

**Study on Isolobal Reactions in Transition Metal Cluster Systems. Synthesis of Heteronuclear Metal Cluster Complexes  $(\eta^5\text{-C}_5\text{H}_5)(\eta^5\text{-RC}_5\text{H}_4)\text{MoNiFe}_2\text{S}(\text{CO})_{10}$ ,  $[(\eta^5\text{-C}_5\text{H}_5)\text{MoNiFe}_2\text{S}(\text{CO})_{10}]_2[\eta^5\text{-C}_5\text{H}_4\text{C}(\text{O})\text{CH}_2]_2$ ,  $[(\eta^5\text{-C}_5\text{H}_5)\text{MoNiFeS}(\text{CO})_5][\text{MoCoFeS}(\text{CO})_8][\eta^5\text{-C}_5\text{H}_4\text{C}(\text{O})\text{CH}_2]_2$ , and  $[(\eta^5\text{-C}_5\text{H}_5)\text{MoNiFeS}(\text{CO})_5]_2[\eta^5\text{-C}_5\text{H}_4\text{C}(\text{O})\text{CH}_2]_2$**

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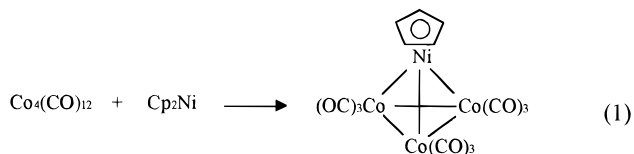
Received July 8, 1997<sup>Ⓢ</sup>

Reactions of the single-tetrahedral MoNiFeS clusters  $(\eta^5\text{-C}_5\text{H}_5)(\eta^5\text{-RC}_5\text{H}_4)\text{MoNiFeS}(\text{CO})_5$  [ $\text{R} = \text{H}$  (**1**),  $\text{MeCO}$  (**2**),  $\text{MeO}_2\text{C}$  (**3**)] with  $\text{Fe}_2(\text{CO})_9$  in toluene at 45–50 °C gave an isolobal displacement/addition type of products  $(\eta^5\text{-C}_5\text{H}_5)(\eta^5\text{-RC}_5\text{H}_4)\text{MoNiFe}_2\text{S}(\text{CO})_{10}$  [ $\text{R} = \text{H}$  (**4**),  $\text{MeCO}$  (**5**),  $\text{MeO}_2\text{C}$  (**6**)], whereas the double-tetrahedral MoNiFeS cluster  $[(\eta^5\text{-C}_5\text{H}_5)\text{MoNiFeS}(\text{CO})_5]_2[\eta^5\text{-C}_5\text{H}_4\text{C}(\text{O})\text{CH}_2]_2$  (**7**) reacted with  $\text{Fe}_2(\text{CO})_9$  under similar conditions to give the same type of reaction product  $[(\eta^5\text{-C}_5\text{H}_5)\text{MoNiFe}_2\text{S}(\text{CO})_{10}]_2[\eta^5\text{-C}_5\text{H}_4\text{C}(\text{O})\text{CH}_2]_2$  (**8**). In contrast to the chemical behavior of such tetrahedral clusters, the tetrahedral MoCoFeS cluster  $[\text{MoCoFeS}(\text{CO})_8]_2[\eta^5\text{-C}_5\text{H}_4\text{C}(\text{O})\text{CH}_2]_2$  (**9**) reacted with  $\text{Cp}_2\text{Ni}$  in refluxing THF to afford only the isolobal displacement type of products  $[(\eta^5\text{-C}_5\text{H}_5)\text{MoNiFeS}(\text{CO})_5]_2[\eta^5\text{-C}_5\text{H}_4\text{C}(\text{O})\text{CH}_2]_2$  (**7**) and  $[(\eta^5\text{-C}_5\text{H}_5)\text{MoNiFeS}(\text{CO})_5][\text{MoCoFeS}(\text{CO})_8][\eta^5\text{-C}_5\text{H}_4\text{C}(\text{O})\text{CH}_2]_2$  (**10**). The X-ray molecular structures of **5** and **7** are described, and a possible pathway for the novel reaction yielding the isolobal displacement/addition type of products has been briefly discussed.

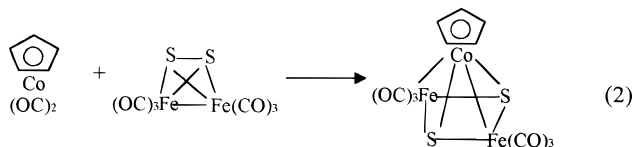
### Introduction

Since the principle of “isolobal analogy” was put forward in 1982,<sup>1</sup> the isolobal reactions, which include isolobal displacement and isolobal addition, have been intensively studied and widely used in the rational synthesis of heteronuclear transition metal clusters.<sup>2–12</sup> It is well-known that for the isolobal displacement reaction the metal nuclearity of the product is equal to that of the displaced cluster substrate (such as in the

formation of  $\text{CpNi}[\text{Co}(\text{CO})_3]_4$  (eq 1)], whereas for the



isolobal addition reaction the metal nuclearity of the product is invariably equal to sum of the metal atoms of two isolobal fragments (such as in the formation of  $\text{CpCo}(\mu_3\text{-S})_2\text{Fe}_2(\text{CO})_6$ <sup>8</sup> (eq 2)). In addition, the two types of isolobal reactions are known to proceed always separately, and no case has been found for them to occur concomitantly.



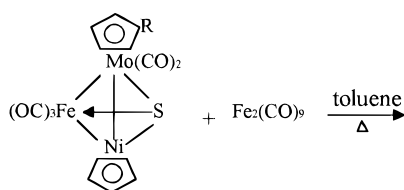
However, during the course of our study on isolobal reactions we found that the isolobal displacement reactions between some tetrahedral MoNiFeS clusters with  $\text{Fe}_2(\text{CO})_9$  took place concurrently with an isolobal addition reaction to afford nuclearity-increased novel MoNiFe<sub>2</sub>S clusters. We also found that the isolobal displacement reaction of the double-tetrahedral MoCoFeS cluster with  $\text{Cp}_2\text{Ni}$  gave two metal-nuclearity-unchanged double-cluster products, one with tetrahedral MoCoFeS and MoNiFeS cluster cores being derived from a single-displacement reaction and the other with

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<sup>Ⓢ</sup> Abstract published in *Advance ACS Abstracts*, September 1, 1997.  
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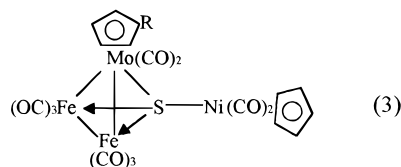
two double MoNiFeS cluster cores derived from a double-displacement reaction. Now we report such two major findings concerning the isolobal reactions and describe the synthesis of a series of novel cluster complexes, as well as two crystal structures for one single cluster  $(\eta^5\text{-C}_5\text{H}_5)(\eta^5\text{-MeCOC}_5\text{H}_4)[\text{MoNiFe}_2\text{S}(\text{CO})_{10}]$  and one double cluster  $[\text{MoNiFeS}(\text{CO})_5]_2[\eta^5\text{-C}_5\text{H}_4\text{C}(\text{O})\text{-CH}_2]_2$ .

