Research Article

Crystallization and Structural Characterization of Dimeric and Trimeric Forms of (Neocuproine) $\text{CdFe}(\text{CO})_4$

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The previously reported (neocuproine) $\text{CdFe}(\text{CO})_4$ has been found to crystallize as either a dimer or a trimer depending on the procedure used. These species exist as nearly planar four- and six-membered rings composed of alternating cadmium and iron atoms. The trimeric form is reasonably similar to its 2,2'-bipyridyl analogue, while the dimeric form had not been observed previously for a $\text{L}_x\text{CdFe}(\text{CO})_4$ complex. The space group for the dimeric isomer is $C2/c$, with $a = 20.7044(3)$, $b = 12.2169(2)$, $c = 13.7837(2)$ Å, and $\beta = 98.1226(11)^\circ$, while for the trimeric isomer the space group is $P1$, with $a = 14.5056(1)$, $b = 15.4331(1)$, $c = 17.3372(2)$ Å, $\alpha = 110.0059(3)^\circ$, $\beta = 104.2580(4)^\circ$, and $\gamma = 95.2796(5)^\circ$.

1. Introduction

The complex $\text{CdFe}(\text{CO})_4$ (1) was first reported in 1933 and due to its insolubility was proposed to be a polymeric species [1, 2]. A subsequent study revealed it to be tetrameric, composed of a square, eight-membered ring having alternating cadmium and iron atoms, bearing nearly linear and cis-disubstituted octahedral coordination geometries, respectively [3]. In contrast to its mercury analogue [4], 1 has been found to readily incorporate ligands into its coordination sphere, generating a variety of $[\text{L}_x\text{CdFe}(\text{CO})_4]_n$ complexes (e.g., $\text{L} = \text{NH}_3$ or py , $x = 2$; $\text{L}_2 = 2,2'$ -bipyridine or 1,10-phenanthroline [2]). As can be seen, these most commonly involve two coordination sites on cadmium being occupied by amines, though THF coordination is also possible, and at least one $[\text{LCdFe}(\text{CO})]_n$ species ($\text{L} = 4$ -phenylpyridine) [5] and at least one monomeric $\text{L}_3\text{CdFe}(\text{CO})_4$ complex, in which L_3 represents a combination of one pyridine and one bidentate neocuproine (2,9-dimethylphenanthroline) ligand [6], are known. The isolation of the monomeric species appears to have been promoted by the use of pyridine as the solvent for crystallization. Further, the use of the chelating tren ligand appears, from spectroscopic data, also to have led to a monomeric complex [5]. In comparison, analogous

complexes with the harder zinc ion are typically found as monomeric $\text{L}_3\text{ZnFe}(\text{CO})_4$ species ($\text{L} = \text{NH}_3$ or pyridine [2] or $\text{L}_3 = (\text{NH}_3)(2,2'\text{-bpy})$ [5, 6]). An unusual dianionic species, $\{[\text{CdFe}(\text{CO})_4]_6\text{Cl}_2(\text{DME})_2\}^{2-}$, has also been reported [7] and found to contain a twelve-membered ring, thus far the largest known. The use of larger iron carbonyl anions, such as $\text{Fe}_2(\text{CO})_8^{2-}$ or $\text{Fe}_3(\text{CO})_{11}^{2-}$, has thus far not led to oligomers but instead to species possessing CdFe_2 rings [8, 9].

To date, the only $[\text{L}_2\text{CdFe}(\text{CO})_4]_n$ species to have been crystallographically characterized have been the $\text{L} = \text{THF}$ or pyridine (py) or $\text{L}_2 = 2,2'$ -bipyridine species [10], which have all been found to exist as trimeric complexes. Notably, the (2,2'-bipyridine)zinc analogue (2) has been found to exist as a dimer [11]. The general structures of these arrangements may be seen in Figure 1. In order to attempt to gain a better understanding of the factors that control the extent of oligomerization in these complexes, we have pursued the characterization of additional complexes which incorporate more highly modified ligands, such as neocuproine. While $[(\text{neocuproine})\text{CdFe}(\text{CO})_4]_n$ has indeed been synthesized [6], its degree of association had not yet been determined, though a structural study of the monomeric $(\text{py})(\text{neocuproine})\text{CdFe}(\text{CO})_4$ complex was carried out. We

FIGURE 1: General structures of $L_2CdFe(CO)_4$ dimers (a) and trimers (b).

now report that the use of neocuproine allows for the isolation of both the dimeric and the trimeric isomers.

