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(Dialkylsulfide)(arene)ruthenium(II) derivatives

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Abstract

The preparations are reported of neutral and cationic (dialkylsufide)(η^6-arene)ruthenium(II) complexes of the types (η^6-arene)$Cl_2Ru(SR_2)$, $[(\eta^6\text{-arene})ClRu(SR_2)_2]^+$ and $[(\eta^6\text{-arene})ClRu(SR_2)(L)]^+$. These complexes containing labile R_2S–Ru bonds can be used in the ready generation of 16 electron ruthenium moieties for the activation of terminal alkynes and access to cationic (carbene)(η^6-arene)ruthenium(II) derivatives such as $\{(1,3,5\text{-}Me_3C_6H_3)ClRu(SMe_2)[C(OMe)CH_2Ph]\}^+$ and $\{(C_6Me_6)ClRu(PMe_3)[C(OMe)CH_2Ph]\}^+$.

Introduction

Studies of the coordination chemistry of organic sulfides have shown their synthetic utility in processes involving the cleavage of a carbon–sulfur bond from allylic sulfides [1] or ortho-metallation of aryl sulfides [2]. Particularly relevant is that simple adducts of dialkylsulfides with transition metal halides are key intermediates in numerous organometallic syntheses [3]. Since (η^6-arene)ruthenium(II) complexes have been shown to act as catalyst precursors for the activation of terminal alkynes [4–6], we decided to undertake a study of complexes involving coordination of various dialkyl sulfides on (η^6-arene)Ru(II) moieties with the aim of modifying the reactivity of the ruthenium site.

We describe here selective routes to three types of complexes: neutral (η^6-arene)$Cl_2Ru(SR_2)$ (**1**), cationic $[(\eta^6\text{-arene})ClRu(SR_2)_2]^+$ (**2**) and $[(\eta^6\text{-arene})ClRu(SR_2)(L)]^+$ (**3**). Complexes **2** and **3** readily undergo displacement of the sulfide ligand by phenylacetylene, allowing the synthesis of (carbene)(η^6-arene)ruthenium (II) derivatives **4** in the presence of methanol.

Results and discussion

The formation of neutral complexes (η^6-arene)$Cl_2Ru(L)$ by reaction of a two electron donor ligand L with the complexes $[(\eta^6\text{-arene})ClRu(\mu\text{-}Cl)]_2$ is well known [7], and occurs with various ligands L. However, little is known about the behaviour

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when L is a dialkyl sulfide, since only two (η^6-C_6H_6)(diethylsulfide)ruthenium derivatives have been described [8].

When a suspension of the almost insoluble complex [(1,3,5-$Me_3C_6H_3$)$Cl_2Ru]_2$ [9] in dichloromethane with two-fold excess of dimethyl sulfide and stirring for several hours leads to a red solution, which gives red crystals of **1a** (74%) upon the addition of diethyl ether. The same reaction occurs with tetrahydrothiophene $\overline{S(CH_2)_3CH_2}$ or trimethylene sulfide $\overline{S(CH_2)_2CH_2}$ and several adducts **1b–1f** have been obtained in good yield (60–80%) according to equation 1.

$$[(\eta^6\text{-arene})ClRu(\mu\text{-}Cl)]_2 + 2\,SR_2 \rightleftharpoons 2\,(\eta^6\text{-arene})Ru(Cl)_2SR_2 \quad (1)$$

(1)

	arene	SR_2
1a	1,3,5-$Me_3C_6H_3$	SMe_2
1b	*p*-$MeC_6H_4CHMe_2$	SMe_2
1c	C_6Me_6	SMe_2
1d	1,3,5-$Me_3C_6H_3$	$S(CH_2)_4$
1e	*p*-$MeC_6H_4CHMe_2$	$S(CH_2)_4$
1f	*p*-$MeC_6H_4CHMe_2$	$S(CH_2)_3$

The complexes **1** were isolated as red crystals which gave satisfactory analyses. Attempts to recrystallize them without the presence of an approximately equimolar amount of the relevant free sulfide SR_2 resulted in the partial recovery of the dimeric starting material, indicating the weakness of the ruthenium–sulfur bond. Moreover, no reaction was observed with the bulkier S^tBu_2 sulfide. Complexes **1** were fully characterized by elemental analysis (Table 1) and ^1H NMR spectroscopy (Table 2).