## Results and Discussion

**Part 1. Isolobal Reactions of Clusters 1–3 and 7 with  $\text{Fe}_2(\text{CO})_9$ . Synthesis and Characterization of Clusters 4–6 and 8.** We found that the single-tetrahedral MoNiFeS clusters  $(\eta^5\text{-C}_5\text{H}_5)(\eta^5\text{-RC}_5\text{H}_4)\text{MoNiFeS}(\text{CO})_5$  [R = H (**1**), MeCO (**2**), MeO<sub>2</sub>C (**3**)] reacted with excess  $\text{Fe}_2(\text{CO})_9$  in toluene at 45–50 °C to afford the single MoNiFe<sub>2</sub>S clusters  $(\eta^5\text{-C}_5\text{H}_5)(\eta^5\text{-RC}_5\text{H}_4)\text{-MoNiFe}_2\text{S}(\text{CO})_{10}$  [R = H (**4**), MeCO (**5**), MeO<sub>2</sub>C (**6**)] (eq 3).

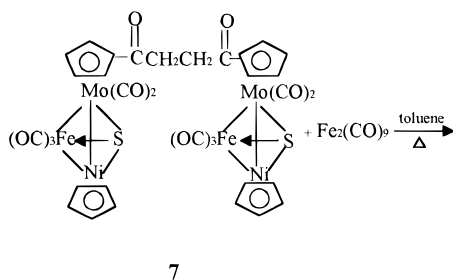


1 R=H; 2 R=MeCO; 3 R=MeO<sub>2</sub>C

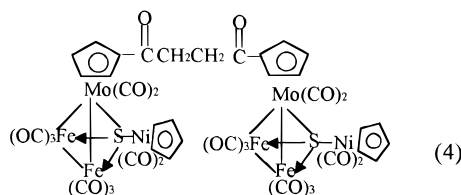


4 R=H; 5 R=MeCO; 6 R=MeO<sub>2</sub>C

We further found that the double-tetrahedral MoNiFeS cluster  $[(\eta^5\text{-C}_5\text{H}_5)\text{MoNiFeS}(\text{CO})_5]_2[\eta^5\text{-C}_5\text{H}_4\text{C}(\text{O})\text{-CH}_2]_2$  (**7**) could similarly react with excess  $\text{Fe}_2(\text{CO})_9$  to give the double MoNiFe<sub>2</sub>S cluster  $[(\eta^5\text{-C}_5\text{H}_5)\text{MoNiFe}_2\text{S}(\text{CO})_{10}]_2[\eta^5\text{-C}_5\text{H}_4\text{C}(\text{O})\text{CH}_2]_2$  (**8**) (eq 4).

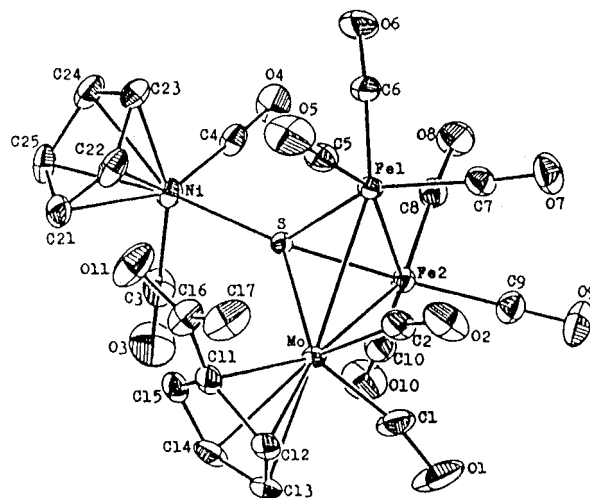


7



8

While starting clusters **1** and **2** were previously reported,<sup>13</sup> clusters **3–8** are new and have been char-



**Figure 1.** Molecular structure of **5** with atomic numbering scheme.

**Table 1.** Selected Bond Lengths (Å) and Bond Angles (deg) for **5**

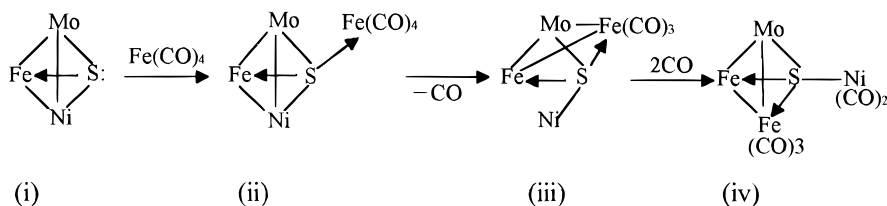
Mo–Fe(1)	2.8372(8)	Mo–Fe(2)	2.845(1)
Fe(1)–Fe(2)	2.614(1)	Mo–C(1)	1.938(6)
Mo–S	2.350(1)	Ni–C(4)	1.789(6)
Ni–S	2.264(1)	Fe(1)–C(5)	1.761(6)
Fe(1)–S	2.161(1)	Fe(2)–C(8)	1.762(6)
Fe(2)–S	2.165(1)		
Fe(1)–Mo–Fe(2)	54.78(2)	Fe(2)–S–Mo	78.00(4)
Fe(1)–Fe(2)–Mo	62.45(3)	C(1)–Mo–S	106.0(2)
C(1)–Mo–Fe(1)	107.1(2)	C(21)–Ni–S	114.2(2)
S–Mo–Fe(1)	48.12(3)	S–Mo–Fe(2)	48.10(3)
S–Fe(1)–Fe(2)	52.89(4)	S–Fe(1)–Mo	54.06(3)
S–Fe(2)–Fe(1)	52.75(4)	S–Fe(2)–Mo	53.90(3)
Fe(1)–S–Fe(2)	74.36(4)	Fe(1)–S–Ni	132.70(6)
Fe(1)–S–Mo	77.81(4)	Fe(2)–S–Ni	132.08(6)
Ni–S–Mo	137.45(5)		

