Synthesis and crystal structure of a novel cobalt(II)coordination complex involving nitronyl nitroxide radical (NIT2-thp), $C_{32}H_{33}CoF_{12}N_4O_8S_2$

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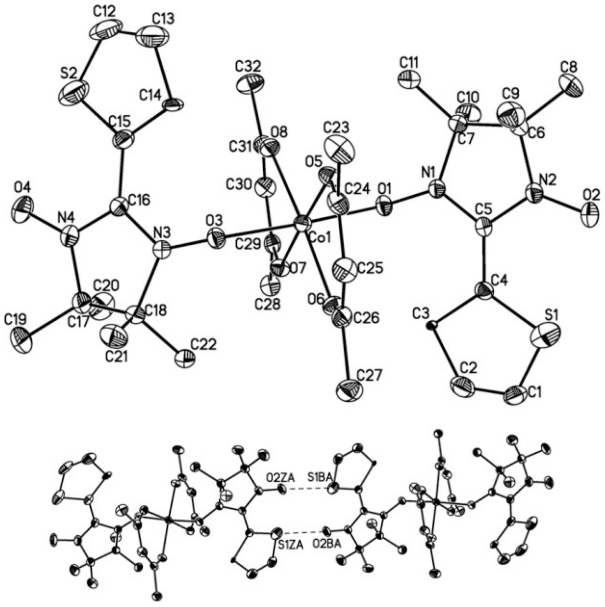

Abstract

$C_{32}H_{33}CoF_{12}N_4O_8S_2$, monoclinic, $P2_1/c$ (no. 14), a = 12.181(1) Å, b = 15.221(1) Å, c = 22.396(2) Å, β = 101.743(1)°, V = 4065.6 Å^3, Z = 4, $R_{gt}(F)$ = 0.0744, $wR_{ref}(F^2)$ = 0.2630, T = 291 K.

Table 1. Data collection and handling.

Crystal:	darkgreen blocks, size 0.29×0.34×0.45 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	6.32 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	24521, 8983
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 6220
$N(param)_{refined}$:	649
Programs:	SHELXS-97 [14]

Source of material

NIT2-thp was prepared according to a literature method [13]. The title complex was synthesized as follows: a mixture of $Co(hfac)_2{\cdot}2H_2O$ (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 darkgreen crystals.

Analysis calculated for $C_{32}H_{33}F_{12}N_4Co_1O_8S_2$ (%): C: 40.34; H: 3.49; N: 5.88. Found (%): C: 40.31; H: 3.46; N: 5.90.

Discussion

Stable organic nitronyl nitroxide radicals (NITR) show promise as molecular magnetic materials [1-8]. We are devoted to the explorations of new building blocks for molecule-based magnetic materials. As part of our own studies of these materials, we now report the synthesis and crystal structure of a novel cobalt(II) complex involving thiophene-substituted nitronyl nitroxide radical(NIT2-thp). The crystal structure of the title complex is shown in Fig. 1, top. The central cobalt atom is hexacoordinated 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, which is confirmed by the bond lengths parameter, the Co1–O1 bond length is 2.136(3)Å and the Co1–O3 bond length is 2.143(3)Å, which are larger than the Co1–O5 bond length (2.039(3)Å) and the Co1–O7 bond length (2.041(3)Å), and that the Co1–O6 bond length is 2.051(3)Å and the Co1–O8 bond length is 2.050(3)Å, respectively. The coordinated N–O bond lengths of N1–O1 and N3–O3 are 1.304(4)Å and 1.292(4)Å, respectively, which are similar to the correlative values observed in dissociative nitroxide (in 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 Co(II)ion in the apical direction. The uncoordinated N–O bond lengths of N2–O2 and N4–O4 are 1.277(5)Å and 1.266(5)Å, respectively. Bond distances and angles in the hfac molecules are in the normal range for this kind of compounds [9-11]. 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 13.6°.This dihedral angle is smaller than reported previous by us [12]. From the unit cell packing (Fig.2), two adjacent molecules of the title complex are linked by pairs of inversion-related O···S weak intermolecular interactions involving O2ZA···S1BA and S1ZA···O2BA, resulting in a dimeric structure, with the distance of O···S being 3.112Å. The CF_3-groups were handled according to the disorder in the crystal structure, the disordered CF_3-groups were considered in the refinement as rotational split models over two positions around the C–C bond. Besides, the C3 atom of the thiophene ring (C1, C2, C3, C4, S1) was refined, resulting in a larger R value.

