

Cluster synthesis by the reactions of $[\text{Cp}'_2\text{M}_2\text{S}_4]$ with metal complexes ($\text{Cp}' = \text{Cp}$ and substituted cyclopentadienyl ligands, $\text{M} = \text{Fe}, \text{Ru}$)

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Abstract

Dinuclear transition-metal sulfur complexes of the type $[\text{Cp}'_2\text{M}_2\text{S}_4]$ ($\text{Cp}' = \text{Cp}$ and substituted cyclopentadienyl ligands) have been used as excellent starting materials for building up multinuclear transition-metal sulfur clusters. However, little is known about the versatility of the iron- and ruthenium-containing complexes. This urged us to examine the nature of $\text{Cp}'_2\text{M}_2\text{S}_4$ ($\text{M} = \text{Fe}, \text{Ru}$). The present review describes the research on homo- and heteronuclear cluster synthesis by the reactions of $\text{Cp}'_2\text{M}_2\text{S}_4$ ($\text{M} = \text{Fe}, \text{Ru}$) with various transition-metal complexes. © 2000 Elsevier Science S.A. All rights reserved.

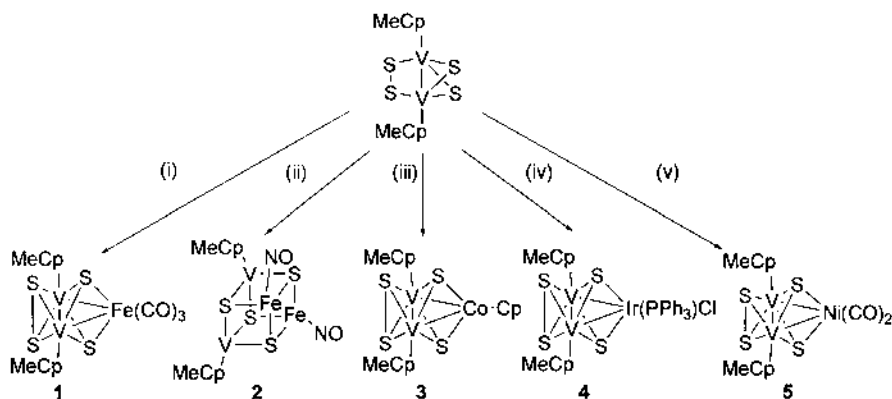
Keywords: Clusters; Iron; Ruthenium; Tungsten; Sulfido; Tetrathiotungstate

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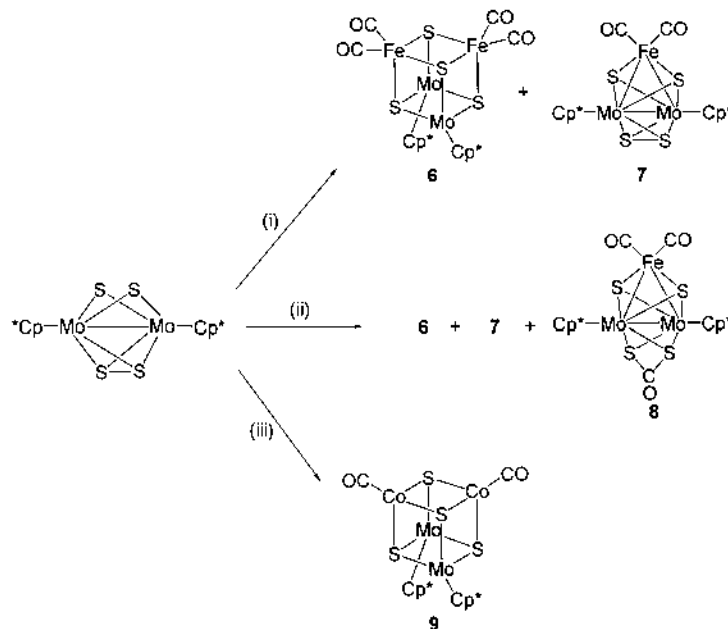
1. Introduction