acterized by elemental and spectroscopic analyses (for discussion of **7**, see Part 2). For example, the <sup>1</sup>H NMR spectrum of **3** showed one singlet at  $\delta$  3.86 for the methoxycarbonyl substituent, one singlet at  $\delta$  5.28 for the Cp ring coordinated to the Ni atom, and two multiplets at  $\delta$  5.40–6.20 for the substituted cyclopentadienyl ligand. The <sup>1</sup>H NMR spectrum of **4** showed one singlet at  $\delta$  5.34 for the Cp ring coordinated to the Ni atom and another singlet at  $\delta$  5.42 for the Cp ring coordinated to the Mo atom, whereas the <sup>1</sup>H NMR spectra of **5** and **6** exhibited one singlet at  $\delta$  2.40 and 3.82 for acetyl and methoxycarbonyl substituents, one singlet at  $\delta$  5.36 and 5.30 for the Cp ring coordinated to the Ni atom, and two apparent triplets at  $\delta$  5.3–5.8 for the substituted cyclopentadienyl ligands. The IR spectra of **3–6** indicated four to six strong absorption bands for terminal carbonyls in the range 1811–2065 cm<sup>-1</sup>, and those of **3**, **5**, and **6** showed one additional strong absorption band at 1655–1729 cm<sup>-1</sup> for the carbonyls of the acetyl group and the ester group, respectively. The IR spectrum of **8** showed four absorption bands from 1860 to 2057 cm<sup>-1</sup> for its terminal carbonyls and one band at about 1680 cm<sup>-1</sup> for its carbonyls of the succinoyl bridge. The <sup>1</sup>H NMR spectrum of **8** exhibited one singlet at  $\delta$  3.12 for the ethylene group, one singlet at  $\delta$  5.68 for the two Cp rings, and one multiplet at  $\delta$  5.8–6.5 for the two substituted cyclopentadienyl ligands.

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**Table 2. Comparison of Some Crystallographic Data between 2 and 5**

cluster	Mo-Fe (Å)	Mo-S (Å)	Fe-S (Å)	Ni-S (Å)	Mo-Cp* (Å)	Ni-Cp* (Å)	Z (deg)
<b>2</b>	2.782(1)	2.362(3)	2.177(3)	2.131(3)	2.0060	1.7149	102.88
<b>5</b>	2.8411(5)	2.350(1)	2.163(1)	2.264(1)	2.0016	1.7081	115.66

**Scheme 1**

**Crystal Structure of 5.** In order to further confirm the structures of those products yielded by the new type of reactions shown in eqs 3 and 4, a single-crystal X-ray diffraction analysis of **5** was undertaken. The molecular structure of **5** is shown in Figure 1, and selected bond lengths and angles are given in Table 1.

As seen in Figure 1, cluster **5** consists of a distorted tetrahedral MoFe<sub>2</sub>S cluster core, which carries two groups of three carbonyls coordinated to two Fe atoms respectively, one acetylcyclopentadienyl and two carbonyls bonded to the Mo atom, and one cyclopentadienyldicarbonylnickel moiety attached to a μ<sub>4</sub>-S atom. It is noteworthy that although numerous transition metal clusters containing a μ<sub>4</sub>-S<sup>14</sup> and another μ<sub>4</sub>-main group element<sup>15</sup> are known, to our knowledge, clusters containing a μ<sub>4</sub>-S coordinated to MoFe<sub>2</sub>Ni, such as clusters **4–6**, have never been reported in the literature. In Table 2 are listed some of the crystallographic data for clusters **2** and **5**. As seen from Table 2, for cluster **5** the bond lengths related to the cluster core MoNiFe<sub>2</sub>S, the distance from Mo to the centroid of the substituted cyclopentadienyl ring (Cp\*), the distance from Ni to the centroid of the cyclopentadienyl ring (Cp\*) and the dihedral angle between two cyclopentadienyl rings (denoted as Z) are basically comparable to those corresponding values of its starting cluster **2**.<sup>13</sup> The bond length of Ni-S and the dihedral angle Z for **5** are somewhat larger than those of **2**,<sup>13</sup> obviously due to the CpNi unit of **5** being outside the cluster core, and that of **2** inside the cluster core. In addition, the acetyl substituent is conjugated quite well with the planar Cp ring, so the bond length of C<sub>11</sub>-C<sub>16</sub> [1.461(9) Å] is much shorter than a normal C-C single bond and the double-bond length of C<sub>16</sub>-O<sub>11</sub> [1.211(7) Å] is much longer than a normal C=O double bond.

**Mechanistic Considerations.** Products **4–6** and **8** all contain a μ<sub>4</sub>-S atom in each tetrahedral MoNiFe<sub>2</sub>S cluster core, which behaves as a 6e ligand to coordinate with two Fe atoms via its two lone pairs of electrons, and with Mo and Ni atoms through its two single electrons, respectively. Since each of the two Fe(CO)<sub>3</sub>

fragments of these products might be viewed as getting one additional electron from the μ<sub>4</sub>-S atom, they are both d<sup>9</sup>ML<sub>3</sub> fragments<sup>1</sup> and thus isolobal to CpNi(d<sup>9</sup>ML<sub>3</sub>)<sup>1</sup> of starting clusters **1–3** and **7**. So, clusters **4–6** and **8** can be formally regarded as derived from an isolobal displacement reaction between CpNi and Fe(CO)<sub>3</sub> fragments, followed by an isolobal addition reaction of the 19e fragment Cp(CO)<sub>2</sub>Ni with the remaining tetrahedral cluster fragments of **4–6** and **8**. This combined type of isolobal reaction, to our knowledge, has never been reported so far in the literature.