The cationic complexes **2a–2e** were initially obtained by treating the corresponding complexes **1** with an excess of the sulfide ligand in the presence of $NaPF_6$ or NH_4PF_6 in a polar solvent (acetone or methanol), but it was found to be more convenient to start from the dimeric precursors according to equation 2.

$$[(\eta^6\text{-arene})ClRu(\mu\text{-}Cl)]_2 + 4SR_2 + 2MPF_6 \rightarrow 2[(\eta^6\text{-arene})ClRu(SR_2)_2]^+(PF_6^-) + 2MCl \quad (2)$$

(2)

(M = Na, NH_4)

	arene	SR_2
2a	(1,3,5-$Me_3C_6H_3$)	SMe_2
2b	(*p*-$MeC_6H_4CHMe_2$)	SMe_2
2c	(1,3,5-$Me_3C_6H_3$)	$S(CH_2)_4$
2d	C_6Me_6	$S(CH_2)_4$
2e	C_6Me_6	$S(CH_2)_3$

Table 1

Analytical data for the arene-ruthenium complexes

Complex	Analyses (Found (calcd.) (%))				
	C	H	Cl	S	P
$(1,3,5\text{-}Me_3C_6H_3)Cl_2Ru(SMe_2)$	37.27	5.58	19.98	8.73	
(1a)	(37.29)	(5.12)	(20.01)	(9.05)	
$(p\text{-}MeC_6H_4CHMe_2)Cl_2Ru(SMe_2)$	39.25	5.30	19.65	8.39	
(1b)	(39.13)	(5.47)	(19.25)	(8.71)	
$(C_6Me_6)Cl_2Ru(SMe_2)$	42.53	5.89	17.90	6.83	
(1c)	(42.42)	(6.10)	(17.89)	(8.09)	
$(1,3,5\text{-}Me_3C_6H_3)Cl_2RuS(CH_2)_4$	41.17	5.40	18.98	8.58	
(1d)	(41.05)	(5.30)	(18.64)	(8.43)	
$(p\text{-}MeC_6H_4CHMe_2)Cl_2RuS(CH_2)_4$	43.27	5.25	18.27	6.95	
(1e)	(42.64)	(5.62)	(17.98)	(8.13)	
$(p\text{-}MeC_6H_4CHMe_2)Cl_2RuS(CH_2)_3$	41.12	5.40	18.68	8.40	
(1f)	(41.05)	(5.30)	(18.64)	(8.43)	
$[(1,3,5\text{-}Me_3C_6H_3)ClRu(SMe_2)_2](PF_6)$	29.78	4.08	7.03	11.29	5.71
(2a)	(29.69)	(4.60)	(6.74)	(12.19)	(5.89)
$[(p\text{-}MeC_6H_4CHMe_2)ClRu(SMe_2)_2](PF_6)$	31.03	4.56	6.92	11.70	5.64
(2b)	(31.14)	(4.85)	(6.57)	(11.88)	(5.74)
$[(1,3,5\text{-}Me_3C_6H_3)ClRu[S(CH_2)_4]_2](PF_6)$	35.51	4.84	6.55	11.17	5.34
(2c)	(35.33)	(4.88)	(6.13)	(11.09)	(5.36)
$[(C_6Me_6)ClRu[S(CH_2)_4]_2](PF_6)$	38.54	5.61	6.17	9.79	3.35
(2d)	(38.74)	(5.53)	(5.72)	(10.34)	(4.99)
$[(C_6Me_6)ClRu[S(CH_2)_3]_2](PF_6)$	37.29	4.99	5.77	11.76	5.16
(2e)	(36.52)	(5.11)	(5.99)	(10.83)	(5.23)
$[(1,3,5\text{-}Me_3C_6H_3)ClRu(SMe_2)(PMe_3)](PF_6)$	31.32	5.11	6.55	5.81	12.09
(3a)	(31.15)	(5.04)	(6.57)	(5.94)	(11.47)
$[(p\text{-}MeC_6H_4CHMe_2)ClRu(SMe_2)(PMe_3)](PF_6)$	32.40	5.35	6.53	8.58	10.21
(3b)	(32.53)	(5.28)	(6.40)	(5.79)	(11.18)
$[(C_6Me_6)ClRu(SMe_2)(PMe_3)](PF_6)$	35.12	5.71	6.23	5.59	10.70
(3c)	(35.09)	(5.72)	(6.09)	(5.51)	(10.64)
$[(p\text{-}MeC_6H_4CHMe_2)ClRu[S(CH_2)_4](PMe_3)](PF_6)$	35.14	5.46	5.95	5.85	8.33
(3d)	(35.21)	(5.39)	(6.11)	(5.53)	(10.68)
$[(1,3,5\text{-}Me_3C_6H_3)ClRu(SMe_2)[C(OMe)CH_2Ph]](PF_6)$	40.00	4.75	6.05	5.99	4.98
(4a)	(40.17)	(4.72)	(5.93)	(5.36)	(5.18)