The syntheses and reactivities of dinuclear transition-metal sulfur complexes of the type $[\text{Cp}'_2\text{M}_2\text{S}_4]$ ($\text{Cp}' = \text{Cp}$ and substituted cyclopentadienyl ligands) have been reported for various transition-metals, and their structures show a surprisingly wide variety depending on the nature of the metal centers [1]. The construction of transition-metal sulfur clusters has been a subject of research in relation to the possible relevance to biological [2] and industrial [3] catalysis. The Mo and V dimers have been employed as excellent starting materials for synthesis of homo- and heteronuclear clusters. Rauchfuss and his co-workers established the construction of the vanadium–sulfur clusters by the reaction of $(\text{MeCp})_2\text{V}_2\text{S}_4$ ($\text{MeCp} = \eta^5\text{-C}_5\text{H}_4\text{Me}$) with various transition-metals. The dimer reacted with $\text{Fe}(\text{CO})_5$, $\text{Hg}[\text{Fe}(\text{NO})(\text{CO})_3]_2$, $\text{CpCo}(\text{CO})_2$, $\text{IrCl}(\text{N}_2)(\text{PPh}_3)_2$, and $\text{Ni}(\text{CO})_4$ to give $(\text{MeCp})_2\text{V}_2\text{S}_4\text{Fe}(\text{CO})_3$ (**1**), $(\text{MeCp})_2\text{V}_2\text{S}_4\text{Fe}_2(\text{NO})_2$ (**2**), $(\text{MeCp})_2\text{V}_2\text{S}_4\text{CoCp}$ (**3**), $(\text{MeCp})_2\text{V}_2\text{S}_4\text{IrCl}(\text{PPh}_3)$ (**4**), and $(\text{MeCp})_2\text{V}_2\text{S}_4\text{Ni}(\text{CO})_2$ (**5**), respectively, as illustrated in Scheme 1 [4]. Wachter and his co-workers employed $\text{Cp}_2^*\text{Mo}_2\text{S}_4$ ($\text{Cp}^* = \eta^5\text{-C}_5\text{Me}_5$) (Scheme 2). Photolysis of $\text{Cp}_2^*\text{Mo}_2(\mu\text{-S}_2)(\mu\text{-S})_2$ and $\text{Fe}(\text{CO})_5$ gave a cubane-type cluster $\text{Cp}_2^*\text{Mo}_2\text{Fe}_2\text{S}_4(\text{CO})_4$ (**6**) and a trinuclear cluster $\text{Cp}_2^*\text{Mo}_2(\mu\text{-S}_2)(\mu_3\text{-S})_2\text{Fe}(\text{CO})_2$ (**7**) [5]. The Mo dimer reacted with $\text{Fe}_2(\text{CO})_9$ in THF at room temperature to give not only **6** and **7** but also a trigonal-bipyramidal cluster **8** composed of one iron, two molybdenum and two sulfur atoms [5]. The $\text{Mo}_2\text{Co}_2\text{S}_4$ cubane-type cluster **9** was synthesized by the reaction of the Mo dimer with $\text{Co}_2(\text{CO})_8$ via liberation of carbon monoxide [6].

Among the $\text{Cp}'_2\text{M}_2\text{S}_4$ complexes, surprisingly little has been known about the reactivity of the iron and ruthenium dimers toward other transition-metals. In 1980, Gianotti et al. reported that the photolysis of $\text{Cp}_2\text{Fe}_2(\text{CO})_4$ in the presence of elemental sulfur gave an isomeric pair of $\text{Cp}_2\text{Fe}_2\text{S}_4$ [7]. The structure of one isomer was solved by the single crystal X-ray analysis, in which two disulfide ligands take different coordination mode [8]: One disulfide ligand bounds to two iron centers in



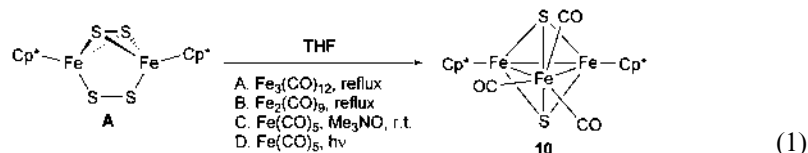
Scheme 1. (i) $\text{Fe}(\text{CO})_5$ (ii) $\text{Hg}[\text{Fe}(\text{NO})(\text{CO})_3]_2$ (iii) $\text{CpCo}(\text{CO})_2$ (iv) $\text{IrCl}(\text{N}_2)(\text{PPh}_3)_2$ (v) $\text{Ni}(\text{CO})_4$.

Scheme 2. (i) $\text{Fe}(\text{CO})_5$, $h\nu$ (ii) $\text{Fe}_2(\text{CO})_9$ (iii) $\text{Co}_2(\text{CO})_8$.

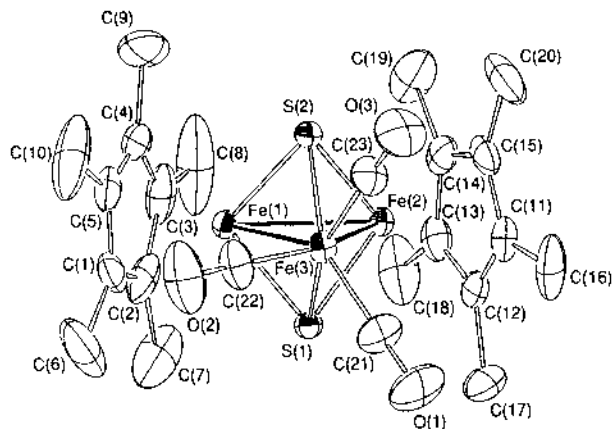
a $\mu\text{-}\eta^2\text{:}\eta^2$ side-on manner, whereas the other adopts a $\mu\text{-}\eta^1\text{:}\eta^1$ *endo*-on manner. The iron-iron distance of 3.49 Å shows no metal–metal interaction. In 1985, Wachter et al. reported that the Cp^* analog, which was obtained by the thermal reaction of $\text{Cp}^*_2\text{Fe}_2(\text{CO})_4$ with elemental sulfur, has a similar structure [9]. Rauchfuss et al. synthesized the ruthenium dimer $(\text{C}_5\text{Me}_4\text{Et})_2\text{Ru}_2\text{S}_4$ by a thermal reaction. The structure was found to be quite similar to the iron analog [10]. The present review deals with the cluster construction by the reactions of $\text{Cp}^*_2\text{M}_2\text{S}_4$ ($\text{M} = \text{Fe}, \text{Ru}$) with transition-metals.