At the present stage the mechanism concerning this new type of isolobal reaction remains unclear. However, since Fe<sub>2</sub>(CO)<sub>9</sub> readily disproportionates to a 16e species Fe(CO)<sub>4</sub><sup>16</sup> and the analogs of the latter, for example, the 16e Cr(CO)<sub>5</sub>, easily add to a μ<sub>3</sub>-S of the cluster SFeCo<sub>2</sub>(CO)<sub>9</sub> to form μ<sub>4</sub>-SCrFeCo<sub>2</sub>(CO)<sub>14</sub>,<sup>17</sup> the following pathway might be suggested as shown in Scheme 1. Namely, the reaction may involve initially nucleophilic attack of the μ<sub>3</sub>-S atom of the tetrahedral cluster core **i** at the Fe(CO)<sub>4</sub> species generated from Fe<sub>2</sub>(CO)<sub>9</sub> to give μ<sub>4</sub>-S adduct **ii**, followed by several steps such as loss of one CO ligand from the coordinated Fe(CO)<sub>4</sub>, cleavage of two old bonds (Mo-Ni and Fe-Ni), re-forming two new bonds (Mo-Fe and Fe-Fe), and addition of two CO ligands to the Ni atom of **iii**, to give **iv** and complete this type of reaction.

**Part 2. Isolobal Reaction of Cluster 9 with Cp<sub>2</sub>Ni. Synthesis and Characterization of 7 and 10.** Besides the isolobal reaction of the bridged double MoNiFeS cluster with Fe<sub>2</sub>(CO)<sub>9</sub> described above, we further carried out another isolobal reaction of a bridged double MoCoFeS cluster [MoCoFeS(CO)<sub>8</sub>]<sub>2</sub>[(η<sup>5</sup>-C<sub>5</sub>H<sub>4</sub>C(O)CH<sub>2</sub>)<sub>2</sub>] (**9**) with excess Cp<sub>2</sub>Ni in refluxing THF. This reaction, in contrast to the reaction shown in eq 4, gave only the isolobal Co(CO)<sub>3</sub>(d<sup>9</sup>ML<sub>3</sub>)/CpNi(d<sup>9</sup>ML<sub>3</sub>) displacement products [(η<sup>5</sup>-C<sub>5</sub>H<sub>5</sub>)MoNiFeS(CO)<sub>5</sub>]<sub>2</sub>[(η<sup>5</sup>-C<sub>5</sub>H<sub>4</sub>C(O)CH<sub>2</sub>)<sub>2</sub>] (**7**) and [(η<sup>5</sup>-C<sub>5</sub>H<sub>5</sub>)MoNiFeS(CO)<sub>5</sub>][MoCoFeS(CO)<sub>8</sub>]<sub>2</sub>[(η<sup>5</sup>-C<sub>5</sub>H<sub>4</sub>C(O)CH<sub>2</sub>)<sub>2</sub>] (**10**), the latter being a single-displacement product of the unsymmetric type and the former a double-displacement product of the symmetric type (eq 5). Such isolobal reactions of eqs 4 and 5, in which the bridged double clusters serve as isolobal substrates, to our knowledge, were not described in previous literature.

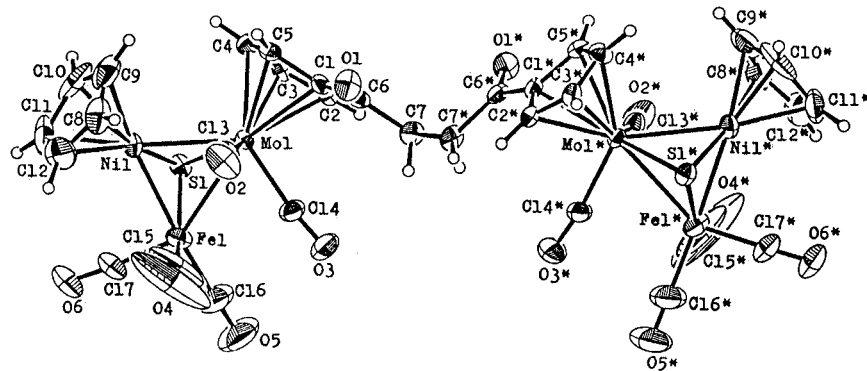
It is believed that the reaction shown in eq 5 occurred in two steps and the double-displacement product **7** was

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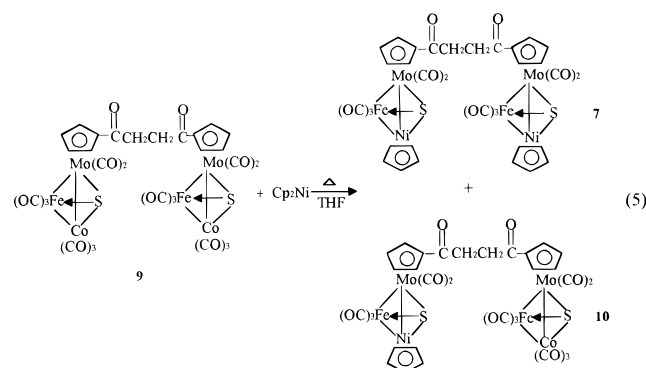
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**Figure 2.** Molecular structure of **7** with atomic numbering scheme.



formed from the single-displacement product **10**. This is because TLC showed no product **7** initially formed and the product **7** increased gradually with increasing reaction time. Clusters **7** and **10** were fully characterized by elemental analysis, IR, and  $^1\text{H}$  NMR spectroscopy, and an X-ray diffraction analysis was carried out for **7**. The IR spectra of **7** and **10** showed four strong absorption bands in the range  $1868\text{--}2073\text{ cm}^{-1}$  for terminal carbonyls and one absorption band at  $1680\text{ cm}^{-1}$  for ketonic carbonyls. The  $^1\text{H}$  NMR spectra of **7** and **10** exhibited one singlet at  $\delta$  5.22 and 5.10 for the Cp ring coordinated to the Ni atom, one singlet at  $\delta$  3.04 and 2.80 for the ethylene group, and two multiplets at  $\delta$  5.24–5.64 for  $\text{H}^3$  and  $\text{H}^4$  protons remote from the succinoyl bridge and at  $\delta$  5.72–6.12 for  $\text{H}^2$  and  $\text{H}^5$  protons close to the bridge.