2. Materials and Methods

All reactions were carried out in a Schlenk apparatus under a nitrogen atmosphere.

Due to the low solubility of the neocuproine complex(es), it appeared that it might be most effective to attempt crystallization of the complex as it was formed. In order to moderate the rate of formation, it seemed advisable to use a reasonably soluble $[L_2CdFe(CO)_4]_n$ complex as starting material, particularly with L_2 being a chelating ligand. While ethylenediamine might have seemed suitable, the low solubility of $[(en)CdFe(CO)_4]_n$ [2], as well as its tetramethyl analogue, led us to utilize tetraethylethylenediamine as the chelate. The teeda complex can be easily prepared by the reaction of stoichiometric teeda with $[(NH_3)_2CdFe(CO)_4]_n$ in THF, after which a concentrated solution crystallizes very nicely on cooling to -30°C . From the $[(teeda)CdFe(CO)_4]_n$ starting material, an apparently lighter and a darker form of crystalline neocuproine complex could be isolated. It was subsequently found that the formation of the paler form could be favored by rapid mixing of the starting materials. As a general example of its isolation, approximately 0.20 g of $[(teeda)CdFe(CO)_4]_n$ was dissolved in 20 mL of THF in a Schlenk flask under nitrogen, and 0.094 g of neocuproine was added. After shaking the mixture to ensure complete dissolution, the flask was set aside in a still location. Within several hours some well-formed red crystals had deposited. In contrast, formation of the darker isomer was favored through reactions involving slower contact between the two reactants. Thus, a small amount of $[(teeda)CdFe(CO)_4]_n$ was placed in a Schlenk tube under nitrogen, and a 1-2 cm long test tube (1 cm or less diameter) with some solid neocuproine in the bottom, covered carefully with liquid THF, was placed therein. THF was then carefully added to the Schlenk tube, attempting to minimize initial dissolution of the cadmium starting material, until a level was reached about 1 cm above the top of the test tube. Within 2-3 weeks some nice dark red prisms had formed.

Single crystals of each compound were mounted on glass fibers with Paratone oil and transferred to a Nonius Kappa

CCD diffractometer. The dimer and the trimer were found to have monoclinic and triclinic unit cells (Table 1), and their space groups were determined from systematic absences and the successful solutions of the structures. Data collection and processing utilized the programs COLLECT, DENZO-SMN, and SCALEPAC [12]. The structures were solved using heavy atom and direct methods with SIR97 [13] and SHELXL97 [14]. Final refinements were carried out with anisotropic thermal parameters for all nonhydrogen atoms. The non-methyl hydrogen atoms in the dimer were successfully refined isotropically, while all other hydrogen atoms were given isotropic thermal parameters and were allowed to ride on their attached carbon atoms. Four molecules of THF were also found in the lattice of the trimer, one of which suffered a severe disorder. Scattering factors were taken from the literature [15, 16].

Molecular plots were obtained using the ORTEP3 for Windows program [17].

The CCDC depositions 967908 and 967909 contain the full crystallographic information for the dimeric and trimeric structures, respectively. These data can be obtained free of charge via <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 or e-mail: deposit@ccdc.cam.ac.uk.

3. Results and Discussion

It is first of interest to try to explain how the differing crystallization schemes could favor different isomeric forms of $[(neocuproine)CdFe(CO)_4]_n$ ($n = 2$ (3) or 3 (4)). One possibility is that the difference is driven by the local concentrations of the starting material $[(teeda)CdFe(CO)_4]_n$ and/or a monomeric $(THF)(teeda)CdFe(CO)_4$ complex. In the case in which both reactants are mixed thoroughly from the start, there will be a higher local concentration of metal complex, so that as two monomers link, but prior to closing to a ring, it will not be long before a third is encountered, leading to the selective formation of the trimer. In the slower mixing process, an incipient but not yet cyclized dimer will not as quickly encounter a third metal complex and thus may ultimately cyclize to the dimer, despite its substantial steric strain (*vide infra*).

TABLE 1: Crystal and experimental data.

