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Control of the photochemistry of $Ru_3(CO)_{12}$ and $Os_3(CO)_{12}$ by variation of the solvent[1]

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Abstract

The synthetic potential of the photosubstitution of CO by two-electron donor ligands in $M_3(CO)_{12}$ [M = Ru, Os] has been investigated. When used as photolysis media, diethyl ether, ethyl acetate and acetonitrile act as photofragmentation quenchers allowing for the synthesis of photosubstitution products in high yield. UV photolysis of $M_3(CO)_{12}$ with added triphenylphosphine in these photolysis media leads to $M_3(CO)_{12-n}(PPh_3)_n$ ($n = 1$, 2 or 3). Prolonged photolysis with added tricyclohexylphosphine generates the highly sterically crowded complex $M_3(CO)_9(PCy_3)_3$. Photolysis with thiols, RSH (R = Et, Ph), leads to the thiolato complexes $HM_3(\mu\text{-SR})(CO)_{10}$, prolonged photolysis of which generates the corresponding sulphido cluster $M_3(\mu_3\text{-S})(CO)_{10}$. Photolysis of $M_3(CO)_{12}$ in acetonitrile with no added ligand results in the generation of $M_3(CO)_{12-n}(MeCN)_n$ ($n = 1$ or 2). This offers a route to these complexes without the need for the use of oxidising agents such as trimethylamine-*N*-oxide. Photolysis of an ethene-saturated diethyl ether or ethyl acetate solution of $M_3(CO)_{12}$ leads to no net photoreaction in the case of ruthenium, whereas, for osmium, the olefin complex $Os(CO)_4(\eta^2\text{-}C_2H_4)$ is formed. This highlights the difference in the photosubstitution mechanism for $Ru_3(CO)_{12}$ and $Os_3(CO)_{12}$. © 1999 Elsevier Science S.A. All rights reserved.

Keywords: Photochemistry; Ruthenium; Osmium; Carbonyl clusters

1. Introduction

Photochemistry offers a simple and often highly selective route to organometallic compounds, overcoming large enthalpy barriers which otherwise require the use of high temperatures or pressures [1]. When considering metal carbonyl compounds, the simplest photoreaction is the dissociative loss of CO from a mononuclear complex, which, in the presence of a ligand L, yields substituted derivatives; this process being called photosubstitution. In the case of dimeric and cluster carbonyl complexes, in addition to the labilisation of carbonyl groups, there is the possibility of metal–metal bond cleavage yielding lower nuclearity fragments; this process being called photofragmentation (Fig. 1).

The trinuclear clusters $Ru_3(CO)_{12}$ (**1a**) and $Os_3(CO)_{12}$ (**1b**) serve as prototypes for the photoreactivity of more complex examples. Our studies have shown that, by broad-band UV irradiation of a dichloromethane solution of **1a** in the presence of a two-electron donor ligand, high yields of photofragmentation products can be obtained [2]. This opens a route to substituted

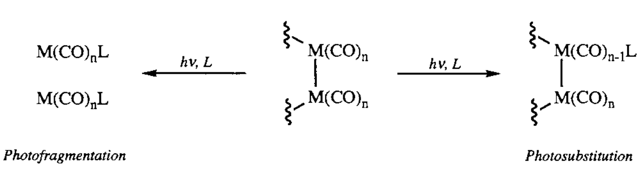

Fig. 1. Photoprocesses in mononuclear and polynuclear metal carbonyls.

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[1] Dedicated to Professor Brian Johnson on the occasion of his 60th birthday.

