Crystal structure of bis(nitrito-*N*)(1,10-phenanthroline-*N*,*N*')(2,4-pentanedionato-*O*,*O*')cobalt(III), $Co(NO_2)_2(C_{12}H_8N_2)(C_5H_7O_2)$

Y.-L. Feng*[I] and H. Lin[II]

[I] Zhejiang Normal University, Institute of Physical Chemistry, Jinhua, Zhejiang, 321004 P. R. China
[II] Jinhua Professional-Technical College, Jinhua, Zhejiang, 321017 P. R. China

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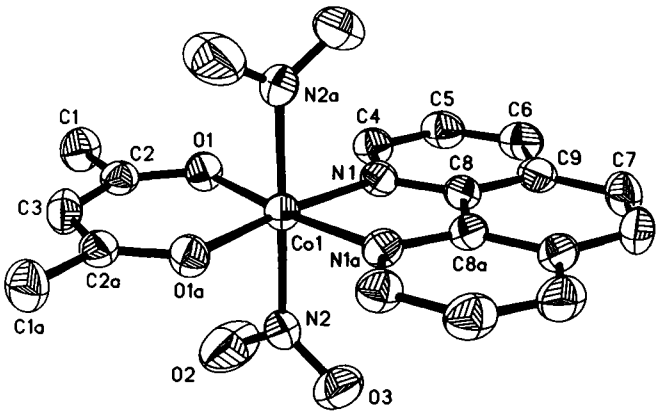

Abstract

$C_{17}H_{15}CoN_4O_6$, monoclinic, *C*12/*c*1 (no. 15), $a = 15.017(3)$ Å, $b = 15.107(3)$ Å, $c = 10.015(2)$ Å, $\beta = 118.68(3)°$, $V = 1993.3$ Å^3, $Z = 4$, $R_{gt}(F) = 0.056$, $wR_{ref}(F^2) = 0.188$, $T = 293$ K.

Source of material

A solution of 2,4-pentanedione (1.0 mmol, acac), 1,10-phenanthroline (1.0 mmol, phen), $CoCl_2 \cdot 6H_2O$ (1.0 mmol) and $NaNO_2$ (2.0 mmol) in 20 mL ethanol was stirred violently for 2 h at room temperature, forming a red solution. The solution was filtered and the filtrate was allowed to stand for slow evaporation to provide the red prismatic title crystals suitable for X-ray structure determination.

Discussion

The cobalt(II) complex of β-diketone has been reported of catalyzing the oxidation of olefins to ketones [1]. We reported some Co(II) complexes of β-diketones [2,3]. Here we report the crystal structure of a cobalt(III) complex with 2,4-pentanedione ligand.

The crystal structure is composed of discrete molecules of $Co(NO_2)_2(phen)(acac)$. The molecule lies on the crystallographic twofold rotation axis. Each Co(III) atom has a distorted octahedral environment defined by two oxygen atoms from the 2,4-pentanedionato, two nitrogen atoms from the 1,10-phenanthroline ligands and axially by two nitrogen atoms from nitrito groups. The bond lengths of Co1—O1, Co1—N1 and Co1—N2 are 1.871(3) Å, 1.939(4) Å and 1.952(5) Å, respectively.

Table 1. Data collection and handling.

Crystal:	red prism, size 0.20 × 0.30 × 0.30 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	9.01 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, ω/φ
$2\theta_{max}$:	46.5°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2036, 1378
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1053
$N(param)_{refined}$:	128
Programs:	SHELXS-97 [4], SHELXL-97 [5]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1A)	8*f*	0.3229	0.0721	−0.1045	0.112
H(1B)	8*f*	0.4066	−0.0015	−0.0537	0.112
H(1C)	8*f*	0.3230	−0.0068	−0.0027	0.112
H(3)	4*e*	½	−0.0230	¼	0.078
H(4)	8*f*	0.3351	0.2859	−0.0826	0.072
H(5)	8*f*	0.2652	0.4129	−0.2258	0.088
H(6)	8*f*	0.3150	0.5512	−0.1179	0.089
H(7)	8*f*	0.4381	0.6446	0.1247	0.094

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Co(1)	4*e*	½	0.25124(5)	¼	0.0438(7)	0.0462(7)	0.0404(6)	0	0.0141(5)	0
O(1)	8*f*	0.4208(2)	0.1685(2)	0.1010(3)	0.054(2)	0.054(2)	0.048(2)	−0.003(2)	0.016(2)	−0.007(2)
O(2)	8*f*	0.5999(5)	0.1922(4)	0.0967(7)	0.155(5)	0.139(5)	0.197(6)	−0.057(4)	0.139(5)	−0.086(5)
O(3)	8*f*	0.6683(3)	0.3058(3)	0.2243(6)	0.091(3)	0.096(4)	0.140(4)	−0.021(3)	0.077(3)	−0.019(3)
N(1)	8*f*	0.4307(3)	0.3470(3)	0.1090(4)	0.047(2)	0.052(3)	0.048(2)	0.005(2)	0.018(2)	0.002(2)
N(2)	8*f*	0.6014(4)	0.2496(3)	0.1808(5)	0.056(3)	0.051(3)	0.056(3)	0.003(2)	0.026(2)	0.007(2)
C(1)	8*f*	0.3650(5)	0.0325(4)	−0.0237(7)	0.083(4)	0.067(4)	0.075(4)	−0.011(3)	0.039(3)	−0.025(3)
C(2)	8*f*	0.4314(4)	0.0845(3)	0.1163(6)	0.052(3)	0.056(3)	0.059(3)	−0.006(2)	0.032(2)	−0.009(3)
C(3)	4*e*	½	0.0406(5)	¼	0.080(5)	0.043(4)	0.078(5)	0	0.043(5)	0

* Correspondence author (e-mail: sky37@zjnu.cn)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(4)	8*f*	0.3584(4)	0.3428(4)	–0.0361(5)	0.052(3)	0.071(4)	0.051(3)	0.001(2)	0.019(2)	0.007(3)
C(5)	8*f*	0.3162(4)	0.4181(4)	–0.1211(6)	0.059(3)	0.089(4)	0.061(3)	0.014(3)	0.020(3)	0.018(3)
C(6)	8*f*	0.3459(5)	0.4992(4)	–0.0586(7)	0.069(4)	0.077(4)	0.077(4)	0.020(3)	0.036(3)	0.032(3)
C(7)	8*f*	0.4635(4)	0.5892(4)	0.1761(7)	0.087(4)	0.048(3)	0.116(5)	0.011(3)	0.061(4)	0.018(3)
C(8)	8*f*	0.4615(4)	0.4283(3)	0.1725(5)	0.048(3)	0.055(3)	0.059(3)	0.004(2)	0.028(2)	0.006(2)
C(9)	8*f*	0.4229(4)	0.5072(3)	0.0951(6)	0.061(3)	0.056(3)	0.084(4)	0.011(3)	0.042(3)	0.014(3)

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