	$C_{36}H_{24}Cd_2Fe_2N_4O_8$	Empirical formula $C_{70}H_{68}Cd_3Fe_3N_6O_{16}$
Formula weight	977.09	1754.05
Temperature (K)	150(1)	150(1)
Wavelength (Å)	0.71073	0.71073
Crystal system	Monoclinic	Triclinic
Space group	$C2/c$	$P\bar{1}$
Unit cell dimensions		
<i>a</i>	20.7044(3)	14.5056(1)
<i>b</i>	12.2169(2)	15.4331(1)
<i>c</i>	13.7837(2)	17.3372(2)
α (°)	90	110.0059(3)
β (°)	98.1226(11)	104.2580(4)
γ (°)	90	95.2796(5)
Volume (Å ³)	3451.52(9)	3467.83(5)
<i>Z</i>	4	2
D_{calcd} (g cm ⁻³)	1.880	1.680
$\mu(\text{MoK}\alpha)$ (mm ⁻¹)	2.098	1.586
$F(000)$	1,920	1,760
θ range (°)	2.53–27.48	2.28–27.87
	$-26 \leq h \leq 26$	$-17 \leq h \leq 19$
Limiting indices	$-15 \leq k \leq 15$	$-20 \leq k \leq 20$
	$-17 \leq l \leq 17$	$-22 \leq l \leq 22$
Reflections collected/unique	7456/3951	30626/16438
Completeness (%)	99.7	99.4
Goodness of fit on F^2	1.053	1.029
<i>R</i> indices [$I > 2\sigma(I)$]	$R_1 = 0.0227, wR_2 = 0.0581$	$R_1 = 0.0248, wR_2 = 0.0587$
<i>R</i> indices (all data)	$R_1 = 0.0289, wR_2 = 0.0609$	$R_1 = 0.0308, wR_2 = 0.0609$
$(\Delta\rho)_{\text{max, min}}$	0.710, -0.611	0.753, -0.674
<i>R</i> (int)	0.0131	0.0166

FIGURE 2: The structure of $[(\text{neocuproine})\text{CdFe}(\text{CO})_4]_2$ (30% probability ellipsoids).

The dimeric isomer **3** displays a structure (Figure 2) which is fairly similar to the related $[(2,2'\text{-bipyridine})\text{ZnFe}(\text{CO})_4]_2$, **2**. As opposed to complex **2**, which is situated on an inversion center and as a result is rigorously planar, **3** has imposed twofold rotational symmetry and is thus

not quite planar, with opposing deviations of 0.042 Å for the cadmium and iron centers. One can next notice that the “axial” carbonyl ligands on iron exhibit significant tilts, such that the C3–Fe–C4 angle is 146.59(10)°, as compared to 150.05(14)° for **2** (Table 2). This tilt is attributed to

TABLE 2: Selected bond lengths (Å) and angles (°) for [(neocuproine)CdFe(CO)₄]₂.

Bond distances			
Cd1–Fe1	2.6532(3)	Cd1–Fe1'	2.8125(3)
Cd1–N1	2.3635(17)	Cd1–N2	2.3621(17)
Fe1–C1	1.780(2)	C1–O1	1.154(3)
Fe1–C2	1.770(2)	C2–O2	1.146(3)
Fe1–C3	1.775(2)	C3–O3	1.163(3)
Fe1–C4	1.772(2)	C4–O4	1.165(3)
Bond angles			
Fe1–Cd1–Fe1'	111.243(9)	Cd1–Fe2–Cd1'	68.642(9)
C1–Fe1–C2	101.75(11)	Fe1–C1–O1	176.3(2)
C1–Fe1–C3	103.96(10)	Fe1–C2–O2	178.2(2)
C1–Fe1–C4	105.24(10)	Fe1–C3–O3	173.5(2)
C2–Fe1–C3	95.81(9)	Fe1–C4–O4	172.4(2)
C2–Fe1–C4	93.83(9)	Cd1–Fe1–C1	81.01(7)
C3–Fe1–C4	146.59(10)	Cd1'–Fe1–C2	108.65(8)
N1–Cd1–N2	71.36(6)		