2. Cluster construction by the reactions of $\text{Cp}^*_2\text{Fe}_2\text{S}_4$ with transition-metals [11]

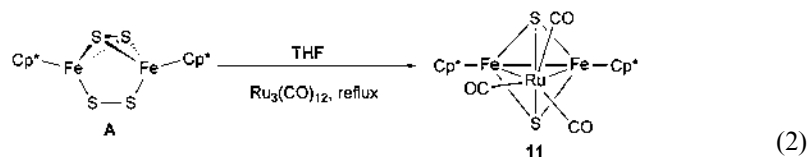
Triiron cluster **10** was synthesized by the reaction of $\text{Cp}^*_2\text{Fe}_2\text{S}_4$ (**A**) with three kinds of iron carbonyls and isolated as yellowish brown crystals (Eq. (1)) [12].



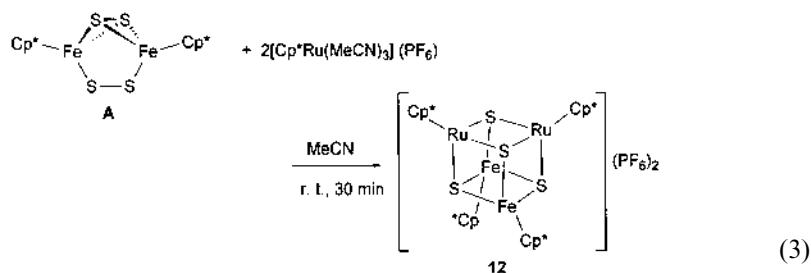
The reaction of **A** with $\text{Fe}_2(\text{CO})_9$ or $\text{Fe}_3(\text{CO})_{12}$ proceeds in refluxing THF. However, no reaction of **A** with $\text{Fe}(\text{CO})_5$ was observed under the thermal conditions. In the reaction with $\text{Fe}(\text{CO})_5$, addition of Me_3NO promoted the formation of **10**,

Fig. 1. ORTEP drawing of **10**.

where Me_3NO works as a decarbonylation reagent. Photolysis of a mixture of **A** and $\text{Fe}(\text{CO})_5$ in THF also produced **10**. These results established that generation of the coordinatively unsaturated complex $\text{Fe}(\text{CO})_4$ is crucial for the reaction of **A** with iron carbonyls to give **10**. An ORTEP drawing of **10** is shown in Fig. 1. The Fe_3S_2 core of **10** adopts a trigonal bipyramidal configuration: The three Fe atoms form a triangle which is capped by two μ_3 -S ligands. Cluster **10** is the first example of *closo* cluster with Fe_3S_2 core. The iron and ruthenium mixed-metal trinuclear cluster $\text{Cp}_2^*(\text{CO})_3\text{Fe}_2\text{RuS}_2$ (**11**) was formed in the reaction of **A** with $\text{Ru}_3(\text{CO})_{12}$ in refluxing THF (Eq. (2)) [12].



Treatment of **A** with two equivalents $[\text{Cp}^*\text{Ru}(\text{MeCN})_3](\text{PF}_6)$ in acetonitrile at room temperature resulted in the formation of the $\text{Fe}_2\text{Ru}_2\text{S}_4$ cubane-type cluster **12** (Eq. (3)) [12].



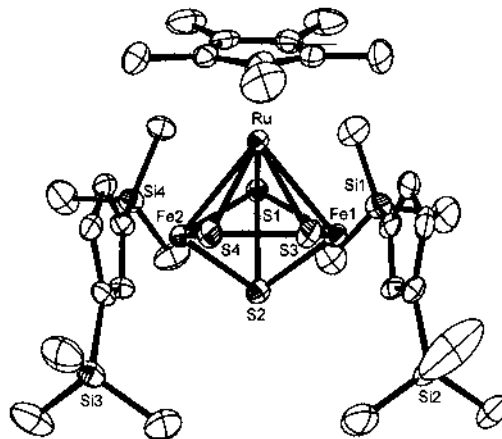
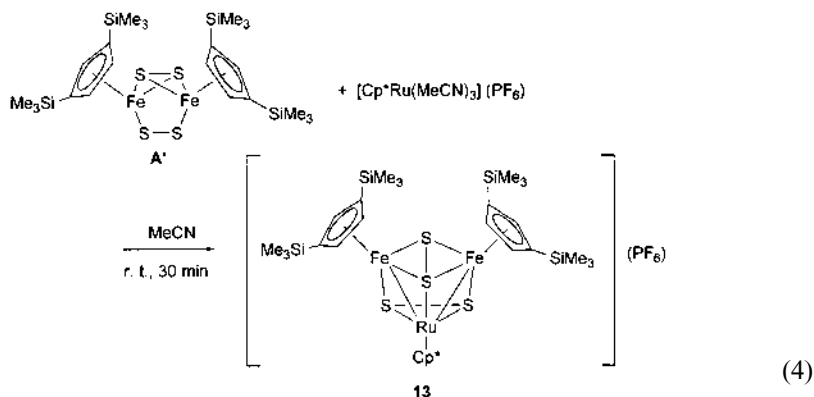