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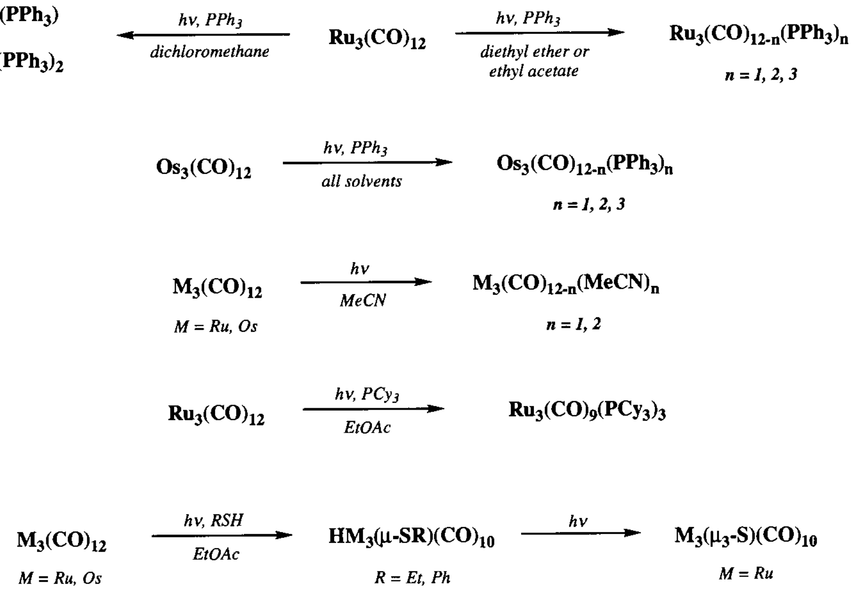

Scheme 1. A summary of the results reported.

mononuclear ruthenium and osmium complexes. Although there has been much work on the synthetic potential of the reactive intermediates generated by photofragmentation of **1a** and **1b**, the corresponding photosubstitution pathway is still relatively unexplored [3]. This work reports that, simply by changing the solvent from dichloromethane to ethyl acetate, ether or acetonitrile, it is possible to prepare photosubstitution products from **1a** and **1b**, thus enabling the synthesis of substituted cluster products.

2. Results and discussion

The results from the investigations are summarised in Scheme 1 and all spectroscopic data are shown in Table 1.

2.1. Photosubstitution in $Ru_3(CO)_{12}$

One method for controlling the outcome of the photolysis of $Ru_3(CO)_{12}$ (**1a**) is to change the wavelength of the incident irradiation. Photophysical studies have shown that irradiation of **1a** in the λ_{max} region (ca. 390 nm) leads essentially to fragmentation, whereas at shorter wavelengths (ca. 320 nm) substitution predominates [4,5]. However, due to the wavelength independence of the photofragmentation quantum yield, it is not possible to obtain quantitative yields of substitution products [6]. As a consequence, our attempts to generate solely photosubstitution products from **1a** have been focused on the control of the photoreactions by variation of the photolysis medium.

By using matrix-isolation and flash-photolysis techniques, previous workers have generated key intermediates involved in the photosubstitution pathway of **1a**, and a mechanism for the photoprocess has been proposed [4,7]. The primary photoreaction is the dissociation of CO to form $[Ru_3(CO)_{11}]$ (**2a**) which is trapped by solvent, S, to generate the solvated species $Ru_3(CO)_{11}S$. To favour the photosubstitution pathway, coordinating solvents must be used and, in addition, a system must be found that quenches the photofragmentation pathway. Tetrahydrofuran (THF) has been shown to act as a quencher of photofragmentation even when added in small volumes to octane photolysis solutions [4,5]. Despite this, our studies have shown that THF can not be used as a solvent for photosubstitution reactions since, rather than obtaining trinuclear products, insoluble red polymers are formed. These polymers have been observed previously and have been shown to have the approximate composition $[Ru(CO)_4]_n$ [8].

Attention turned to diethyl ether, ethyl acetate and acetonitrile as photolysis media. Broad-band UV irradiation of a cooled diethyl ether solution of **1a** with n equivalents of triphenylphosphine leads quantitatively to the formation of $Ru_3(CO)_{12-n}(PPh_3)_n$ [$n = 1$ (**3a**), 2 (**4a**) or 3 (**5a**)]. The same is true of ethyl acetate or acetonitrile solutions of **1a**. Products were purified by thin layer chromatography (TLC) and characterised by comparison of spectral data with that in the literature. Prolonged irradiation of the reaction mixture leads to the formation of traces of the mononuclear complexes $Ru(CO)_4(PPh_3)$ and $Ru(CO)_3(PPh_3)_2$, together with extensive decomposition. This is most probably as a result of secondary photoprocesses.

