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X-Ray Structural Analysis of Trinuclear Cobalt Carbonyl Complex $SCo_3(CO)_9$

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A trinuclear cobalt carbonyl complex $SCo_3(CO)_9$ was characterized by single crystal X-ray diffraction analysis. The crystals are triclinic, space group P-1 with $a = 9.4646(9)$, $b = 12.944(3)$, $c = 13.182(3)$ Å, $\alpha = 109.57(3)$, $\beta = 107.78(3)$, $\gamma = 97.61(3)^\circ$, $V = 1398.5(5)$ Å^3, $Z = 4$, $F(000) = 892$, $D_c = 2.189$ g/cm^3, $\mu = 3.706$ mm^{-1}, the final $R = 0.0440$, and $wR = 0.0913$. A total of 14,299 reflections were collected, of which 6539 were independent ($R_{int} = 0.0789$). In the crystal packing diagram, the van der Waals' interactions stabilize the solid state.

Keywords Carbonyl; Cobalt; crystal structure; trinuclear

Introduction

In recent years, metal carbonyl complexes have attracted great interest due to their potential applications in a great variety of research fields, for example, diiron carbonyl complexes [1–5], triiron complexes [6], and dicobalt carbonyl complexes [7]. The trinuclear cobalt carbonyl complex $SCo_3(CO)_9$ was previously synthesized by the accidental reaction of dicobalt octacarbonyl with phenyl mercaptan [8]. However, we prepared the complex $SCo_3(CO)_9$ by the reaction of $Co_2(CO)_8$ with $(\mu\text{-}S_2)Fe_2(CO)_6$ as a side product [9] and its structure was characterized by X-ray crystallography.

Experimental

Crystal Structure Determination

The crystal of the title complex with dimensions of 0.20 mm ×0.14 mm × 0.10 mm was mounted on a Rigaku Saturn CCD area detector diffractometer with a graphite-monochromated Mo$K\alpha$ radiation ($\lambda = 0.71073$Å) by using a phi and scan modes at 173(2) K in the range of $1.73^\circ \leq \theta \leq 27.99^\circ$. The crystal belongs to Monoclinic system with space group P-1 and crystal parameters of $a = 9.4646(19)$ Å, $b = 12.944(3)$ Å, $c = 13.182(3)$ Å, $\alpha = 109.57(3)^\circ$, $\beta = 107.78(3)^\circ$, $\gamma = 97.61(3)^\circ$, $V = 1398.5(5)$ A^3, $D_c = 2.189$ g/cm^3, The absorption coefficient $\mu = 3.706$ mm^{-1}, and $Z = 4$. A summary of crystal data is presented in Table 1.

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Table 1. Crystal data and structure refinement

Empirical formula	$C_9Co_3O_9S$
Formula weight	460.94
Crystal system	Triclinic
Unit cell dimensions	
a (Å)	9.4646(19)
b (Å)	12.944(3)
c (Å)	13.182(3)
Unit cell angles (°)	
α	109.57(3)
β	107.78(3)
γ	97.61(3)
Volume (Å^3)	1398.5(5)
Z	4
Temperature (K)	173(2)
Space group	P-1
Wavelength (Å)	0.71073
Calculated density (g/cm^3)	2.189
Absorption coefficient (mm^{-1})	3.706
F(000)	892
Crystal size (mm)	0.20 × 0.14 × 0.10
Theta range for data collection (°)	1.73–27.99
Reflections collected	14299
Independent reflections	6539 [$R_{(int)} = 0.0789$]
Final *R* indices [$I > 2\sigma(I)$]	$R_1 = 0.0440$, $wR_2 = 0.0913$

The structure was solved by direct methods with SHELXS-97 [10] and refined by the full-matrix least squares method on F^2 data using SHELXL-97 [11]. The empirical absorption corrections were applied to all intensity data. H atom of N-H was initially located in a difference Fourier map and were refined with the restraint Uiso(H) = 1.2 Ueq(N). Other H atoms were positioned geometrically and refined using a riding model, with d(C—H) = 0.93–0.97 Å and Uiso(H) = 1.2 Ueq(C) or 1.5 Ueq (Cmethyl). The final full-matrix least squares refinement gave $R = 0.0440$ and $wR = 0.0913$.

Results and Discussion

The title complex $SCo_3(CO)_9$ was confirmed by single crystal X-ray diffraction analysis. The selected bond lengths and bond angles listed in Tables 2–4, respectively. The structure was solved by direct methods. Anisotropic displacement parameters were applied to all nonhydrogen atoms in full-matrix least-square refinements based on F^2. The hydrogen atoms were set in calculated positions with a common fixed isotropic thermal parameter.