Fig. 2. ORTEP drawing of the cationic moiety in **13**.

The reaction of **A** with one equivalent $[\text{Cp}^*\text{Ru}(\text{MeCN})_3](\text{PF}_6)$ was examined. It took place at room temperature to give **12** and several unidentified products. The isolation of the latter products was unsuccessful.

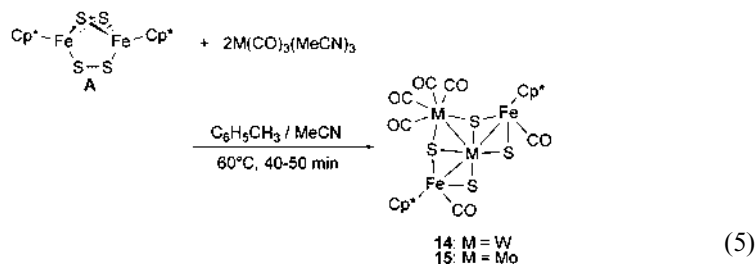
Treatment of $\text{Cp}_2^{\text{S}2}\text{Fe}_2\text{S}_4$ (**A'**) with one equivalent $[\text{Cp}^*\text{Ru}(\text{MeCN})_3](\text{PF}_6)$ afforded a Fe_2RuS_4 cluster **13** (Eq. (4)), where $\text{Cp}^{\text{S}2}$ ($= 1,3\text{-C}_5\text{H}_3(\text{SiMe}_3)_2$) is a much bulkier ligand than Cp^* .



Even if two equivalents $[\text{Cp}^*\text{Ru}(\text{MeCN})_3](\text{PF}_6)$ was added, no tetranuclear cluster $[(\text{Cp}^{\text{S}2}\text{Fe})_2(\text{Cp}^*\text{Ru})_2\text{S}_4](\text{PF}_6)_2$ which corresponds to **12** was formed. This means that the use of the bulky $\text{Cp}^{\text{S}2}$ ligand prevents the formation of the tetranuclear cluster such as **12**. The trinuclear cluster **13** is considered to be the intermediate to the tetranuclear cluster. The structure of **13** was determined by X-ray crystal structure analysis. The ORTEP drawing of the cationic moiety in **13** is shown in Fig. 2. The cluster contains an Fe_2RuS_4 core with two $\mu_3\text{-}\eta^1\text{:}\eta^2\text{:}\eta^2$ and $\mu_3\text{-}\eta^1\text{:}\eta^1\text{:}\eta^2$ disulfido groups. The torsion angle of two disulfido groups is 84.1° . Cluster **13** can be considered as a Cp^*Ru -capped $\text{Cp}_2^{\text{S}2}\text{Fe}_2\text{S}_4$ in which the $\text{Cp}_2^{\text{S}2}\text{Fe}_2\text{S}_4$ unit is coordi-

nated in η^5 -fashion to the ruthenium atom by the Fe1–S1–Fe2–S4–S3 five-membered ring.

Thermolysis of **A** and $\text{W}(\text{CO})_3(\text{MeCN})_3$ in 1:2 molar ratio at 60°C afforded a tetranuclear cluster **14** (Eq. (5)) [13].



Elemental analysis and mass spectral data indicate that **14** has the formula $\text{Cp}^*_2\text{Fe}_2\text{W}_2\text{S}_4(\text{CO})_6$. The composition is the same as a cubane-type mixed-metal sulfur cluster $(\text{Cp}^*\text{Fe})_2\{\text{W}(\text{CO})_3\}_2(\mu_3\text{-S})_4$. However, X-ray diffraction study disclosed that this is not the case. Cluster **14** was unequivocally determined to be $\{\text{Cp}^*\text{Fe}(\text{CO})\}_2\{\text{W}(\mu_3\text{-S})_2(\mu\text{-S})_2\}\{\text{W}(\text{CO})_4\}$ by X-ray crystal structure analysis (Fig. 3). The WS_4 unit is bound to one tungsten by two S atoms and two iron atoms by four S atoms, two of which bridge one iron and two tungsten atoms in a μ_3 -fashion and the others are bound to one iron and one tungsten atoms in a μ_2 -fashion. The W1 atom with four S ligands and W2 atom with two S and four CO ligands adopts a distorted tetrahedral and octahedral geometry, respectively. In the reaction of Eq. (5), six MeCN ligands were released, and two $\text{W}(\text{CO})_3$ moieties were converted to WS_4 and $\text{W}(\text{CO})_4$. Interestingly, redistribution of CO and S ligands took place between Fe and W atoms without the loss of any CO and S ligands, where the driving force for the formation of **14** may be due to the exceptionally high stability of the $[\text{WS}_4]^{2-}$.