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	4*e*	0.6801	0.1401	0.6015	0.087
H(2)	4*e*	0.6971	0.1403	0.705	0.099
H(3A)	4*e*	0.5096	0.0963	0.7292	0.026
H(3B)	4*e*	0.5878	0.0118	0.7358	0.026
H(8A)	4*e*	0.2406	–0.2194	0.5212	0.13
H(8B)	4*e*	0.1819	–0.2607	0.5707	0.13
H(8C)	4*e*	0.3131	–0.2588	0.5811	0.13
H(9A)	4*e*	0.1461	–0.0285	0.5852	0.118
H(9B)	4*e*	0.0788	–0.1144	0.5635	0.118
H(9C)	4*e*	0.1538	–0.0711	0.5224	0.118
H(10A)	4*e*	0.4069	–0.2145	0.6824	0.11
H(10B)	4*e*	0.2991	–0.271	0.6819	0.11
H(10C)	4*e*	0.3474	–0.2114	0.7382	0.11
H(11A)	4*e*	0.1707	–0.1383	0.7327	0.106
H(11B)	4*e*	0.1061	–0.1833	0.6728	0.106
H(11C)	4*e*	0.1171	–0.0807	0.6762	0.106
H(12)	4*e*	–0.1528	–0.0075	0.9121	0.135

Table 2. continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(13)	4*e*	–0.1531	0.0005	0.8111	0.129
H(14)	4*e*	–0.0107	0.1103	0.7784	0.063
H(19A)	4*e*	0.1742	0.4179	0.9494	0.145
H(19B)	4*e*	0.3049	0.4275	0.968	0.145
H(19C)	4*e*	0.2383	0.3831	1.0129	0.145
H(20A)	4*e*	0.3476	0.2453	1.0208	0.136
H(20B)	4*e*	0.4241	0.2794	0.9772	0.136
H(20C)	4*e*	0.3572	0.1915	0.9624	0.136
H(21A)	4*e*	0.1735	0.3681	0.7961	0.112
H(21B)	4*e*	0.2234	0.4325	0.8494	0.112
H(21C)	4*e*	0.1128	0.3806	0.8508	0.112
H(22A)	4*e*	0.4085	0.248	0.8739	0.093
H(22B)	4*e*	0.4106	0.3509	0.8706	0.093
H(22C)	4*e*	0.3593	0.2961	0.8123	0.093
H(25)	4*e*	0.1387	0.2486	0.6169	0.07
H(30)	4*e*	0.4045	–0.0778	0.9214	0.069