a contribution from an ionic resonance hybrid involving coordinated Cd²⁺ and the Fe(CO)₄^{2−} ions. This angle is very close to being midway between the octahedral and face-bicapped tetrahedral extremes, as is also the C1–Fe–C2 angle of 101.75(11)° (103.25(16)° for **2**). These tilts lead to a close approach between the axial carbonyls on one iron center and those of the other, which then results in a twist of the tetrahedra toward opposite cadmium centers. Even so, the O3–O3' and O4–O4' contacts are still rather close at 3.327 and 3.788 Å, with shorter O3–C17', O4–C18', and Cd–Cd' contacts of 3.190, 3.148, and 3.085 Å. One way of gauging this distortion is to compare the Cd1–Fe1–C1 and Cd1'–Fe1–C2 angles of 81.01(7)° and 108.65(8)°, reflecting by this measure an approximate twist of 14° (84.40(14)° and 106.42(9)° for **2**). The twist also causes each cadmium to adopt something closer to an edge- rather than face-capping position on one of the adjacent iron centers. In fact, while face-capped tetrahedral resonance forms can contribute substantially to the overall makeup of these compounds, in this case the structure actually appears to resemble an equatorial edge-capped trigonal bipyramidal arrangement (Figure 3).

The respective Cd1–Fe1–Cd1' and Fe1–Cd1–Fe1' angles of 68.642(9)° and 111.243(9)° also compare well with the values of 66.02(2)° and 113.98(2)° for **2**. However, other parameters differ markedly. While a modest difference in Zn–Fe bond distances was observed in **2** (2.585(1) and 2.532(1) Å), the Cd1–Fe1 and Cd1–Fe1' bond lengths were observed to be 2.6532(3) and 2.8125(3) Å. Additionally, there is a greater nonlinearity of the axial carbonyl ligands, with the Fe1–C3–O3 and Fe1–C4–O4 angles being 173.5(2)° and 172.4(2)° (versus 176.3(3)° and 173.9(3)° in **2**). These enhanced distortions can most likely be attributed to contacts between the axial carbonyl oxygen atoms and methyl groups of the neocuproine ligand.

Overall, the trimeric isomer (Figure 4) exhibits far less distortion compared to the dimer. This can first be seen

TABLE 3: Selected bond lengths (Å) and angles (°) for [(neocuproine)CdFe(CO)₄]₃.

Bond distances			
Cd1–Fe2	2.7258(3)	Cd1–Fe3	2.7307(3)
Cd2–Fe1	2.6969(3)	Cd2–Fe3	2.7287(3)
Cd3–Fe1	2.7307(3)	Cd3–Fe2	2.7251(3)
Fe1–C1	1.777(2)	C1–O1	1.149(3)
Fe1–C2	1.780(2)	C2–O2	1.152(3)
Fe1–C3	1.774(2)	C3–O3	1.162(3)
Fe1–C4	1.777(2)	C4–O4	1.159(3)
Fe2–C5	1.766(2)	C5–O5	1.155(3)
Fe2–C6	1.767(2)	C6–O6	1.156(3)
Fe2–C7	1.779(2)	C7–O7	1.160(3)
Fe2–C8	1.781(2)	C8–O8	1.159(3)
Fe3–C9	1.774(2)	C9–O9	1.154(3)
Fe3–C10	1.772(3)	C10–O10	1.151(3)
Fe3–C11	1.776(2)	C11–O11	1.153(3)
Fe3–C12	1.768(2)	C12–O12	1.165(3)
Cd1–N1	2.3814(16)	Cd1–N2	2.4616(17)
Cd2–N3	2.3901(19)	Cd2–N4	2.4395(18)
Cd3–N5	2.4967(17)	Cd3–N6	2.4301(17)
Bond angles			
Fe1–Cd2–Fe3	133.732(10)	C1–Fe1–C2	94.54(11)
Fe1–Cd3–Fe2	137.829(10)	C1–Fe1–C3	102.61(11)
Fe2–Cd1–Fe3	138.272(10)	C1–Fe1–C4	100.21(11)
Cd1–Fe2–Cd3	95.439(10)	C2–Fe1–C3	104.13(10)
Cd1–Fe3–Cd2	99.481(10)	C2–Fe1–C4	101.90(10)
Cd2–Fe1–Cd3	101.855(10)	C3–Fe1–C4	143.59(10)
Fe1–C1–O1	176.1(2)	C5–Fe2–C6	98.01(10)
Fe1–C2–O2	175.9(2)	C5–Fe2–C7	104.93(11)
Fe1–C3–O3	174.7(2)	C5–Fe2–C8	97.20(11)
Fe1–C4–O4	172.6(2)	C6–Fe2–C7	95.15(10)
Fe2–C5–O5	175.0(2)	C6–Fe2–C8	97.19(10)
Fe2–C6–O6	177.4(2)	C7–Fe2–C8	152.84(10)
Fe2–C7–O7	170.1(2)	C9–Fe3–C10	95.00(11)
Fe2–C8–O8	169.4(2)	C9–Fe3–C11	101.71(12)
Fe3–C9–O9	175.6(2)	C9–Fe3–C12	102.79(10)
Fe3–C10–O10	175.8(2)	C10–Fe3–C11	98.78(13)
Fe3–C11–O11	172.2(2)	C10–Fe3–C12	106.88(11)
Fe3–C12–O12	173.8(2)	C11–Fe3–C12	142.47(10)
N1–Cd1–N2	70.10(6)	N3–Cd2–N4	70.51(7)
N5–Cd3–N6	68.54(6)		