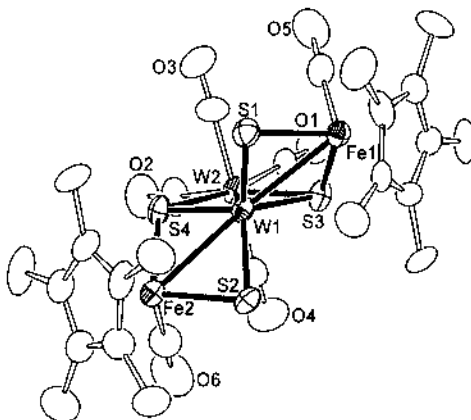
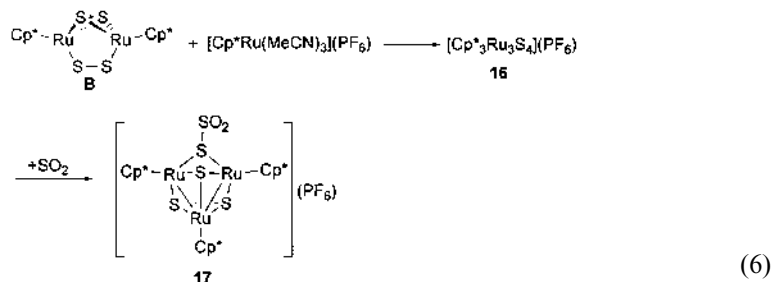


Fig. 3. ORTEP drawing of **14**.

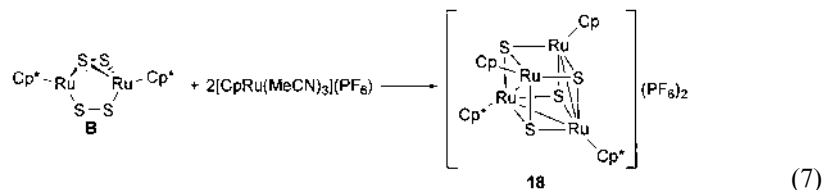
The iron–molybdenum cluster **15** was also obtained by the reaction of **A** with $\text{Mo}(\text{CO})_3(\text{MeCN})_3$ in 1:2 molar ratio at 60°C (Eq. (5)) [13].

3. Cluster construction by the reactions of $\text{Cp}_2^*\text{Ru}_2\text{S}_4$ with transition-metals

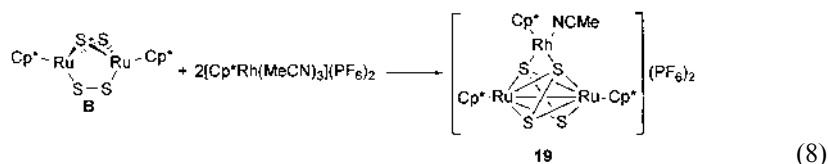
Rauchfuss et al. showed a versatility of $\text{Cp}_2^*\text{Ru}_2\text{S}_4$ (**B**) as a precursor for synthesis of polynuclear transition-metal sulfur clusters. The reaction of $\text{Cp}_2^*\text{Ru}_2\text{S}_4$ with $[\text{Cp}^*\text{Ru}(\text{MeCN})_3](\text{PF}_6)$ in refluxing acetonitrile afforded a trinuclear cluster $[\text{Cp}_3^*\text{Ru}_3\text{S}_4](\text{PF}_6)$ (**16**) (Eq. (6)) [14].



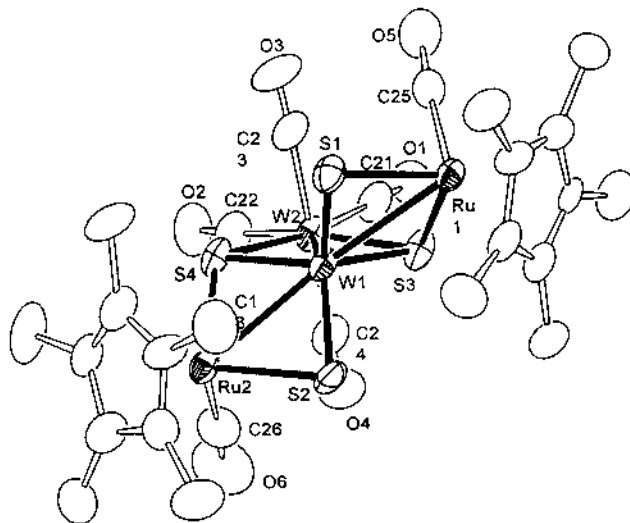
The structure of **16** is uncertain but can be inferred from its derivative. Treatment of **16** with SO_2 produced $[\text{Cp}_3^*\text{Ru}_3\text{S}_4\text{SO}_2](\text{PF}_6)$ (**17**) which was characterized crystallographically (Eq. (6)). Cluster **17** has an isosceles Ru_3 core with two Ru – Ru bonds. Three Ru metals are connected with $\mu_3\text{-S}$, $\mu_3\text{-}\eta^1\text{:}\eta^1\text{:}\eta^2\text{-S}_2$, and $\mu_2\text{-S}$ to which the SO_2 moiety is bound. The reaction of **B** with sterically less congested $[\text{CpRu}(\text{MeCN})_3](\text{PF}_6)$ produced a cubane-type cluster $[\text{Cp}_2^*\text{Cp}_2\text{Ru}_4\text{S}_4](\text{PF}_6)_2$ (**18**) which was characterized crystallographically (Eq. (7)) [15].



