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Crystal structure of tetramethylammonium (2-(3-ethoxy-2-oxidobenzylidene)-1-(2-hydroxybenzoyl)hydrazin-1-ido- $\kappa^3 N, N', O$)cobalt(III), $C_{36}H_{40}N_5O_8Co$

The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Red block
Size:	0.10 × 0.08 × 0.06 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.54 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
$\theta_{\max}$, completeness:	26.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	33219, 7286, 0.041
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 5421
$N(\text{param})_{\text{refined}}$:	469
Programs:	Bruker [1], SHELX [2, 3]

Source of material

3-ethoxy-2-oxidobenzylidene(2-hydroxybenzo)hydrazine was synthesized according to the reference [4], the other reagents were commercially available and used as purchased.

A mixture of 3-ethoxy-2-oxidobenzylidene(2-hydroxybenzo)hydrazine (299.3 mg, 0.3 mmol), $CoCl_2$ (129.8 mg, 0.1 mmol) and methanol (10 mL) was heated at 60 °C for 10 h, and then cooled to room temperature. The resulting light red block crystals of the title compound were obtained. The yield is ca. 26%. IR(KBr-pellet): 3212(s), 1514(s), 1511(s), 1411(s), 1401(s), 1310(s), 1210(s), 1113(s), 1101(s), 1010(s), 1000(s), 914(s), 817(s), 801(m), 731(m), 711(m), 600(m), 444(m) cm⁻¹.

Experimental details

Hydrogen atoms are added using the standard options of the SHELX [3] system (Table 2). The ethyl group of the ligand shows a disorder (C15, C16; Figure).

Comment

The aroylhydrazones and their reactions still attract much attention due to their coordination ability to metal ions and their biological activity. In particular, aroylhydrazones with a pyridinyl moiety present high sensitivity and selectivity to some metals and have therefore been applied as analytical

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Abstract

$C_{36}H_{40}N_5O_8Co$, monoclinic, $P2_1/c$ (no. 14), $a = 17.353(4)$ Å, $b = 11.474(2)$ Å, $c = 18.074(4)$ Å, $\beta = 101.056(2)^\circ$, $V = 3532.0(13)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0464$, $wR_{\text{ref}}(F^2) = 0.1323$, $T = 296(2)$ K 1–3.

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

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
Co1	0.24045(2)	0.25721(3)	0.57882(2)	0.03228(12)
N1	0.27353(12)	0.48825(18)	0.61118(12)	0.0384(5)
O1	0.24994(14)	0.70445(19)	0.63845(14)	0.0668(6)
H1	0.2725	0.6413	0.6434	0.100*
C1	−0.02061(17)	0.0614(3)	0.75505(17)	0.0553(8)
H1A	−0.0584	0.0030	0.7510	0.066*
O2	0.00632(14)	0.0043(2)	0.63735(12)	0.0659(6)
H2	0.0329	0.0236	0.6065	0.099*
N2	0.30763(11)	0.37843(18)	0.62017(11)	0.0347(5)
C2	−0.00747(18)	0.1320(3)	0.81745(18)	0.0615(8)
H2A	−0.0368	0.1215	0.8550	0.074*
O3	0.18907(10)	0.24943(14)	0.66318(9)	0.0368(4)
N3	0.11436(12)	0.11507(18)	0.58660(11)	0.0369(5)
C3	0.0489(2)	0.2184(3)	0.82504(19)	0.0659(9)
H3	0.0572	0.2666	0.8672	0.079*
O4	0.17116(10)	0.38264(14)	0.54242(9)	0.0373(4)
N4	0.16744(11)	0.14182(17)	0.54015(11)	0.0335(4)
C4	0.09257(18)	0.2325(3)	0.76988(17)	0.0510(7)
H4	0.1308	0.2903	0.7754	0.061*
C5	0.08082(14)	0.1619(2)	0.70552(14)	0.0362(5)
N5	0.23963(17)	0.9136(3)	0.82449(16)	0.0680(8)
O5	0.30993(10)	0.13781(16)	0.61862(10)	0.0432(4)
C6	0.02189(16)	0.0762(2)	0.69779(15)	0.0432(6)
O6	0.28588(11)	0.26709(16)	0.49345(10)	0.0442(4)
C7	0.13130(14)	0.1775(2)	0.64891(13)	0.0335(5)
O7	0.40203(14)	−0.0588(2)	0.63875(15)	0.0720(7)
O8	0.36805(16)	0.2996(2)	0.38529(16)	0.0802(8)
C8	0.16446(16)	0.0828(2)	0.47908(14)	0.0400(6)
H8	0.1265	0.0248	0.4678	0.048*
C9	0.21680(16)	0.1019(2)	0.42756(13)	0.0427(6)
C10	0.20912(19)	0.0264(3)	0.36479(15)	0.0545(8)
H10	0.1732	−0.0346	0.3599	0.065*
C13	0.31704(19)	0.2068(3)	0.37957(16)	0.0559(8)
C12	0.3072(2)	0.1335(4)	0.31849(17)	0.0674(10)
H12	0.3368	0.1452	0.2813	0.081*
C11	0.2539(2)	0.0421(4)	0.31121(17)	0.0694(10)
H11	0.2487	−0.0080	0.2701	0.083*
C14	0.27247(16)	0.1933(2)	0.43720(14)	0.0422(6)
C15 ^a	0.4388(4)	0.2880(7)	0.4152(8)	0.194(7)
H15A ^a	0.4577	0.2187	0.3936	0.233*
H15B ^a	0.4407	0.2715	0.4682	0.233*
C15 ^b	0.4200(9)	0.2908(18)	0.3388(8)	0.106(15)
H15C ^b	0.4035	0.3299	0.2908	0.127*
H15D ^b	0.4365	0.2115	0.3317	0.127*
C36	0.2924(5)	0.8335(8)	0.8706(5)	0.224(4)
H36A	0.3146	0.7815	0.8388	0.336*
H36B	0.2639	0.7893	0.9017	0.336*
H36C	0.3336	0.8764	0.9020	0.336*
C35	0.1679(4)	0.8486(6)	0.7889(4)	0.151(2)
H35A	0.1330	0.9000	0.7565	0.227*
H35B	0.1421	0.8189	0.8274	0.227*
H35C	0.1823	0.7850	0.7599	0.227*
C34	0.2750(3)	0.9649(5)	0.7641(3)	0.1205(18)
H34A	0.2375	1.0151	0.7336	0.181*
H34B	0.2899	0.9038	0.7334	0.181*