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}
F(1)	4*e*	0.66	–0.0460(7)	0.0537(6)	0.6097(5)	0.074(4)	0.070(4)	0.132(7)	–0.011(3)	–0.025(4)	–0.006(4)
F(2)	4*e*	0.66	–0.0851(7)	0.1509(7)	0.6647(4)	0.055(3)	0.124(6)	0.100(5)	0.015(4)	0.013(3)	0.002(4)
F(3)	4*e*	0.66	–0.041(1)	0.1911(9)	0.5803(6)	0.076(6)	0.106(7)	0.069(5)	0.019(5)	–0.015(4)	0.018(4)
F(4)	4*e*	0.66	0.288(1)	0.3435(7)	0.6107(4)	0.124(5)	0.070(5)	0.057(4)	–0.015(4)	0.016(4)	0.031(3)
F(5)	4*e*	0.66	0.443(1)	0.287(1)	0.6549(6)	0.118(7)	0.133(8)	0.152(9)	–0.027(6)	0.045(7)	0.030(7)
F(6)	4*e*	0.66	0.351(1)	0.3625(6)	0.7040(4)	0.175(8)	0.075(5)	0.093(5)	–0.045(5)	0.044(5)	–0.010(4)
F(7)	4*e*	0.66	0.626(1)	0.0404(8)	0.8761(6)	0.060(4)	0.104(7)	0.107(5)	0.010(6)	0.019(4)	0.007(6)
F(8)	4*e*	0.66	0.586(1)	–0.0266(9)	0.9543(5)	0.072(6)	0.082(4)	0.060(5)	0.016(4)	–0.005(4)	0.023(4)
F(9)	4*e*	0.66	0.570(1)	0.116(1)	0.9447(7)	0.076(6)	0.077(5)	0.095(8)	–0.014(4)	–0.009(5)	–0.008(5)
F(10)	4*e*	0.66	0.2517(8)	–0.1937(5)	0.8989(7)	0.116(5)	0.068(4)	0.182(9)	0.005(4)	0.045(6)	0.033(5)
F(11)	4*e*	0.66	0.1690(9)	–0.0979(6)	0.9391(4)	0.158(7)	0.138(6)	0.126(6)	–0.033(5)	0.099(5)	0.020(4)
F(12)	4*e*	0.66	0.1017(8)	–0.1433(7)	0.8478(5)	0.112(6)	0.110(6)	0.124(6)	–0.058(5)	0.007(5)	0.038(5)
F(1')	4*e*	0.34	–0.083(2)	0.105(2)	0.655(1)	0.083(8)	0.14(1)	0.13(1)	–0.012(9)	0.009(7)	–0.004(9)
F(2')	4*e*	0.34	–0.056(3)	0.197(2)	0.592(1)	0.09(1)	0.081(9)	0.10(1)	0.047(7)	0.002(7)	–0.004(7)
F(3')	4*e*	0.34	–0.015(1)	0.068(1)	0.5823(8)	0.077(7)	0.078(8)	0.091(8)	0.021(6)	–0.033(6)	–0.036(6)
F(4')	4*e*	0.34	0.428(2)	0.269(1)	0.6322(7)	0.077(7)	0.082(7)	0.070(6)	–0.019(5)	0.043(5)	0.015(5)
F(5')	4*e*	0.34	0.417(1)	0.336(1)	0.7080(6)	0.087(7)	0.078(7)	0.071(6)	–0.044(6)	–0.002(6)	0.007(5)
F(6')	4*e*	0.34	0.292(2)	0.358(2)	0.632(1)	0.113(9)	0.086(9)	0.11(1)	–0.006(7)	0.008(9)	0.027(9)
F(7')	4*e*	0.34	0.629(2)	0.072(1)	0.880(1)	0.049(6)	0.073(8)	0.082(7)	–0.007(7)	0.012(5)	–0.001(6)
F(8')	4*e*	0.34	0.605(2)	–0.024(2)	0.940(1)	0.071(8)	0.077(8)	0.08(1)	0.018(6)	0.009(7)	0.010(7)
F(9')	4*e*	0.34	0.553(2)	0.106(2)	0.955(1)	0.051(6)	0.063(8)	0.043(5)	–0.003(6)	–0.003(5)	–0.009(5)
F(10')	4*e*	0.34	0.246(2)	–0.164(1)	0.9325(7)	0.130(9)	0.104(9)	0.086(7)	–0.038(7)	0.010(7)	0.058(7)
F(11')	4*e*	0.34	0.095(1)	–0.114(1)	0.880(1)	0.075(7)	0.106(9)	0.13(1)	–0.012(6)	0.045(7)	0.033(7)