The molecular structure and the packing view of the title complex are shown in Figs. 1 and 2, respectively. The title complex $SCo_3(CO)_9$ crystallizes in the triclinic space group P-1. The unit cell contains four molecules and the asymmetric unit contains two molecules. As shown in Fig. 1, the molecular structure of the title complex consists of a tricobalt

Table 2. Selected bond lengths (Å) and bond angles (°)

Bond lengths			
Co(1)−C(1)	1.800(6)	Co(1)−Co(3)	2.5404(16)
Co(1)−C(2)	1.809(6)	Co(1)−Co(2)	2.5513(12)
Co(1)−C(3)	1.827(6)	Co(2)−S(1)	2.1637(16)
Co(1)−S(1)	2.1649(16)	Co(2)−Co(3)	2.5457(13)
Bond angles			
C(1)−Co(1)−C(2)	100.3(2)	S(1)−Co(1)−Co(2)	53.86(5)
C(1)−Co(1)−C(3)	101.2(3)	Co(3)−Co(1)−Co(2)	59.99(4)
C(2)−Co(1)−C(3)	99.0(3)	S(1)−Co(2)−Co(3)	53.92(5)
C(1)−Co(1)−S(1)	144.07(19)	S(1)−Co(2)−Co(1)	53.91(5)
C(1)−Co(1)−Co(3)	95.89(19)	Co(3)−Co(2)−Co(1)	59.79(4)
S(1)−Co(1)−Co(3)	54.00(5)	S(1)−Co(3)−Co(1)	54.10(5)
C(1)−Co(1)−Co(2)	96.06(17)	S(1)−Co(3)−Co(2)	53.98(5)

triangle cluster capped by a μ_3-S ligand with nine terminal carbonyls. Each cobalt atom was coordinated by three terminal carbonyls, of which two carbonyl occupying the basal position of the distorted square-pyramidal geometry of the cobalt atom and one carbonyl occupying the apical position of the above-mentioned geometry. The Co-Co bond lengths [Co1−Co2 = 2.5513(12) Å, Co1−Co3 = 2.5404(16) Å, and Co2−Co3 = 2.5457(13) Å], Co−S bond lengths [Co1−S1 = 2.1649(16) Å, Co2−S1 = 2.1637(16) Å and Co3−S1 = 2.1622(18) Å], Co−Co−Co bond angles [Co3−Co1−Co2 = 59.99(4)°, Co3−Co2−Co1 = 59.79°, and Co1−Co3−Co2 = 60.22°] and S−Co−Co bond angles [S1−Co1−Co2 = 53.86(5)°, S1−Co2−Co1 = 53.91(5)°, S1−Co1−Co3 = 53.86(5)°, S1−Co1−Co3 = 54.00(5)°, S1−Co2−Co3 = 53.92(5)°, S1−Co3−Co1 = 54.10(5)°, and S1−Co3−Co2 = 53.98(5)°] are comparable to the corresponding tricobalt complexes [12].

As shown in Fig. 2, there are no hydrogen bonds in the crystal packing diagram of the title complex. The van der Waals' interactions stabilize the solid state of the title complex.

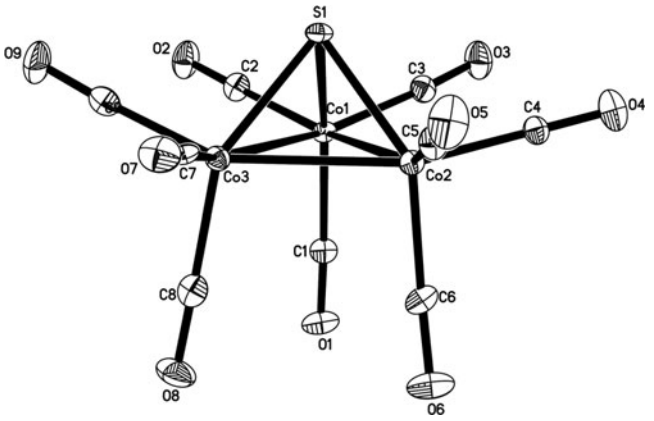

Figure 1. Molecular structure of the title complex.

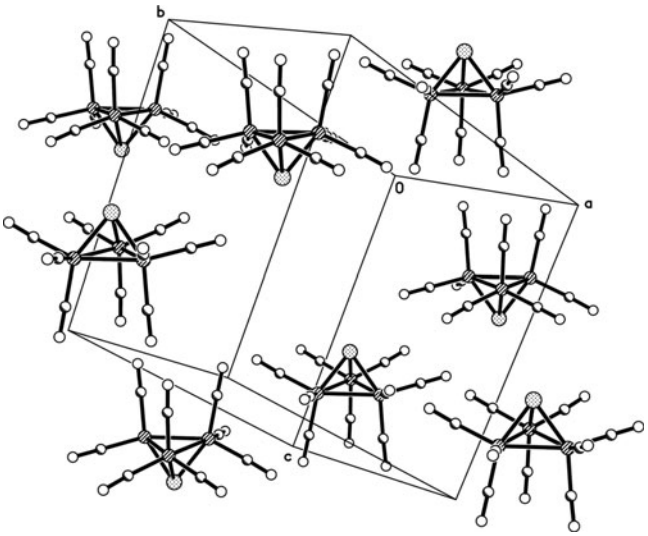

Figure 2. The crystal packing view of the title complex.

Conclusions

In summary, the title complex $SCo_3(CO)_9$ has been prepared and structurally characterized by X-ray crystallography.

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Supplemental Materials

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication number CCDC 1011036 for the title compound. Copies of the data can be obtained free of charge at http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223–336–033; e-mail: deposit@ccdc.cam.ac.uk.

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