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Crystal structure of bis(*N*-(1-(3-ethylpyrazin-2-yl)ethylidene)-3-hydroxy-2-naphthohydrazonato- $\kappa^3 N, N', O$)cobalt(II) – dimethylformamide (1/1), $C_{41}H_{41}N_9O_5Co$

Table 1: Data collection and handling.

Crystal:	Black block
Size:	0.20 × 0.18 × 0.15 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.52 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	19010, 6608, 0.047
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4465
$N(\text{param})_{\text{refined}}$:	543
Programs:	Bruker [1], SHELX [2, 3]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Co1	0.28725(3)	0.25688(2)	0.17613(2)	0.03814(13)
N1	0.1221(2)	0.25634(12)	0.21637(11)	0.0467(6)
N2	−0.1046(3)	0.26224(16)	0.25884(15)	0.0743(8)
N3	0.1735(2)	0.33064(12)	0.11905(11)	0.0432(6)
N4	0.2204(2)	0.36677(12)	0.06932(11)	0.0453(6)
N5	0.3965(2)	0.32311(12)	0.25655(11)	0.0429(6)
N6	0.5482(3)	0.39816(14)	0.36735(13)	0.0581(7)
N7	0.4031(2)	0.18804(11)	0.23951(11)	0.0421(6)
N8	0.3951(2)	0.11689(11)	0.21985(11)	0.0439(6)
N9	−0.3524(4)	0.0796(3)	0.1293(2)	0.0938(11)
O1	0.39689(18)	0.29172(10)	0.10729(9)	0.0497(5)
O2	0.2107(2)	0.45151(11)	−0.03427(10)	0.0600(6)
H2	0.1870	0.4324	−0.0018	0.090*
O3	0.24146(18)	0.15949(10)	0.12402(9)	0.0501(5)
O4	0.4284(3)	−0.01861(11)	0.23656(10)	0.0746(7)
H4	0.4391	0.0245	0.2456	0.112*
O5	−0.1980(5)	0.0592(4)	0.2262(2)	0.212(3)
C1	0.0986(3)	0.21099(17)	0.26449(16)	0.0598(9)
H1	0.1599	0.1763	0.2839	0.072*
C2	−0.0135(4)	0.2144(2)	0.28572(19)	0.0762(11)
H2A	−0.0267	0.1825	0.3199	0.091*
C3	−0.0841(3)	0.30867(18)	0.21002(16)	0.0581(8)
C4	0.0312(3)	0.30614(15)	0.18782(14)	0.0450(7)
C5	−0.1922(3)	0.3621(2)	0.1828(2)	0.0942(14)
H5A	−0.1553	0.4102	0.1890	0.113*
H5B	−0.2238	0.3542	0.1326	0.113*
C6	−0.2999(4)	0.3612(3)	0.2132(2)	0.1202(18)
H6A	−0.3573	0.4000	0.1941	0.180*
H6B	−0.2708	0.3668	0.2632	0.180*

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Abstract

$C_{41}H_{41}N_9O_5Co$, monoclinic, $P2_1/n$ (no. 14), $a = 10.666(4)$ Å, $b = 18.538(6)$ Å, $c = 19.585(7)$ Å, $\beta = 104.036(7)^\circ$, $V = 3757(2)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0420$, $wR_{\text{ref}}(F^2) = 0.1062$, $T = 296$ K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

3-Hydroxy-2-naphthohydrazide (0.202 g, 1 mmol) and 3-ethyl-2-aceto-pyrazine (0.150 g, 1 mmol) was dissolved in methanol (20 mL). The reaction mixture was refluxed for 1 h and cooled to room temperature. Then cobalt(II) acetate tetrahydrate (0.125 g, 0.5 mmol) was added. After stirring for 1 h, the mix-