F(12')	4*e*	0.34	0.203(2)	–0.1958(9)	0.8453(8)	0.15(1)	0.078(7)	0.120(9)	–0.026(7)	0.046(8)	0.001(6)
Co(1)	4*e*		0.27016(4)	0.08313(4)	0.76830(2)	0.0420(3)	0.0402(3)	0.0323(3)	–0.0048(2)	0.0058(2)	0.0018(2)
S(1)	4*e*		0.5321(2)	0.0487(2)	0.5816(1)	0.089(1)	0.093(1)	0.115(2)	–0.001(1)	0.036(1)	0.020(1)
S(2)	4*e*		–0.0167(2)	0.0937(2)	0.9425(1)	0.099(1)	0.098(2)	0.169(2)	–0.006(1)	0.073(2)	0.024(1)
O(1)	4*e*		0.3528(2)	–0.0256(2)	0.7362(1)	0.050(2)	0.054(2)	0.036(1)	0.002(1)	0.007(1)	–0.008(1)
O(2)	4*e*		0.3827(4)	–0.0852(3)	0.5379(2)	0.092(3)	0.088(3)	0.043(2)	–0.014(2)	0.024(2)	–0.015(2)
O(3)	4*e*		0.1796(3)	0.1894(2)	0.7985(1)	0.051(2)	0.051(2)	0.042(2)	0.000(1)	0.010(1)	–0.008(1)
O(4)	4*e*		0.1107(5)	0.2403(4)	0.9897(2)	0.124(4)	0.129(4)	0.076(3)	–0.028(3)	0.067(3)	–0.024(3)
O(5)	4*e*		0.1345(3)	0.0798(2)	0.6979(1)	0.053(2)	0.043(2)	0.044(2)	–0.004(1)	–0.004(1)	–0.000(1)
O(6)	4*e*		0.3368(3)	0.1767(2)	0.7201(1)	0.051(2)	0.050(2)	0.043(2)	–0.005(1)	0.011(1)	0.008(1)
O(7)	4*e*		0.4092(3)	0.0882(2)	0.8367(1)	0.051(2)	0.045(2)	0.040(2)	–0.007(1)	0.001(1)	0.003(1)
O(8)	4*e*		0.2056(3)	–0.0061(2)	0.8204(1)	0.051(2)	0.047(2)	0.049(2)	–0.003(1)	0.014(1)	0.007(1)
N(1)	4*e*		0.3329(3)	–0.0591(2)	0.6815(2)	0.045(2)	0.045(2)	0.037(2)	0.001(2)	0.009(1)	–0.003(1)
N(2)	4*e*		0.3478(3)	–0.0872(3)	0.5880(2)	0.058(2)	0.051(2)	0.037(2)	–0.003(2)	0.011(2)	–0.004(2)
N(3)	4*e*		0.1883(3)	0.2206(2)	0.8529(2)	0.044(2)	0.042(2)	0.045(2)	–0.003(2)	0.013(2)	–0.004(2)
N(4)	4*e*		0.1547(4)	0.2461(3)	0.9433(2)	0.075(3)	0.066(3)	0.060(3)	–0.008(2)	0.034(2)	–0.011(2)
C(1)	4*e*		0.6314(5)	0.1082(4)	0.6203(3)	0.072(3)	0.061(3)	0.096(5)	–0.013(3)	0.043(3)	–0.002(3)
C(2)	4*e*		0.6397(5)	0.1091(5)	0.6802(4)	0.067(4)	0.070(4)	0.114(6)	–0.017(3)	0.024(4)	–0.031(4)
C(3)	4*e*		0.5537(2)	0.0582(2)	0.7086(1)	0.016(1)	0.022(2)	0.033(2)	–0.008(1)	0.015(1)	–0.010(1)
C(4)	4*e*		0.4814(4)	0.0203(3)	0.6431(2)	0.044(2)	0.038(2)	0.053(2)	0.000(2)	0.011(2)	–0.004(2)
C(5)	4*e*		0.3897(4)	–0.0388(3)	0.6383(2)	0.047(2)	0.042(2)	0.037(2)	0.004(2)	0.008(2)	–0.003(2)
C(6)	4*e*		0.2467(4)	–0.1382(3)	0.5951(2)	0.057(3)	0.048(3)	0.051(2)	–0.008(2)	0.008(2)	–0.011(2)
C(7)	4*e*		0.2635(4)	–0.1408(3)	0.6653(2)	0.057(3)	0.042(2)	0.053(2)	–0.009(2)	0.016(2)	–0.006(2)
C(8)	4*e*		0.2455(7)	–0.2276(4)	0.5642(3)	0.112(5)	0.059(4)	0.091(4)	–0.023(4)	0.025(4)	–0.031(3)