Table 1
IR and ^{1}H-NMR spectra for complexes prepared

Compound[a]	IR spectrum[b]	^{1}H-NMR spectrum[c]
$Ru_3(CO)_{12}$ **1a**	2059(vs), 2027(s), 2007(m)	– –
$Os_3(CO)_{12}$ **1b**	2068(s), 2035(s), 2014(m), 2002(m)	– –
$Ru_3(CO)_{11}(PPh_3)$ **3a**	2097(m), 2047(s), 2031(sh), 2017(s), 2001(w), 1986(w)	7.48(m)
$Ru_3(CO)_{10}(PPh_3)_2$ **4a**	2072(w), 2060(w), 2047(m), 2034(sh), 2024(s), 1990(s), 1968(s), 1950(m)	7.50(m)
$Ru_3(CO)_9(PPh_3)_3$ **5a**	2056(w), 2044(m), 1967(vs)	7.42(m)
$Os_3(CO)_{11}(PPh_3)$ **3b**	2108(m), 2055(s), 2035(ms), 2019(s), 2000(m), 1989(m), 1978(m), 1956(mw)	7.52(m)
$Os_3(CO)_{10}(PPh_3)_2$ **4b**	2085(mw), 2030(s), 2012(m), 1998(s), 1969(m), 1951(mw)	7.55(m)
$Os_3(CO)_9(PPh_3)_3$ **5b**	2053(w), 1999(sh), 1990(m), 1976(s), 1944(m)	7.63(m)
$Ru_3(CO)_{11}(MeCN)$ **6a**	2098(w), 2045(s), 2037(s), 2021(m), 2001(s), 1994(s), 1972(vw) [d]	2.47(s)[e]
$Ru_3(CO)_{10}(MeCN)_2$ **7a**	2086(w), 2055(sh), 2018(vs), 1999(s), 1987(sh), 1954(m), 1819(mw) [d]	2.63(s)[e]
$Os_3(CO)_{11}(MeCN)$ **6b**	2103(w), 2052(s), 2040(s), 2020(m), 2000(vs), 1984(sh), 1981(m), 1969(w), 1969(w)	2.74(s)
$Os_3(CO)_{10}(MeCN)_2$ **7b**	2077(w), 2025(sh), 2019(vs), 1982(s), 1953(m)	2.86(s)
$Ru(CO)_4(\eta^2\text{-}C_2H_4)$ **8a**	2104(m), 2021(vs), 1995(s)	[f]
$Os(CO)_4(\eta^2\text{-}C_2H_4)$ **8b**	2102(w), 2029(vs),2015(sh), 1995(m)	[f]
$Ru_3(CO)_9(PCy_9)_3$ **9a**	1962(vs), 1945(vs)	2.05 (m)
$HRu_3(CO)_{10}SEt$	2104(s), 2063(vs), 2045(vs), 2020(vs), 2016(vs), 1997(vs), 1982(s)	1.85(m), −17.45 (s)
$HRu_3(CO)_{10}SPh$	2105(s), 2067(vs), 2054(vs), 2021(vs), 2017(vs), 1998(vs), 1988(s)	7.63(m), −14.65 (s)
$HOs_3(CO)_{10}SEt$	2103(s), 2067(vs), 2049(vs), 2023(vs), 2017(vs), 1994(vs), 1978(s)	2.32(m), −16.95 (s)
$HOs_3(CO)_{10}SPh$	2107(s), 2065(vs), 2053(vs), 2021(vs), 2019(vs), 1997(vs), 1981(s)	7.64(m), −15.95 (s)
$Ru_3(\mu_3\text{-}S)(CO)_{10}$	2072(s), 2035(vs), 1735(m)	– –

[a] Compound numbers refer to text.
[b] In CH_2Cl_2 unless stated otherwise.
[c] In $CDCl_3$ unless stated otherwise.
[d] In CH_3CN.
[e] In CD_3CN.
[f] Complex too unstable for NMR.

Photolysis times for the substitution reactions are considerably longer than those for fragmentation reactions. This agrees with previous studies which show that the photogenerated intermediate $[Ru_3(CO)_{11}]$ (**2a**), formed in the photosubstitution pathway, shows a significant reaction selectivity, reaction of **2a** with photoejected CO being eight times faster than with PPh_3 [7]. This may be contrasted with the photofragmentation pathway where the intermediate $[Ru_3(\mu\text{-}CO)(CO)_{11}]$, formed by heterolytic cleavage of a Ru–Ru bond with concomitant movement of a carbonyl from a terminal to a bridging position, is very reactive, and further reaction unselective [4,5].