**Crystal Structure of 7.** The single-crystal molecular structure of **7** is presented in Figure 2, and selected bond lengths and angles are listed in Table 3. Molecule **7** consists of two identical tetrahedral subclusters, MoNiFeS, carrying 10 carbonyls, two cyclopentadienyls, and a 1,4-bis(cyclopentadienyl)-1,4-butanedione ligand. So, the structure is very similar to that of its starting double cluster  $[\text{MoCoFeS}(\text{CO})_8]_2[\eta^5\text{-C}_5\text{H}_4\text{C}(\text{O})\text{CH}_2]_2$  (**9**). However, the two subclusters in **7** are connected through Mo atoms to two  $\eta^5$ -cyclopentadienyl rings in a cis fashion, whereas those in **9** are connected in a trans fashion.<sup>12b</sup> Similar to **9**, the two carbonyls attached to the Mo atom in each subcluster of **7** are semibridging and the other three terminal. The existence of both terminal and semibridging CO's confirmed by X-ray diffraction is consistent with the IR spectra of **7** described above. For semibridging carbonyls Curtis's definition<sup>18</sup> is  $0.1 \leq \alpha \leq 0.6$  ( $\alpha = (d_2 - d_1)/d_2$ ). Since  $d_2 = \text{Fe}(1)\cdots\text{C}(14) = 2.74\text{ \AA}$  and  $d_1 = \text{Mo}(1)\text{--}\text{C}(14) = 1.99\text{--}$

**Table 3.** Selected Bond Lengths (Å) and Bond Angles (deg) for **7**

Mo(1)–Ni(1)	2.639(2)	Mo(1)–Fe(1)	2.779(2)
Mo(1)–S(1)	2.351(3)	Mo(1)–C(1)	2.287(9)
Mo(1)–C(13)	2.00(1)	Ni(1)–S(1)	2.123(3)
Ni(1)–Fe(1)	2.451(2)	Fe(1)–S(1)	2.172(3)
Ni(1)–C(8)	2.09(2)	O(1)–C(6)	1.20(1)
Fe(1)–C(15)	1.75(2)	C(1)–C(6)	1.49(1)
Ni(1)–Mo(1)–Fe(1)	53.72(5)	Ni(1)–Mo(1)–S(1)	49.95(7)
Ni(1)–Mo(1)–C(1)	142.5(3)	Ni(1)–Mo(1)–C(2)	149.0(3)
Fe(1)–Mo(1)–C(1)	158.9(3)	Fe(1)–Mo(1)–C(2)	143.0(3)
S(1)–Mo(1)–C(2)	114.9(7)	Mo(1)–Ni(1)–Fe(1)	66.07(6)
Mo(1)–Ni(1)–S(1)	57.97(8)	Mo(1)–Ni(1)–C(8)	113.1(5)
Fe(1)–Ni(1)–S(1)	56.14(9)	Fe(1)–Ni(1)–C(8)	126.2(7)
Mo(1)–Fe(1)–S(1)	55.07(8)	Mo(1)–Fe(1)–Ni(1)	60.21(5)
Ni(1)–Fe(1)–S(1)	54.27(8)	Mo(1)–Fe(1)–C(15)	103.0(5)
Mo(1)–S(1)–Ni(1)	72.08(9)	Ni(1)–Fe(1)–C(15)	100.8(9)
Ni(1)–S(1)–Fe(1)	69.6(1)	Mo(1)–S(1)–Fe(1)	75.71(10)

(1) Å, for C(14)–O(3)  $\alpha = 0.38$ . Since  $d_2 = \text{Ni}(1)\cdots\text{C}(13) = 2.95\text{ \AA}$  and  $d_1 = \text{Mo}(1)\text{--}\text{C}(13) = 2.00(1)\text{ \AA}$ , for C(13)–O(2)  $\alpha = 0.47$ . So, they all fall into the  $\alpha$  value range for semibridging carbonyls. Since  $\text{Fe}(1)\text{--}\text{Mo}(1)\text{--}\text{C}(14) = 67.9(3)^\circ$ , the C(14)–O(3) is bridged across the Mo(1)–Fe(1) bond, while C(13)–O(2) is bridged across the Ni(1)–Mo(1) bond due to the angle Ni(1)–Mo(1)–C(13) =  $77.6(3)^\circ$ . Also similar to **9**, the  $\pi$ -system of half of the succinoyl bridge C(6)–O(1)–C(7) would be quite well conjugated with the Cp ring  $\pi$ -system since the atoms C(6), O(1), and C(7) are basically coplanar with the Cp ring and the bond length of C(1)–C(6) (1.49 Å) becomes much shorter than a normal C–C single bond. Table 4 lists the comparable crystallographic data between **7** and **2**.

It should be pointed out that the crystal molecule of **7** is that of a chiral molecule containing a 2-fold symmetric axis, which is the *R* form of three possible optical isomers.

## Experimental Section

All reactions were carried out under prepurified tank nitrogen using standard Schlenk or vacuum line techniques. Tetrahydrofuran (THF) and toluene were distilled from sodium–benzophenone ketyl.  $\text{Cp}_2\text{Ni}$ ,<sup>19</sup>  $\text{Fe}_2(\text{CO})_9$ ,<sup>20</sup> ( $\eta^5\text{-MeO}_2\text{-CC}_5\text{H}_4\text{MoCoFeS}(\text{CO})_8$ ),<sup>12b</sup> ( $\eta^5\text{-C}_5\text{H}_5$ )( $\eta^5\text{-RC}_5\text{H}_4$ )MoNiFeS( $\text{CO})_5$  ( $\text{R} = \text{H, MeCO}$ ),<sup>13</sup> and  $[\text{MoCoFeS}(\text{CO})_8]_2[\eta^5\text{-C}_5\text{H}_4\text{C}(\text{O})\text{CH}_2]_2$ <sup>12b</sup> were prepared according to the literature. TLC was carried out by using silica gel G (10–40  $\mu\text{m}$ ). IR spectra were recorded on a NICOLET FT-IR 5DX infrared spectrophotometer.  $^1\text{H}$  NMR spectra were obtained on a JEOL FX 90Q NMR

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**Table 4. Comparison of Some Crystallographic Data between 7 and 2**

cluster	Mo–Ni (Å)	Ni–Fe (Å)	Fe–Mo (Å)	Mo–S (Å)	Ni–S (Å)	Fe–S (Å)	Mo–Cp* (Å)	Ni–Cp* (Å)	Z (deg)
<b>2</b>	2.652(1)	2.466(2)	2.782(1)	2.362(3)	2.131(3)	2.177(3)	2.0060	1.7149	102.88
<b>7</b>	2.639(2)	2.451(2)	2.779(2)	2.351(3)	2.123(3)	2.172(3)	1.993		85.7

spectrometer. C/H analyses and MS determinations were performed on a Perkin-Elmer Model 240C analyzer and an HP 5988A spectrometer, respectively. Melting points were determined on a Yanaco MP-500 apparatus.