Table 2 (continued)

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
H34C	0.3206	1.0092	0.7860	0.181*
C33	0.2132(4)	1.0072(4)	0.8684(3)	0.136(2)
H33A	0.2579	1.0438	0.8992	0.204*
H33B	0.1799	0.9755	0.9001	0.204*
H33C	0.1845	1.0638	0.8349	0.204*
C16	0.4953(3)	0.3799(6)	0.4114(4)	0.150(2)
H16A	0.5279	0.3584	0.3763	0.225*
H16B	0.5274	0.3913	0.4604	0.225*
H16C	0.4680	0.4509	0.3950	0.225*
C17	0.3987(3)	−0.0599(5)	0.5066(3)	0.1246(19)
H17A	0.4051	0.0229	0.5028	0.187*
H17B	0.3702	−0.0895	0.4596	0.187*
H17C	0.4494	−0.0964	0.5182	0.187*
C19	0.43366(18)	0.0516(3)	0.65431(17)	0.0557(8)
C18	0.3573(3)	−0.0840(4)	0.5636(3)	0.0901(13)
H18A	0.3425	−0.1656	0.5609	0.108*
H18B	0.3094	−0.0382	0.5552	0.108*
C20	0.38539(15)	0.1525(3)	0.64365(14)	0.0443(6)
C21	0.42262(16)	0.2617(3)	0.66253(16)	0.0467(7)
C22	0.50355(18)	0.2662(3)	0.6952(2)	0.0662(9)
H22	0.5270	0.3378	0.7093	0.079*
C23	0.5476(2)	0.1672(4)	0.7064(2)	0.0804(11)
H23	0.6006	0.1710	0.7284	0.097*
C24	0.5125(2)	0.0599(4)	0.6845(2)	0.0723(10)
H24	0.5431	−0.0073	0.6904	0.087*
C25	0.38076(14)	0.3702(3)	0.65106(14)	0.0425(6)
H25	0.4080	0.4384	0.6668	0.051*
C26	0.20202(14)	0.4792(2)	0.56884(13)	0.0347(5)
C27	0.15713(15)	0.5884(2)	0.55158(14)	0.0384(6)
C28	0.18269(17)	0.6950(2)	0.58620(16)	0.0476(7)
C29	0.1373(2)	0.7942(3)	0.5679(2)	0.0628(9)
H29	0.1542	0.8651	0.5902	0.075*
C30	0.0673(2)	0.7885(3)	0.5170(2)	0.0626(9)
H30	0.0372	0.8555	0.5057	0.075*
C31	0.04119(18)	0.6846(3)	0.48248(18)	0.0574(8)
H31	−0.0059	0.6815	0.4479	0.069*
C32	0.08590(16)	0.5857(2)	0.49999(16)	0.0463(6)
H32	0.0683	0.5156	0.4770	0.056*

^aOccupancy: 0.834(13), ^bOccupancy: 0.166(13).

reagents. Such compounds as *cis*-(dimethyl sulfoxide-κO) [N'-(3-ethoxy-2-oxidobenzylidene-κO)-2-hydroxybenzohydrazidato-κ²N',O]-dioxidomolybdenum(VI) and 2-hydroxy-3-methoxybenzaldehyde(pyridine-2-carbonyl)hydrazone have been synthesized and characterized by X-ray diffraction [5–8]. We now report a similar compound, whose structure was determined by X-ray diffraction.

The compound crystallizes in the monoclinic space group *P*₂₁/*c* with four formula units in the unit cell. As shown in the Figure, one formula unit consists of one cation, two dianionic ligands and one Co atom. The formal Co(III) metal ion is in a slightly distorted octahedral coordination environment, with two nitrogen atoms and four oxygen atoms from the ligands. The average bond length of Co–N is 1.873 Å, while the

average bond length of Co—O is 1.892 Å. Bond lengths and angles in the cation and the organic ligands are all in the expected regions [9].

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