by the relatively narrow range of Cd–Fe bonds, 2.6969(3)–2.7307(3) Å. In addition, there is a much lesser degree of tetrahedral twisting. Only Fe2 exhibits a notable twist, as evidenced by the Cd1–Fe2–C6 and Cd3–Fe2–C5 angles of 89.98(7)° and 76.87(7)°. There are, however, other distortions that are greater for the trimer. The ring is significantly nonplanar, as opposed to the trimeric isomers containing THF, pyridine [10], or bipyridine [11]. Thus, the deviations for Cd(1–3) and Fe(1–3) from planarity are −0.37, −0.02, 0.33,

FIGURE 3: Alternative view of $[(\text{neocuproine})\text{CdFe}(\text{CO})_4]_2$ (30% probability ellipsoids).

FIGURE 4: The structure of $[(\text{neocuproine})\text{CdFe}(\text{CO})_4]_3$ (30% probability ellipsoids).

−0.26, 0.01, and 0.31 Å. Additionally, the axial Fe–C–O angles are slightly smaller, ranging from 169.4(2)° to 174.7(2)°.

Some additional comparisons may be made with [(2,2'-bipyridine)CdFe(CO)₄]₃, **5**. The Cd–N distances range from 2.3814(16) to 2.4967(17) Å, as compared to an average value of 2.37(1) Å for **5**, while the N–Cd–N angles range from 68.54(6) to 70.51(7)°, versus an average of 68.9(4)° for **5**. The longer distances may reasonably be attributed to enhanced steric interactions brought about by the methyl substituents on the cuproin ligands. The Cd–Fe–Cd and Fe–Cd–Fe angles range from 95.439(10)° to 101.855(10)° and 133.732(10)° to 138.272(10)°, while the corresponding ranges for **5** were 94.78(14)°–102.31(15)° and 138.61(14)°–148.40(15)°.

The trimeric isomer **4** has much less severe Cd–Cd contacts as compared to the dimer **3**. The shortest for **4** is 4.033 Å (Cd1/Cd3), nearly 1 Å longer than the 3.085 Å in **3**. On the other hand, **3** has two interactions between carbonyl oxygen atoms (O3/O7, 2.975 Å; O8/O12, 2.939 Å) that are significantly shorter than those in the dimer (*vide supra*). Furthermore, while **3** has carbonyl oxygen/methyl contacts of 3.190 and 3.148 Å, **4** has not only such contacts but also shorter ones of 3.028 (O7/C25), 3.031 (O4/C54), and 3.122 (O8/C26) Å. One also observes a marked asymmetry in the Cd–N bonds for the trimer (Table 3) but not for the dimer.

4. Conclusions

Through variation of the crystallization method, it has been found possible to isolate both dimeric and trimeric forms of (neocuproine)CdFe(CO)₄. Trimeric cadmium iron complexes already had a precedent in other complexes with THF and amine ligands, while the dimeric structure had not previously been observed. Due to the methyl groups on the neocuproine ligands, these species exhibited substantially greater distortions as compared to the related [(2,2'-bipyridine)ZnFe(CO)₄]₂ and [(2,2'-bipyridine)CdFe(CO)₄]₃ complexes.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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