Although the exact reasoning for why diethyl ether, ethyl acetate and acetonitrile quench the photoprocess is at present unknown, the most plausible explanation is that these solvents (Solv) transform the primary photofragmentation product $[Ru_3(\mu\text{-}CO)(CO)_{11}]$ into $Ru_3(CO)_{11}(Solv)$ [5].

Previous results have shown that the substitution reaction of **3a** with PPh_3 to yield **4a** and **5a** occurs spontaneously at room temperature [1]. A blank experiment where **3a** was stirred in the dark with PPh_3 was carried out to ascertain the effect of light on the reaction. The results showed that, although the reaction does occur thermally, irradiation of the reaction mixture facilitates the substitution process.

Broad-band UV irradiation of an acetonitrile solution of **1a** with no added ligand leads to the formation of the mono- and bis-acetonitrile substituted clusters $Ru_3(CO)_{11}(MeCN)$ (**6a**) and $Ru_3(CO)_{10}(MeCN)_2$ (**7a**). The ratio of **6a** to **7a** is dependent on the irradiation time, short photolyses leading to the formation of **6a** and longer photolyses to **7a**. As before, products were purified by TLC and characterised by comparison of spectral data with that in the literature. Prolonged photolysis of the reaction mixture leads to extensive decomposition.

This offers a simple and high yield route to the highly versatile acetonitrile substituted clusters, prepared previously using trimethylamine-*N*-oxide (Me_3NO) as an oxidant [9]. The main disadvantage of the amine oxide route to **6a** and **7a** are that it is hard to control the oxidation reaction and hence obtaining a pure sample of **7a** without any **6a** being present is very difficult. In addition, **6a** and **7a** are highly reactive and hence are difficult to purify. This has the drawback that, in subsequent reactions, a trace of Me_3NO left in the starting material mixture can affect the reaction chemistry of **6a** and **7a** leading either to unwanted by-products or totally altering the course of the reaction [10]. The photochemical path to **6a** and **7a** reported here is significant since the clusters can be prepared selectively by control of the photolysis time and are generated without the use of any oxidant.

The quenching of the photofragmentation of **1a** when diethyl ether or ethyl acetate are used as photolysis media is confirmed by the observation that, when an ethene-saturated solution of **1a** in these solvents is irradiated, no net photoreaction is observed. A photofragmentation pathway would lead to the easily characterised η^2-alkene complex $Ru(CO)_4(\eta^2\text{-}C_2H_4)$ (**8a**), whereas a substitution pathway is more complex but, ultimately, no net photoreaction would be observed. As with the case of triphenylphosphine, labilisation of a CO group from **1a** leads to the unsaturated intermediate $[Ru_3(CO)_{11}]$ (**2a**) which, after being solvent stabilised, would react either with photoejected CO to regenerate **1a** or, in this case, with ethene to produce the previously characterised η^2-alkene complex $Ru_3(CO)_{11}(\eta^2\text{-}C_2H_4)$ (**9a**) [1]. The selectivity of **2a** would have the consequence that back reaction with CO would be favoured greatly over reaction with ethene therefore making the formation of **9a** a highly inefficient process. In addition, **9a** is highly unstable and reacts with CO in solution to regenerate **1a**. As a result, no net photoreaction is observed under the present experimental conditions. Acetonitrile could not be used in the ethene studies since any results could be misleading. It would not be possible to determine whether products formed resulted from a photoreaction between **1a** and ethene or from secondary reactions, **1a** reacting with the solvent to form **6a** or **7a** before reacting with the olefin.

2.2. Photosubstitution in $Os_3(CO)_{12}$

Broad-band UV irradiation of a diethyl ether, ethyl acetate or acetonitrile solution of $Os_3(CO)_{12}$ **1b** in the presence of *n* equivalents of triphenylphosphine leads to the quantitative formation of $Os_3(CO)_{12-n}(PPh_3)_n$ [$n =$ 1 (**3b**), 2 (**4b**) or 3 (**5b**)]. These results can be compared with those where hydrocarbon solvents are used as the photolysis medium [11]. In the case of hexane, sequential substitution occurs to yield firstly **3b,** then **4b,** and then **5b**. After this, photofragmentation occurs to yield $Os(CO)_3(PPh_3)_2$. This reflects the previous observation that, although structurally similar, $Ru_3(CO)_{12}$ **1a** and $Os_3(CO)_{12}$ **1b** are significantly different electronically [12]. The metal–metal orbital interactions are greater in **1b** than in **1a** with the consequence that photofragmentation is much less efficient in **1b**. It is concluded that, with the donor solvents used in the present studies, it may not be the photofragmentation of **1b** that is quenched but rather that of **5b**.