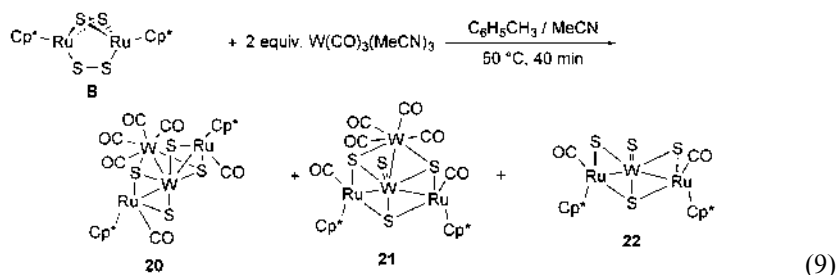
Furthermore, Rauchfuss et al. succeeded in the isolation of a heteronuclear sulfido cluster by using **B**. The Ru dimer **B** reacted with $[\text{Cp}^*\text{Rh}(\text{MeCN})_3](\text{PF}_6)_2$ in THF to afford a RhRu_2S_4 cluster **19** (Eq. (8)) [16].



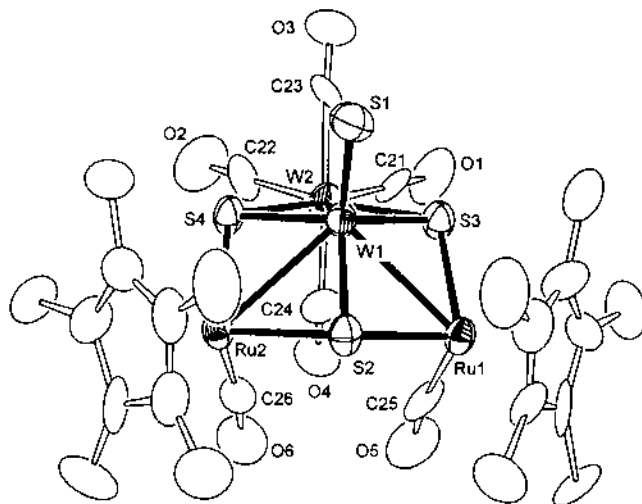
Based on X-ray crystal structure analysis, the dication **19** consists of an unsymmetrical RhRu_2S_4 core with one Ru – Ru bond. Three transition-metals are connected with two $\mu_3\text{-}\eta^1\text{:}\eta^2\text{:}\eta^2$ disulfido ligands.

Fig. 4. ORTEP drawing of **20**.

We examined the reaction of **B** with an early-transition-metal complex. Thermal reaction of **B** with $\text{W}(\text{CO})_3(\text{MeCN})_3$ gave two kinds of $\text{Ru}_2\text{W}_2\text{S}_4$ clusters **20** and **21** and a Ru_2WS_4 cluster **22** (Eq. (9)) [17,18].