**Preparation of 3.** To a 100 mL three-necked flask fitted with a magnetic stir bar, a rubber septum, and a reflux condenser topped with a nitrogen inlet tube were charged 1.160 g (1.97 mmol) of  $(\eta^5\text{-MeO}_2\text{CC}_5\text{H}_4)\text{MoCoFeS}(\text{CO})_8$ , 0.840 g (4.43 mmol) of  $\text{Cp}_2\text{Ni}$ , and 25 mL of THF. The mixture was stirred at reflux for 18 h. Solvent was removed under reduced pressure, and the residue was extracted with 1:1 (v/v)  $\text{CH}_2\text{Cl}_2$ /petroleum ether. Four bands were developed. From the major fourth blue band was obtained 0.330 g (29%) of **3** as a black-green solid. Mp: 71–72 °C. Anal. Calcd for  $\text{C}_{17}\text{H}_{12}\text{FeMoNiO}_7\text{S}$ : C, 35.77; H, 2.12. Found: C, 35.61; H, 2.12. IR (KBr disk):  $\nu_{(\text{C}=\text{O})}$  2040.6 (vs), 1991.4 (s), 1966.8 (vs), 1884.8 (s);  $\nu_{(\text{C}=\text{O})}$  1712.5 (s)  $\text{cm}^{-1}$ .  $^1\text{H NMR}$  ( $\text{CDCl}_3$ ): 3.86 (s, 3H,  $\text{CH}_3$ ), 5.28 (s, 5H,  $\text{C}_5\text{H}_5$ ), 5.40–5.68 (m, 2H,  $\text{H}^3$ ,  $\text{H}^4$ ), 5.96–6.20 (m, 2H,  $\text{H}^2$ ,  $\text{H}^5$ ) ppm. MS (EI,  $\text{Mo}^{98}$ ,  $\text{Ni}^{58}$ ):  $m/z$  (relative intensity) 572 ( $\text{M}^+$ , 1.2), 516 ( $\text{M}^+ - 2\text{CO}$ , 0.9), 488 ( $\text{M}^+ - 3\text{CO}$ , 3.2), 460 ( $\text{M}^+ - 4\text{CO}$ , 1.0), 432 ( $\text{M}^+ - 5\text{CO}$ , 6.4), 309 ( $\text{C}_5\text{H}_5\text{NiMoFeS}^+$ , 1.3), 308 ( $\text{C}_5\text{H}_4\text{MoFeNiS}^+$ , 1.8), 244 ( $\text{MoFeNiS}^+$ , 4.6), 123 ( $\text{C}_5\text{H}_5\text{-Ni}^+$ , 6.8).

**Preparation of 4.** To the flask described above were charged 0.180 g (0.35 mmol) of  $(\eta^5\text{-C}_5\text{H}_5)_2\text{MoNiFeS}(\text{CO})_5$ , 0.182 g (0.50 mmol) of  $\text{Fe}_2(\text{CO})_9$ , and 15 mL of toluene. The mixture was stirred at 45–50 °C for 8 h. Solvent was removed under reduced pressure, and the residue was extracted with  $\text{CH}_2\text{Cl}_2$ . The extracts were subjected to TLC using 3:1 (v/v)  $\text{CH}_2\text{Cl}_2$ /petroleum ether as eluent. From the main brown-red band was obtained 0.018 g (8%) of **4** as a black solid. Mp: 178 °C dec. Anal. Calcd for  $\text{C}_{20}\text{H}_{10}\text{Fe}_2\text{MoNiO}_{10}\text{S}$ : C, 33.89; H, 1.42. Found: C, 33.39; H, 1.43. IR (KBr disk):  $\nu_{(\text{C}=\text{O})}$  2057.0 (s), 2024.2 (vs), 1975.0 (vs), 1958.6 (vs), 1925.8 (s), 1876.6 (s)  $\text{cm}^{-1}$ .  $^1\text{H NMR}$  ( $\text{CDCl}_3$ ): 5.34 (s, 5H,  $\text{NiC}_5\text{H}_5$ ), 5.42 (s, 5H,  $\text{MoC}_5\text{H}_5$ ) ppm.

**Preparation of 5.** To the flask described above were charged 0.385 g (0.69 mmol) of  $(\eta^5\text{-C}_5\text{H}_5)(\eta^5\text{-MeCOC}_5\text{H}_4)\text{MoNiFeS}(\text{CO})_5$ , 0.364 g (1.00 mmol) of  $\text{Fe}_2(\text{CO})_9$ , and 20 mL of toluene. The mixture was stirred at 45–50 °C for 18 h. Solvent was removed under reduced pressure, and the residue was extracted with  $\text{CH}_2\text{Cl}_2$ . The extracts were subjected to TLC using 3:1 (v/v)  $\text{CH}_2\text{Cl}_2$ /petroleum ether as eluent. From the main brown-red band was obtained 0.100 g (19%) of **5** as a black solid. Mp: 152 °C dec. Anal. Calcd for  $\text{C}_{22}\text{H}_{12}\text{Fe}_2\text{MoNiO}_{11}\text{S}$ : C, 35.20; H, 1.61. Found: C, 35.03; H, 1.68. IR (KBr disk):  $\nu_{(\text{C}=\text{O})}$  2065.2 (s), 2032.4 (s), 1991.4 (vs), 1934.0 (s), 1851.9 (s), 1810.9 (s);  $\nu_{(\text{C}=\text{O})}$  1655.1 (s)  $\text{cm}^{-1}$ .  $^1\text{H NMR}$  ( $\text{CDCl}_3$ ): 2.40 (s, 3H,  $\text{CH}_3$ ), 5.36 (s, 5H,  $\text{C}_5\text{H}_5$ ), 5.44 (t, 2H,  $\text{H}^3$ ,  $\text{H}^4$ ), 5.80 (t, 2H,  $\text{H}^2$ ,  $\text{H}^5$ ) ppm. MS (EI,  $\text{Mo}^{98}$ ,  $\text{Ni}^{58}$ ):  $m/z$  (relative intensity): 377 [ $\text{CH}_3\text{COC}_5\text{H}_4\text{MoFe}_2\text{S}(\text{CO})^+$ , 0.9], 228 [ $\text{Fe}_2\text{S}(\text{CO})_3^+$ , 1.8], 202 ( $\text{Fe}_2\text{NiS}^+$ , 1.6), 200 ( $\text{Fe}_2\text{S}(\text{CO})_2^+$ , 1.3), 188 ( $\text{MoNiS}^+$ , 2.2), 144 ( $\text{Fe}_2\text{S}^+$ , 0.7), 123 ( $\text{C}_5\text{H}_5\text{Ni}^+$ , 0.9), 112 ( $\text{Fe}_2^+$ , 3.3), 58 ( $\text{Ni}^+$ , 1.1), 56 ( $\text{Fe}^+$ , 75.7).