The broad-band UV irradiation of an acetonitrile solution of **1b** with no added ligand leads to the generation of the acetonitrile substituted trinuclear clusters $Os_3(CO)_{11}(MeCN)$ (**6b**) and $Os_3(CO)_{10}(MeCN)_2$ (**7b**), analogous to the case of **1a**.

Broad-band UV irradiation of an ethene-saturated diethyl ether or ethyl acetate solution of **1b** leads to the formation of the mononuclear η^2-alkene complex $Os(CO)_4(\eta^2\text{-}C_2H_4)$ (**8b**). This is analogous to the case of irradiation of **1b** in ethene-saturated hydrocarbon solutions thereby confirming that the mechanisms for photosubstitution of **1a** and **1b** are significantly different [13].

2.3. Synthesis of $Ru_3(CO)_9(PCy_3)_3$

Broad-band UV photolysis of a diethyl ether solution of $Ru_3(CO)_{12}$ containing a 10-fold excess of tricyclohexylphosphine leads to the generation of the highly sterically crowded tris-substituted cluster $Ru_3(CO)_9(PCy_3)_3$ (**9a**) in 50% yield. This complex has been inaccessible using conventional routes until very recently [14], the photochemical route being only the second report of the synthesis of **9a**. The fact that **9a** can be formed photochemically demonstrates the potential of photolysis as a synthetic route to organometallic complexes and shows how it can both compliment and augment conventional synthetic routes.

2.4. Synthesis of $HM_3(\mu\text{-}SR)(CO)_{10}$ [$M = Ru$, Os; $R = Et$, Ph]

Broad-band UV photolysis of an ethyl acetate solution of $M_3(CO)_{12}$ [$M = Ru$, Os] containing a 10-fold excess of RSH [$R = Et$, Ph] leads initially to the formation of the thiolato complex $HM_3(\mu\text{-}SR)(CO)_{10}$ in good yield (60–75%). This represents an improved synthetic route to this class of substituted cluster, previous synthetic routes involving more severe reaction conditions [15]. Prolonged photolysis of the reaction mixtures of $Ru_3(CO)_{12}$ and RSH leads, as already reported, to the previously characterised sulphido complexes $Ru_3(\mu_3\text{-}S)(CO)_{10}$ [16,17]. The sulphido complexes are easily recognised by their characteristic μ_3 bridging carbonyl group, this being observed in the infrared (IR) spectrum at around 1700 cm^{-1}.

3. Experimental section

All reagents were used as obtained from the suppliers. All solvents were dried and degassed prior to use. Literature methods were used to prepare $Ru_3(CO)_{12}$

(**1a**) [18] and $Os_3(CO)_{12}$ (**1b**) [19] from $RuCl_3$ and OsO_4, respectively. IR spectra were recorded using a Perkin–Elmer PE 1710 Fourier transform IR spectrometer in NaCl solution cells (path length 0.5 mm). The ^1H-NMR spectra were recorded using a Bruker WM250 Fourier-transform NMR spectrometer and data reported using the chemical shift scale in units of ppm relative to the solvent resonance. Routine separation of products was carried out by TLC, using commercially prepared glass plates, precoated to a thickness of 0.25 mm with Merk Kieselgel 60 F_{254} as supplied by Merk. Alternatively, laboratory-prepared glass plates, coated to a thickness of 1.0 mm with Merk Kieselgel 60 F_{254}, were used.

All photochemical reactions were carried out in a specially designed glass reaction vessel fitted with a nitrogen bubbler, reflux condenser and dry-ice cooling finger. A 125-W mercury arc broad-band UV lamp was used as the irradiation source and reflectors placed around the reaction vessel to maximise efficiency. Photolysis mixtures were kept at between 2 and −2°C.

All spectroscopic data are shown in Table 1.