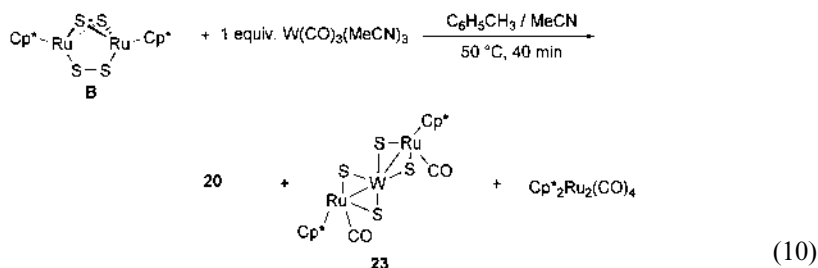


In the reaction of the iron dimer **A** with $\text{M}(\text{CO})_3(\text{MeCN})_3$ ($\text{M} = \text{Mo}, \text{W}$), only one $\text{Fe}_2\text{M}_2\text{S}_4$ cluster was obtained (Eq. (5)). Clusters **20** and **21** were characterized by single-crystal X-ray diffraction analysis. The ORTEP drawings of **20** and **21** are shown in Figs. 4 and 5, respectively. In cluster **20**, the WS_4 fragment is bound to one W by two S atoms and two Ru atoms by four S atoms, two of which bridge one Ru and two W atoms in a μ_3 -fashion and the other two are bound to one Ru and one W atoms in a μ_2 -fashion. The W2 atom with four CO and two S ligands adopts a distorted octahedral geometry. Unlike cluster **20**, there is a terminal S ligand in **21**. The WS_4 fragment is bound to one W atom by two S atoms and two Ru atoms by three S atoms, two of which bridge one Ru and two W atoms in a μ_3 -fashion and the other one is bound to two Ru and one W atoms in a μ_3 -fashion. The crystal structure of **22** is shown in Fig. 6. The structure of **22** is similar to **21** except that **22** has no $\text{W}(\text{CO})_4$ moiety. The $[\text{WS}_4]^{2-}$ fragment is bound to two Ru atoms by three S atoms, two of which bridge one W and one Ru atoms in a

Fig. 5. ORTEP drawing of **21**.

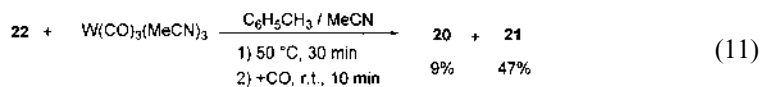
μ_2 -fashion, and the other one bridges one W and two Ru atoms in a μ_3 -fashion. The residual S atom is bound to the W atom in a terminal mode. Cluster **22** has a pseudo-mirror plane composed of S1, S2, and W atoms.

When the reaction of **B** with one equivalent $\text{W}(\text{CO})_3(\text{MeCN})_3$ was performed at 50°C , a Ru_2WS_4 trinuclear cluster **23** was obtained (Eq. (10)).

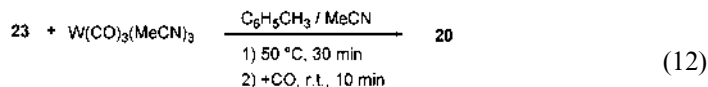


The structure of the $\text{C}_5\text{Me}_4\text{Et}$ analog $\{(\text{C}_5\text{Me}_4\text{Et})\text{Ru}(\text{CO})\}_2\{\text{W}(\mu\text{-S})_4\}$ (**24**) was determined by the X-ray crystal structure analysis. An ORTEP drawing of **24** is shown in Fig. 7. The $[\text{WS}_4]^{2-}$ fragment adopts a slightly distorted tetrahedral geometry, and each of four S atoms is bound to one ruthenium and one tungsten atoms in a μ_2 -fashion. Thus, cluster **20** can be regarded as the cluster **23** capped with a $\text{W}(\text{CO})_4$ in which the tetrathiotungstate moiety of **23** is coordinated in η^3 -fashion to the tungsten atom by two sulfur and one tungsten atoms.

Trinuclear clusters **22** and **23** may be the intermediates to give tetranuclear clusters **20** and **21**. To clarify this, the reactions of **22** and **23** with $\text{W}(\text{CO})_3(\text{MeCN})_3$ and CO were carried out. Upon heating a solution of the trinuclear cluster **22** and $\text{W}(\text{CO})_3(\text{MeCN})_3$, followed by bubbling CO, **21** with a small amount of **20** was obtained (Eq. (11)).



On the other hand, thermolysis of **23** and $\text{W}(\text{CO})_3(\text{MeCN})_3$, followed by bubbling CO, led to the exclusive formation of **20** (Eq. (12)).



Under the same conditions as those in Eq. (9) (50°C, 40 min), no isomerization reactions between **20** and **21** and between **22** and **23** were observed. The cross-reaction of a 1:1 molar mixture of **B** and its $\text{C}_5\text{Me}_4\text{Et}$ derivative $(\text{C}_5\text{Me}_4\text{Et})_2\text{Ru}_2\text{S}_4$ with $[\text{W}(\text{CO})_3(\text{MeCN})_3]$ gave **20**, $\{(\text{C}_5\text{Me}_4\text{Et})\text{Ru}(\text{CO})\}_2\{\text{W}(\mu_3\text{-S})(\mu_2\text{-S})\}\{\text{W}(\text{CO})_4\}$ (**20'**), **21**, and $\{(\text{C}_5\text{Me}_4\text{Et})\text{Ru}(\text{CO})\}_2\{\text{W}(\mu_3\text{-S})_3(=\text{S})\}\{\text{W}(\text{CO})_4\}$ (**21'**). No crossover

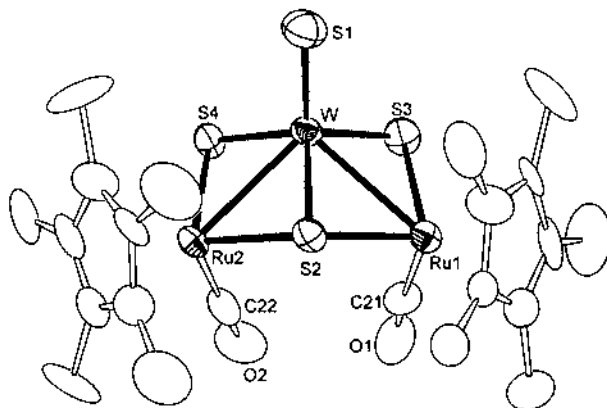


Fig. 6. ORTEP drawing of **22**.

