Zhang Cuihong, Ge Yuan, Huo Xiaoping, Yang Ligu, Li Xin and Miao Zongcheng*

Crystal structure of tris(1-phenyl-1,2-propanedione-2-oximato- κ^2N,O)cobalt(III), $C_{27}H_{24}N_3O_6Co$

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 :	0.76 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
$\theta_{\max}$, completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	38447, 4240, 0.021
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3919
$N(\text{param})_{\text{refined}}$:	337
Programs:	Bruker [1], SHELX [2, 3]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Co1	0.85316(2)	0.11780(2)	0.35469(2)	0.03228(10)
O1	1.00135(12)	0.13107(6)	0.49349(11)	0.0361(3)
O2	0.88545(15)	0.20474(8)	0.17625(13)	0.0599(4)
O3	0.93222(11)	0.04504(6)	0.29438(10)	0.0357(3)
O4	0.61910(13)	0.13382(8)	0.17691(14)	0.0532(4)
O5	0.75114(14)	0.24365(6)	0.38475(13)	0.0488(3)
O6	0.78811(11)	0.05412(6)	0.44362(11)	0.0344(3)
N1	0.93226(15)	0.18253(8)	0.28302(14)	0.0410(4)
N2	0.72124(14)	0.10204(8)	0.20801(14)	0.0393(3)
N3	0.76138(13)	0.18163(7)	0.41373(13)	0.0350(3)
C1	1.20322(17)	0.17829(10)	0.56864(19)	0.0450(5)
C2	1.2794(2)	0.23622(13)	0.5796(3)	0.0648(7)
H2	1.2560	0.2718	0.5234	0.078*
C3	1.3935(2)	0.23941(17)	0.6788(3)	0.0840(10)
H3	1.4463	0.2770	0.6860	0.101*
C4	1.4269(2)	0.18808(19)	0.7635(3)	0.0853(10)
H4	1.5015	0.1910	0.8281	0.102*
C5	1.3511(2)	0.13291(15)	0.7533(3)	0.0721(7)
H5	1.3737	0.0985	0.8119	0.087*
C6	1.2405(2)	0.12706(12)	0.6567(2)	0.0519(5)
H6	1.1905	0.0884	0.6506	0.062*
C7	1.08132(16)	0.17002(9)	0.47006(17)	0.0374(4)
C8	1.04865(18)	0.20016(9)	0.35243(19)	0.0428(4)
C9	1.1254(2)	0.24358(12)	0.2975(3)	0.0658(7)
H9A	1.0962	0.2378	0.2107	0.099*
H9B	1.2116	0.2299	0.3297	0.099*
H9C	1.1178	0.2909	0.3170	0.099*
C10	0.91793(16)	−0.03330(9)	0.13723(16)	0.0367(4)
C11	1.01071(17)	−0.07356(10)	0.21723(17)	0.0424(4)

<https://doi.org/10.1515/ncrs-2018-0479>

Received November 5, 2018; accepted December 14, 2018; available online January 30, 2019

Abstract

$C_{27}H_{24}N_3O_6Co$, monoclinic, $P2_1/c$ (no. 14), $a = 11.309(3)$ Å, $b = 19.477(5)$ Å, $c = 11.615(3)$ Å, $\beta = 109.071(9)^\circ$, $V = 2418.0(11)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0296$, $wR_{\text{ref}}(F^2) = 0.0845$, $T = 293(2)$ K [1–3].

CCDC no.: 1885223

The asymmetric unit of the title 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.

*Corresponding author: Miao Zongcheng, Key Laboratory of Organic Polymer Photoelectric Materials, School of Science, Xijing University, Xi'an 710123, Shaanxi, P.R. China, e-mail: miaocongcheng@xijing.edu.cn

Zhang Cuihong, Ge Yuan, Huo Xiaoping and Li Xin: Key Laboratory of Organic Polymer Photoelectric Materials, School of Science, Xijing University, Xi'an 710123, Shaanxi, P.R. China

Yang Ligu: College of Chemistry and Environmental Engineering, Anyang Institute of Technology, Anyang 455000, Henan, P.R. China

Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
H11	1.0332	−0.0659	0.3007	0.051*
C12	1.0695(2)	−0.12468(10)	0.1740(2)	0.0507(5)
H12	1.1314	−0.1511	0.2284	0.061*
C13	1.0370(2)	−0.13691(11)	0.0503(2)	0.0512(5)
H13	1.0770	−0.1713	0.0214	0.061*
C14	0.9452(2)	−0.09795(11)	−0.02947(19)	0.0532(5)
H14	0.9232	−0.1061	−0.1127	0.064*
C15	0.8849(2)	−0.04661(11)	0.01251(18)	0.0482(5)
H15	0.8222	−0.0209	−0.0425	0.058*
C16	0.86540(16)	0.02350(9)	0.18961(15)	0.0353(4)
C17	0.74568(17)	0.05470(10)	0.13459(17)	0.0423(4)
C18	0.6506(2)	0.04153(16)	0.0124(2)	0.0760(8)
H18A	0.6736	0.0660	−0.0489	0.114*
H18B	0.5700	0.0569	0.0125	0.114*
H18C	0.6472	−0.0068	−0.0049	0.114*
C19	0.67859(15)	0.03677(8)	0.58067(14)	0.0318(3)
C20	0.73713(18)	−0.02688(10)	0.61056(17)	0.0424(4)
H20	0.8055	−0.0375	0.5861	0.051*
C21	0.6945(2)	−0.07406(11)	0.6761(2)	0.0531(5)
H21	0.7345	−0.1163	0.6958	0.064*
C22	0.5929(2)	−0.05927(11)	0.71275(18)	0.0508(5)
H22	0.5641	−0.0914	0.7566	0.061*
C23	0.53463(19)	0.00304(11)	0.68405(18)	0.0470(5)
H23	0.4662	0.0131	0.7088	0.056*
C24	0.57629(17)	0.05134(10)	0.61857(16)	0.0389(4)
H24	0.5360	0.0935	0.5999	0.047*
C25	0.72486(14)	0.08301(8)	0.50316(14)	0.0308(3)
C26	0.70598(15)	0.15528(9)	0.49066(15)	0.0331(4)
C27	0.63657(19)	0.20187(10)	0.54813(19)	0.0449(4)
H27A	0.6668	0.2480	0.5487	0.067*
H27B	0.6493	0.1872	0.6302	0.067*
H27C	0.5489	0.2004	0.5024	0.067*

Source of material

The educt 1-phenyl-1,2-propanedione-2-oxime was synthesized according to the literature [4], the other reagents were commercially available and used as purchased.

A mixture of bisalicylhydrazide (163.16 mg, 0.3 mmol), CoCl₂ (129.84 mg, 0.1 mmol) and methanol (10 mL) heated at 60 °C for 10 h, and then cooled to room temperature. Red block crystals of the title complex were obtained. The yield is ca. 26%. IR(KBr): 3216(s), 1518(s), 1514(s), 1412(s), 1401(s), 1312(s), 1211(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

H atoms were added using the standard riding options of the SHELX system [3].

Comment

Most carbonyl compounds, including carbonyloximes, readily react with primary amines to yield Schiff bases and, in the presence of metal ions, and chelates complexes of imine-oxime type can be isolated. In the present case preservation of the carbonyl function of the ligand was assured by preparing first the Co(en)₃ complex from Co(ClO₄)₂ and ethylenediamine in methanol and the replacing of one of the en ligands by 1-phenyl-1,2-propanedione 2-oximate. During the process the Co ion is oxidized to the trivalent state. A similar structure has been reported [5]. The complex containing (1-phenyl-1,2-propanedione-2-oxime) ligand is rare. Now we synthesized the new complex directly.

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 three oxime ligands and one Co atom. The Co ion is in a slightly distorted octahedral coordination environment, with one nitrogen atom and one oxygen atom from each ligand. The average bond length of Co—N is 1.887 Å, while the average bond length of Co—O is 1.920 Å.

Acknowledgements: This work was supported by the National Natural Science Foundation of China (Grant No. 51602007, Grant No. 51673157), the Natural Science Basic Research Plan in Shaanxi Province of China (Grant No. 2017JM5134, Grant No. 2018JM5047, Scientific Research Program Funded by Shaanxi Provincial Education Department (Grant No. 18JK1206), the Science Research Foundation of Xijing University (Grant No. XJ17B07), the National Students' Platform for Innovation and Entrepreneurship Training Program (Grant No. 201812715003).

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