**Preparation of 6.** To the flask described above were charged 0.850 g (1.49 mmol) of  $(\eta^5\text{-C}_5\text{H}_5)(\eta^5\text{-MeCO}_2\text{C}_5\text{H}_4)\text{MoNiFeS}(\text{CO})_5$ , 0.791 g (2.17 mmol) of  $\text{Fe}_2(\text{CO})_9$ , and 25 mL of toluene. The mixture was stirred at 45–50 °C for 18 h. Solvent was removed under reduced pressure and the residue was extracted with  $\text{CH}_2\text{Cl}_2$ . The extracts were subjected to TLC using 3:1 (v/v)  $\text{CH}_2\text{Cl}_2$ /petroleum ether as eluent. From the main brown-red band was obtained 0.205 g (18%) of **6** as a black solid. Mp: 138–139 °C. Anal. Calcd for  $\text{C}_{22}\text{H}_{12}\text{Fe}_2\text{MoNiO}_{12}\text{S}$ : C, 34.46; H, 1.58. Found: C, 34.19; H, 1.78. IR (KBr disk):  $\nu_{(\text{C}=\text{O})}$  2065.2 (s), 2024.2 (vs), 1991.4 (vs), 1950.4 (vs), 1909.4 (vs), 1868.4 (s);  $\nu_{(\text{C}=\text{O})}$  1728.9 (s)  $\text{cm}^{-1}$ .  $^1\text{H NMR}$  ( $\text{CDCl}_3$ ): 3.82 (s, 3H,  $\text{CH}_3$ ), 5.30 (s, 5H,  $\text{C}_5\text{H}_5$ ), 5.36 (t, 2H,  $\text{H}^3$ ,

$\text{H}^4$ ), 5.78 (t, 2H,  $\text{H}^2$ ,  $\text{H}^5$ ) ppm. MS (EI,  $\text{Mo}^{98}$ ,  $\text{Ni}^{58}$ ):  $m/z$  (relative intensity) 384 [ $\text{MoFe}_2\text{NiS}(\text{CO})_3^+$ , 7.6], 298 ( $\text{MoFe}_2\text{S}(\text{CO})_2^+$ , 11.8), 286 [ $\text{Fe}_2\text{NiS}(\text{CO})_3^+$ , 4.4], 242 ( $\text{MoFe}_2\text{S}^+$ , 4.3), 162 ( $\text{C}_5\text{H}_4\text{-Mo}^+$ , 4.1), 123 ( $\text{C}_5\text{H}_5\text{Ni}^+$ , 10.7), 112 ( $\text{Fe}_2^+$ , 7.6), 98 ( $\text{Mo}^+$ , 6.4), 58 ( $\text{Ni}^+$ , 12.3), 56 ( $\text{Fe}^+$ , 20.3).

**Preparation of 8.** To the flask described above were charged 0.438 g (0.40 mmol) of  $[(\eta^5\text{-C}_5\text{H}_5)\text{MoNiFeS}(\text{CO})_5]_2[\eta^5\text{-C}_5\text{H}_4\text{C}(\text{O})\text{CH}_2]_2$ , 0.424 g (1.17 mmol) of  $\text{Fe}_2(\text{CO})_9$ , and 25 mL of toluene. The mixture was stirred for 8 h at 45–50 °C. Solvent was removed at reduced pressure, and the residue was subjected to TLC using 2:1:1 (v/v)  $\text{Et}_2\text{O}/\text{CH}_2\text{Cl}_2$ /petroleum ether as eluent. From the main brown-black band was obtained 0.116 g (19%) of **8** as a black solid. Mp: 161 °C dec. Anal. Calcd for  $\text{C}_{44}\text{H}_{22}\text{Fe}_4\text{Mo}_2\text{Ni}_2\text{O}_{22}\text{S}_2$ : C, 35.25; H, 1.48. Found: C, 34.88; H, 1.67. IR (KBr disk):  $\nu_{(\text{C}=\text{O})}$  2057.0 (m), 2032.4 (vs), 1966.8 (vs), 1860.1 (w);  $\nu_{(\text{C}=\text{O})}$  1679.7 (w)  $\text{cm}^{-1}$ .  $^1\text{H NMR}$  (acetone- $d_6$ ): 3.12 (s, 4H,  $\text{CH}_2\text{CH}_2$ ), 5.68 (s, 10H,  $2\text{C}_5\text{H}_5$ ), 5.86–6.48 (m, 8H,  $\text{C}_5\text{H}_4$ ) ppm.