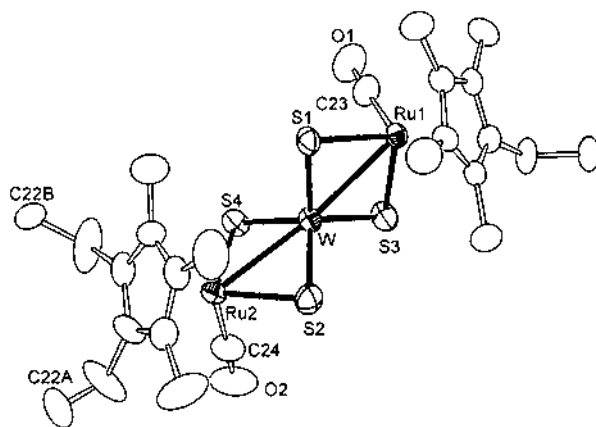
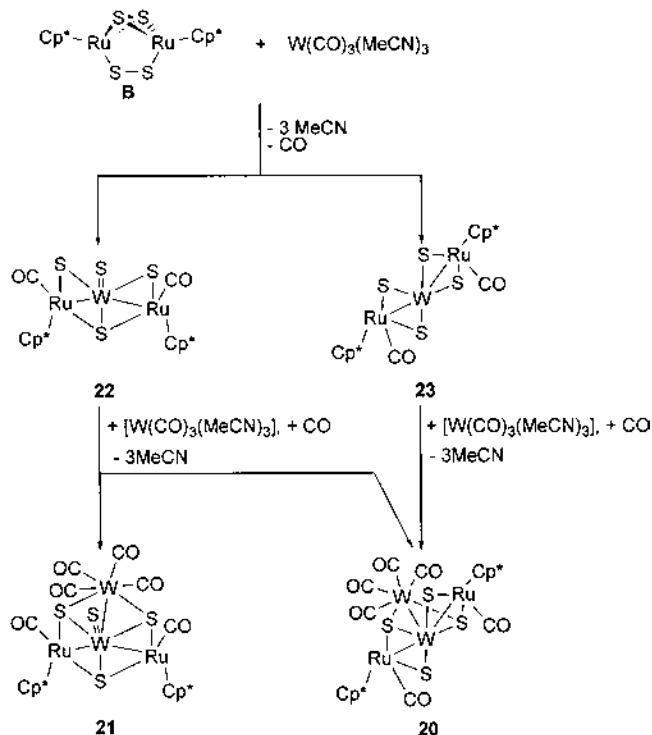


Fig. 7. ORTEP drawing of **24**.

Scheme 3. A plausible reaction pathway for the formation of **20** and **21**.

product such as $\{\text{Cp}^*\text{Ru}(\text{CO})\}\{(\text{C}_5\text{Me}_4\text{Et})\text{Ru}(\text{CO})\}\{\text{W}(\mu_3\text{-S})(\mu_2\text{-S})\}\{\text{W}(\text{CO})_4\}$ or $\{\text{Cp}^*\text{Ru}(\text{CO})\}\{(\text{C}_5\text{Me}_4\text{Et})\text{Ru}(\text{CO})\}\{\text{W}(\mu_3\text{-S})_3(=\text{S})\}\{\text{W}(\text{CO})_4\}$ was detected. This indicates that the reaction of $\text{Cp}_2^*\text{Ru}_2\text{S}_4$ (**B**) with $\text{W}(\text{CO})_3(\text{MeCN})_3$ takes place intramolecularly. A plausible reaction pathway for the formation of **20** and **21** is illustrated in Scheme 3. The reaction of **B** with $\text{W}(\text{CO})_3(\text{MeCN})_3$ gives two kinds of trinuclear clusters **22** and **23** intramolecularly via liberation of one CO and three MeCN ligands. During the reaction, reorganization of CO and S ligands took place between one W and two Ru atoms, where the driving force may be the high stability of the tetrathiotungstate $[\text{WS}_4]^{2-}$. Upon subsequent reactions of **22** and **23** with $\text{W}(\text{CO})_3(\text{MeCN})_3$ and CO, cluster **22** is converted to **21** with a small amount of **20**, whereas **23** is converted to **20**, exclusively.

4. Conclusion

This review has described the cluster construction methodology by the reactions of the reactive transition-metal sulfur complexes of the type $\text{Cp}'\text{M}_2\text{S}_4$ with various transition-metals. The iron or ruthenium dimers were shown to be excellent starting materials for synthesizing not only homo- but also heteronuclear clusters.

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