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Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
H6C	−0.3447	0.3161	0.2027	0.180*
C7	0.0679(3)	0.35240(14)	0.13394(13)	0.0429(7)
C8	−0.0009(3)	0.41893(16)	0.10160(16)	0.0602(9)
H8A	0.0568	0.4480	0.0824	0.090*
H8B	−0.0292	0.4460	0.1369	0.090*
H8C	−0.0743	0.4056	0.0648	0.090*
C9	0.3375(3)	0.34075(14)	0.06746(14)	0.0421(7)
C10	0.3964(3)	0.37545(14)	0.01451(14)	0.0424(7)
C11	0.3309(3)	0.42834(15)	−0.03423(14)	0.0468(7)
C12	0.3891(3)	0.45611(15)	−0.08355(15)	0.0551(8)
H12	0.3440	0.4889	−0.1164	0.066*
C13	0.5148(3)	0.43646(15)	−0.08580(14)	0.0498(8)
C14	0.5770(4)	0.46575(17)	−0.13496(17)	0.0624(9)
H14	0.5334	0.4989	−0.1679	0.075*
C15	0.6995(4)	0.44655(17)	−0.13538(17)	0.0643(9)
H15	0.7393	0.4669	−0.1681	0.077*
C16	0.7665(3)	0.39605(17)	−0.08659(17)	0.0620(9)
H16	0.8507	0.3831	−0.0868	0.074*
C17	0.7084(3)	0.36622(16)	−0.03927(16)	0.0574(8)
H17	0.7531	0.3324	−0.0075	0.069*
C18	0.5817(3)	0.38514(14)	−0.03688(14)	0.0468(7)
C19	0.5178(3)	0.35548(14)	0.01174(14)	0.0480(7)
H19	0.5605	0.3208	0.0432	0.058*
C20	0.3888(3)	0.39388(15)	0.26052(15)	0.0504(8)
H20	0.3290	0.4188	0.2262	0.060*
C21	0.4682(3)	0.43136(17)	0.31491(16)	0.0588(8)
H21	0.4653	0.4815	0.3146	0.071*
C22	0.5553(3)	0.32569(16)	0.36571(14)	0.0497(7)
C23	0.4839(3)	0.28740(15)	0.30768(13)	0.0428(7)
C24	0.6423(3)	0.29476(18)	0.43133(15)	0.0693(10)
H24A	0.6264	0.2433	0.4330	0.083*
H24B	0.6211	0.3166	0.4721	0.083*
C25	0.7838(4)	0.3070(2)	0.4351(2)	0.0969(13)
H25A	0.8080	0.2802	0.3984	0.145*
H25B	0.8345	0.2912	0.4800	0.145*
H25C	0.7987	0.3575	0.4293	0.145*
C26	0.4911(3)	0.20909(15)	0.29322(14)	0.0440(7)
C27	0.5938(3)	0.15981(18)	0.33216(17)	0.0810(11)
H27A	0.5671	0.1388	0.3712	0.121*
H27B	0.6722	0.1865	0.3492	0.121*
H27C	0.6083	0.1223	0.3012	0.121*
C28	0.3073(3)	0.10864(14)	0.15846(14)	0.0407(7)
C29	0.2895(3)	0.03377(14)	0.13145(13)	0.0390(6)
C30	0.3512(3)	−0.02674(15)	0.17111(14)	0.0469(7)
C31	0.3294(3)	−0.09456(15)	0.14410(15)	0.0568(8)
H31	0.3698	−0.1334	0.1705	0.068*
C32	0.2475(3)	−0.10733(15)	0.07725(15)	0.0479(7)
C33	0.2181(3)	−0.17728(16)	0.04934(17)	0.0671(9)
H33	0.2551	−0.2172	0.0753	0.081*
C34	0.1364(3)	−0.18705(18)	−0.01497(18)	0.0732(10)
H34	0.1169	−0.2336	−0.0320	0.088*
C35	0.0814(3)	−0.12809(18)	−0.05578(17)	0.0645(9)
H35	0.0264	−0.1355	−0.0999	0.077*
C36	0.1083(3)	−0.06018(16)	−0.03105(15)	0.0540(8)
H36	0.0729	−0.0211	−0.0589	0.065*
C37	0.1896(3)	−0.04804(14)	0.03665(14)	0.0420(7)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C38	0.2133(3)	0.02157(14)	0.06536(14)	0.0436(7)
H38	0.1759	0.0608	0.0384	0.052*
C39 ^a	−0.4320(10)	0.1261(6)	0.0810(6)	0.101(4)
H39A ^a	−0.3987	0.1308	0.0399	0.151*
H39B ^a	−0.4339	0.1727	0.1023	0.151*
H39C ^a	−0.5180	0.1068	0.0679	0.151*
C39A ^b	−0.3512(15)	0.1604(7)	0.1381(9)	0.140(7)
H39D ^b	−0.2983	0.1817	0.1102	0.210*
H39E ^b	−0.3168	0.1724	0.1867	0.210*
H39F ^b	−0.4377	0.1786	0.1229	0.210*
C40 ^a	−0.3592(9)	0.0011(5)	0.1125(6)	0.102(4)
H40A ^a	−0.4447	−0.0164	0.1105	0.153*
H40B ^a	−0.2980	−0.0245	0.1483	0.153*
H40C ^a	−0.3392	−0.0064	0.0678	0.153*
C40A ^b	−0.4328(12)	0.0560(11)	0.0623(7)	0.147(8)
H40D ^b	−0.5181	0.0758	0.0561	0.220*
H40E ^b	−0.4376	0.0043	0.0615	0.220*
H40F ^b	−0.3960	0.0724	0.0249	0.220*
C41 ^a	−0.2658(13)	0.1000(9)	0.1862(6)	0.131(5)
H41 ^a	−0.2569	0.1491	0.1959	0.157*
C41A ^b	−0.2808(16)	0.0398(11)	0.1727(8)	0.133(6)
H41A ^b	−0.2897	−0.0096	0.1646	0.159*

^aOccupancy: 0.536(12), ^bOccupancy: 0.464(12).

ture was filtered and set aside to crystallize for several days, giving black block crystals.

Experimental details

The structure was solved by direct methods and refined with the SHELX crystallographic software package [3]. The C39—C41 atoms occupied two positions, with the occupancy value of C39/C39A, C40/C40A, or C41/C41A being 0.54/0.46. The hydrogen atoms were placed at calculated positions as riding atoms with isotropic displacement parameters.

Comment

As Schiff bases, hydrazones are an important class of ligands with interesting ligation properties due to the presence of several coordination sites, and are widely applied [4]. In particular, pyrazine-contained hydrazone and their metal complexes have been investigated mainly due to their excellent biological activities [5–8].

In the title structure, the asymmetric unit contains one neutral complex molecule and one DMF molecule (see the Figure). The C=O bond of the ligand is enolized, which could be confirmed by the bond length of hydrazone C—O being 1.263(3)/1.268(3) Å [5–7]. All bond distances and angles are in their normal ranges, and can be compared with those in previously reported Co(II) or Ni(II) complexes with pyrazine hydrazones [6–9]. The central Co(II) ion with a distorted octahedral

coordination geometry is surrounded by two anionic ligands with ON_2 donor set, which is the most common coordination mode in pyrazine hydrazone complexes [6–8]. In the solid state, there exist intramolecular $N-H\cdots O$ hydrogen bonds.

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