**Preparation of 7 and 10.** To the flask described above were charged 1.226 g (1.07 mmol) of  $[\text{MoCoFeS}(\text{CO})_8]_2[\eta^5\text{-C}_5\text{H}_4\text{C}(\text{O})\text{CH}_2]_2$ , 1.012 g (5.36 mmol) of  $\text{Cp}_2\text{Ni}$ , and 30 mL of THF. The mixture was stirred at reflux for 18 h. Solvent was removed at reduced pressure, and the residue was extracted with 4:1 (v/v)  $\text{CH}_2\text{Cl}_2$ /petroleum ether. The extracts were subjected to TLC using 4:1 (v/v)  $\text{CH}_2\text{Cl}_2$ /petroleum ether as eluent. From the main deep-green band was obtained 0.170 g (14%) of **7** as a black solid. Mp: 205–207 °C. Anal. Calcd for  $\text{C}_{34}\text{H}_{22}\text{Fe}_2\text{Mo}_2\text{Ni}_2\text{O}_{12}\text{S}_2$ : C, 36.87; H, 2.00. Found: C, 36.49; H, 2.19. IR (KBr disk):  $\nu_{(\text{C}=\text{O})}$  2032.4 (vs), 1983.2 (vs), 1950.4 (vs), 1884.8 (vs);  $\nu_{(\text{C}=\text{O})}$  1679.7 (s)  $\text{cm}^{-1}$ .  $^1\text{H NMR}$  ( $\text{CDCl}_3$ ): 3.04 (s, 4H,  $\text{CH}_2\text{CH}_2$ ), 5.22 (s, 10H,  $2\text{C}_5\text{H}_5$ ), 5.44–5.64 (m, 4H,  $2\text{H}^3$ ,  $2\text{H}^4$ ), 5.94–6.12 (m, 4H,  $2\text{H}^2$ ,  $2\text{H}^5$ ) ppm. From brown-green band was obtained 0.020 g (5%) of **10** as a brown-green solid. Mp: 96–97 °C. Anal. Calcd for  $\text{C}_{32}\text{H}_{17}\text{CoFe}_2\text{Mo}_2\text{NiO}_{15}\text{S}_2$ : C, 34.11; H, 1.52. Found: C, 34.16; H, 1.61. IR (KBr disk):  $\nu_{(\text{C}=\text{O})}$  2073.4 (s), 2032.4 (vs), 1966.8 (vs), 1868.4 (s);  $\nu_{(\text{C}=\text{O})}$  1679.7 (s)  $\text{cm}^{-1}$ .  $^1\text{H NMR}$  ( $\text{CDCl}_3$ ): 2.80 (s, 4H,  $\text{CH}_2\text{CH}_2$ ), 5.10 (s, 5H,  $\text{C}_5\text{H}_5$ ), 5.24–5.56 (m, 4H,  $\text{H}^3$ ,  $\text{H}^3'$ ,  $\text{H}^4$ ,  $\text{H}^4'$ ), 5.72–6.04 (m, 4H,  $\text{H}^2$ ,  $\text{H}^2'$ ,  $\text{H}^5$ ,  $\text{H}^5'$ ) ppm.

**Crystal Structure Determination of 5.** Single crystals of **5** suitable for X-ray diffraction were obtained by slow evaporation of its hexane solution. A single crystal measuring  $0.30 \times 0.20 \times 0.15$  mm was mounted on a glass fiber and placed on an Enraf-Nonius CAD4 diffractometer equipped with a graphite monochromator. A total of 4799 independent reflections were collected at 23 °C by the  $\omega/2\theta$  scan mode, of which 3818 independent reflections  $I \geq 3\sigma(I)$  were considered to be observed and used in subsequent refinement. Crystal data are listed in Table 5.

The structures were solved by a direct phase determination method (MULTAN 82). The final refinement was accomplished by a full-matrix least-squares method with anisotropic thermal parameters for non-hydrogen atoms. All calculations were performed on a MICRO-VAX II computer using the TEXSAN program system.

**Crystal Structure Determination of 7.** Single crystals of **7** suitable for X-ray diffraction were obtained by slow evaporation of its  $\text{CH}_2\text{Cl}_2$ /hexane solution. A single crystal measuring  $0.20 \times 0.20 \times 0.30$  mm was mounted on a glass fiber and placed on a Rigaku AFC7R diffractometer with graphite-monochromated Mo  $K\alpha$  radiation ( $\lambda = 0.71069$  Å) and a 12 kW rotating anode generator. A total of 3488 independent reflections were collected at 20 °C by the  $\omega-2\theta$  scan mode, of which 1736 independent reflections  $I \geq 3\sigma(I)$  were considered to be observed and used in subsequent refinement. The data were corrected for Lp factors. Crystal data are listed in Table 5.

Table 5. Crystal Data Collection and Refinement of Compounds for 5 and 7

	5	7
mol formula	C <sub>22</sub> H <sub>12</sub> Fe <sub>2</sub> MoNiO <sub>11</sub> S	C <sub>17</sub> H <sub>11</sub> FeMoNiO <sub>6</sub> S
mol wt	750.72	553.82
cryst syst	monoclinic	monoclinic
space group	<i>P</i> 2 <sub>1</sub> / <i>n</i> (No. 14)	<i>C</i> 2/ <i>c</i> (No. 15)
<i>a</i> /Å	16.288(4)	23.889(3)
<i>b</i> /Å	9.226(1)	10.330(3)
<i>c</i> /Å	17.081(3)	15.703(3)
$\beta$ /deg	95.54(2)	105.75(1)
<i>V</i> /Å <sup>3</sup>	2555(1)	3729(1)
<i>Z</i>	4	8
density (calcd)/g cm <sup>-3</sup>	1.95	1.972
<i>F</i> (000)	1480	2184.00
$\mu$ (Mo K $\alpha$ )/cm <sup>-1</sup>	24.47	25.70
diffractometer	Enraf-Nonius CAD4	Rigaku AFC7R
temp/°C	23	20
radiation	Mo K $\alpha$ ( $\lambda$ = 0.710 69 Å)	Mo K $\alpha$ ( $\lambda$ = 0.710 69 Å)
scan type	$\omega/2\theta$	$\omega/2\theta$
$2\theta_{\max}$ /deg	49.9	50
no. of observns, <i>n</i>	3818	1736
no. of variables, <i>p</i>	343	247
<i>R</i>	0.039	0.046
<i>R</i> <sub>w</sub>	0.054	0.051
goodness of fit indicator	1.55	1.55
max shift in final cycle	0.06	0.01
largest peak in final diff map/e Å <sup>-3</sup>	0.71	1.52

The structure was solved by direct methods and expanded using Fourier techniques. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included but not refined. Final refinement by a full-matrix least-squares method for non-hydrogen atoms converged to give the unweighted and weighted agreement factors of 0.046 (*R*) and 0.051 (*R*<sub>w</sub>). The highest peak on the final difference Fourier map has a height of 1.52 e Å<sup>-3</sup>. All calculations were performed using the TEXSAN program system crystallographic software package of the Molecular Structure Corporation.

**Acknowledgment.** We are grateful to the National Natural Science Foundation of China, the State Key Laboratory of Structural Chemistry, and the Laboratory of Organometallic Chemistry for financial support of this work.

**Supporting Information Available:** Full tables of crystal data, atomic coordinates and thermal parameters, and bond lengths and angles for 5 and 7 (14 pages). Ordering information is given on any current masthead page.

OM970577Y