	0.005(3)	0.016(3)
C(26)	4e		0.4339(4)	0.0302(3)	0.8754(2)	0.051(3)	0.043(3)	0.032(2)	0.003(2)	0.005(2)	−0.002(2)
C(27)	4e		0.5548(5)	0.0415(3)	0.9127(2)	0.049(3)	0.061(4)	0.060(3)	0.010(3)	0.005(3)	0.004(3)
C(28)	4e		−0.0154(6)	0.1316(3)	0.6276(2)	0.068(4)	0.058(4)	0.069(4)	0.010(3)	−0.016(3)	−0.001(3)
C(29)	4e		0.1070(4)	0.1376(3)	0.6615(2)	0.050(3)	0.045(3)	0.041(3)	0.003(2)	0.000(2)	−0.004(2)
C(30)	4e		0.1716(5)	0.2065(4)	0.6478(2)	0.071(4)	0.051(3)	0.041(3)	0.004(3)	−0.002(3)	0.010(2)
C(31)	4e		0.2820(5)	0.2194(3)	0.6791(2)	0.060(3)	0.045(3)	0.038(3)	−0.003(2)	0.017(2)	0.000(2)
C(32)	4e		0.3454(5)	0.2986(4)	0.6615(2)	0.091(5)	0.058(4)	0.048(3)	−0.012(3)	0.014(3)	0.011(3)

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