3.1. Photolyses

3.1.1. Diethyl ether, ethyl acetate and acetonitrile solutions of $M_3(CO)_{12}$ containing PPh_3

To a diethyl ether solution of **1a** (30 mg, 4.7 mmol in 100 ml) was added one equivalent of triphenylphosphine (12 mg, 4.7 mmol) and the reaction mixture irradiated. The reaction was monitored by IR spectroscopy and was deemed complete when there was no further change in the spectrum (1 h). The substituted cluster **3a** was formed in quantitative yield. Repeating the reaction with two (24 mg, 9.4 mmol) and three (36 mg, 14.1 mmol) equivalents of PPh_3 yielded **4a** and **5a**, respectively, as the only photoproducts, the photolysis times being 1 and 1.5 h, respectively. Analogous results were obtained with ethyl acetate and acetonitrile. Products were separated by TLC (1:1 dichloromethane:hexane as eluent) and characterised by comparison of spectral data with that in the literature [20].

In the case of **1b**, the substitution products **3a**, **4b** and **5b** were formed on photolysis in ethyl acetate, diethyl ether and acetonitrile with one, two and three equivalents of PPh_3, respectively. Photolysis times were 1, 2 and 3 h, respectively.

3.1.2. An acetonitrile solution of $M_3(CO)_{12}$

Photolysis of an acetonitrile solution of **1a** (30 mg, 4.7 mmol in 100 ml) for 1 h led to the formation of $Ru_3(CO)_{11}(MeCN)$ (**6a**) in approximately 75% yield. Continuing the photolysis for a further 1 h led to $Ru_3(CO)_{10}(MeCN)_2$ (**7a**) in 65% yield. In the case of **1b**, the substituted cluster **6b** was formed in 70% yield after 1 h photolysis. Continuing the photolysis for a further 1 h lead to **7b** in 60% yield. Products were separated by TLC (35:60:5 dichloromethane:hexane:acetonitrile as eluent) and characterised by comparison of spectral data with that in the literature [9].

3.1.3. An ethene-saturated diethyl ether or ethyl acetate solution of $Ru_3(CO)_{12}$ (1a)

Photolysis of an ethene-saturated diethyl ether or ethyl acetate solution of **1a** (30 mg, 4.7 mmol in 100 ml) for 2 h showed no net reaction.

3.1.4. An ethene-saturated diethyl ether or ethyl acetate solution of $Os_3(CO)_{12}$ (1b)

The method was as for the case of **1a** but using **1b** as the starting cluster. The mononuclear complex $Os(CO)_4(\eta^2\text{-}C_2H_4)$ (**8b**) was formed slowly, the reaction taking 3 h to reach a 1:1 ratio of **1b** to **8b**.

3.1.5. An ethyl acetate solution of $Ru_3(CO)_{12}$ containing PCy_3

To an ethyl acetate solution of **1a** (30 mg, 4.7 mmol in 100 ml) was added a large excess (ten equivalents) of tricyclohexylphosphine (39 mg, 14.1 mmol) and the reaction mixture irradiated. The reaction was monitored by IR spectroscopy and was deemed complete when there was no further change in the spectrum (1 h). The reaction mixture was purified by TLC (1:1 dichloromethane:hexane as eluent), the substituted cluster **9a** being recovered in 50% yield.

3.1.6. An ethyl acetate solution of $M_3(CO)_{12}$ [M = Ru, Os] containing RSH [R = Et, Ph]

To an ethyl acetate solution of **1a** (30 mg, 4.7 mmol in 100 ml) was added a large excess (ten equivalents) of ethane thiol and the reaction mixture irradiated. The reaction was monitored by IR spectroscopy and was deemed complete when there was no further change in the spectrum (1 h). The reaction mixture was purified by TLC (1:1 dichloromethane:hexane as eluent), the substituted cluster $HM_3(\mu\text{-SEt})(CO)_{10}$ being recovered in 75% yield. The reaction was repeated using **1b** in the place of **1a** and using phenyl thiol in the place of EtSH. On prolonged photolysis, the $HRu_3(\mu\text{-SR})(CO)_{10}$ [R = Et, Ph] complexes yielded $Ru_3(\mu\text{-S}_3)(CO)_{10}$ in 50% yield.

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