Table 3. continued.

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(9)	4*e*		0.1471(5)	–0.0829(5)	0.5636(3)	0.062(3)	0.093(5)	0.072(4)	–0.004(3)	–0.007(3)	–0.001(3)
C(10)	4*e*		0.3359(5)	–0.2164(4)	0.6946(3)	0.082(4)	0.053(3)	0.082(4)	–0.002(3)	0.012(3)	0.012(3)
C(11)	4*e*		0.1541(5)	–0.1353(4)	0.6889(3)	0.062(3)	0.071(4)	0.084(4)	–0.024(3)	0.027(3)	–0.020(3)
C(12)	4*e*		–0.1047(6)	0.0302(5)	0.8972(5)	0.065(4)	0.085(5)	0.20(1)	–0.019(4)	0.061(6)	0.013(6)
C(13)	4*e*		–0.1043(7)	0.0348(6)	0.8389(5)	0.083(5)	0.079(5)	0.163(8)	–0.026(4)	0.031(5)	–0.006(5)
C(14)	4*e*		–0.0226(3)	0.0974(3)	0.8172(3)	0.030(2)	0.038(2)	0.094(4)	–0.016(2)	0.025(2)	–0.016(2)
C(15)	4*e*		0.0348(4)	0.1327(3)	0.8819(3)	0.049(2)	0.048(3)	0.086(4)	–0.001(2)	0.028(2)	0.000(2)
C(16)	4*e*		0.1242(4)	0.1972(3)	0.8919(2)	0.052(2)	0.046(3)	0.058(3)	–0.002(2)	0.023(2)	–0.002(2)
C(17)	4*e*		0.2562(5)	0.3006(4)	0.9421(2)	0.062(3)	0.067(3)	0.057(3)	–0.008(3)	0.018(2)	–0.017(2)
C(18)	4*e*		0.2534(4)	0.3027(3)	0.8732(2)	0.049(2)	0.045(2)	0.050(2)	–0.009(2)	0.012(2)	–0.006(2)
C(19)	4*e*		0.2421(7)	0.3907(5)	0.9708(3)	0.110(5)	0.093(5)	0.093(5)	–0.023(4)	0.035(4)	–0.051(4)
C(20)	4*e*		0.3555(6)	0.2494(6)	0.9791(3)	0.085(4)	0.126(6)	0.056(3)	–0.002(4)	0.004(3)	0.008(4)
C(21)	4*e*		0.1843(6)	0.3780(4)	0.8392(3)	0.084(4)	0.050(3)	0.089(4)	–0.001(3)	0.013(3)	0.004(3)
C(22)	4*e*		0.3687(4)	0.2991(4)	0.8559(3)	0.058(3)	0.063(3)	0.070(3)	–0.023(2)	0.026(2)	–0.020(3)
C(23)	4*e*		–0.0150(5)	0.1326(4)	0.6252(3)	0.068(3)	0.063(4)	0.074(4)	0.011(3)	–0.015(3)	–0.001(3)
C(24)	4*e*		0.1073(4)	0.1404(3)	0.6599(2)	0.053(2)	0.049(3)	0.045(2)	0.004(2)	–0.002(2)	–0.008(2)
C(25)	4*e*		0.1703(5)	0.2091(4)	0.6472(2)	0.069(3)	0.058(3)	0.043(2)	0.002(2)	0.001(2)	0.011(2)
C(26)	4*e*		0.2815(4)	0.2223(3)	0.6783(2)	0.067(3)	0.049(3)	0.038(2)	–0.002(2)	0.017(2)	0.005(2)
C(27)	4*e*		0.3450(6)	0.3007(4)	0.6615(3)	0.090(4)	0.059(3)	0.056(3)	–0.014(3)	0.017(3)	0.014(3)
C(28)	4*e*		0.5566(4)	0.0419(4)	0.9134(2)	0.052(3)	0.064(3)	0.057(3)	0.009(2)	0.003(2)	0.002(2)
C(29)	4*e*		0.4363(4)	0.0302(3)	0.8767(2)	0.051(2)	0.047(2)	0.035(2)	0.005(2)	0.005(2)	–0.002(2)
C(30)	4*e*		0.3721(5)	–0.0380(3)	0.8914(2)	0.070(3)	0.052(3)	0.047(2)	0.002(2)	0.006(2)	0.014(2)
C(31)	4*e*		0.2612(4)	–0.0489(3)	0.8630(2)	0.062(3)	0.043(2)	0.047(2)	–0.004(2)	0.018(2)	0.006(2)
C(32)	4*e*		0.1956(6)	–0.1231(4)	0.8863(3)	0.076(4)	0.064(4)	0.092(5)	–0.007(3)	0.020(3)	0.035(3)

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