Table 2

^{1}H and ^{31}P NMR [a] data for the arene-ruthenium complexes

Complex (yield %)	^{1}H NMR, δ			^{31}P NMR, δ
	Arene	Sulfide ligand	Other ligand	
1a (74)	5.00 s, 3H, C_6H_3 2.22 s, 9H, CH_3	2.29 s, 6H, CH_3		
1b (80)	5.39 AB, 4H, C_6H_4 $^{3}J(HH) = 6.1$ Hz 3.01 m, 1H, $CHMe_2$ 2.26 s, 3H, CH_3Ar 1.33 d, 6H, $(CH_3)_2C$ $^{3}J(HH) = 6.8$ Hz	2.29 s, 6H, CH_3		
1c	2.08 s, 18H, CH_3	2.17 s, 6H, CH_3		
1d (55)	4.98 s, 3H, C_6H_3 2.22 s, 9H, CH_3	3.04 m, 4H, SCH_2 2.00 m, 4H, $(CH_2)_2$		
1e (76)	5.37 AB, 4H, C_6H_4 $^{3}J(HH) = 5.9$ Hz 2.25 s, 3H, CH_3Ar 1.33 d, 6H, $(CH_3)_2C$ $^{3}J(HH) = 7.1$ Hz	3.04 m, 4H, SCH_2 2.00 m, 4H, $(CH_2)_2$		
1f	5.35 AB, 4H, C_6H_4 $^{3}J(HH) = 6.1$ Hz 2.24 s, 3H, CH_3Ar 1.31 d, 6H, $(CH_3)_2C$ $^{3}J(HH) = 6.8$ Hz	2.92 m (broad), 6H, $S(CH_2)_3$		
2a (70)	5.40 s, 3H, C_6H_3 2.26 s, 9H, CH_3	2.39 s, 12H, CH_3		
2b (55)	5.68 s, 4H, C_6H_4 2.81 m, 1H, $CHMe_2$ 2.23 s, 3H, CH_3Ar 1.30 d, 6H, $(CH_3)_2C$ $^{3}J(HH) = 6.8$ Hz	2.45 s, 12H, CH_3		
2c (90)	5.46 s, 3H, C_6H_3 2.23 s, 9H, CH_3	3.04 m, 8H, SCH_2 2.09 m, 8H, $(CH_2)_2$		
2d (60)	2.10 s, 18H, CH_3	2.83 m, 8H, SCH_2 2.05 m, 8H, $(CH_2)_2$		
2e	2.06 s, 18H, CH_3	3.41 m, 8H, SCH_2 2.88 m, 4H, CH_2		
3a (55)	5.63 s, 3H, C_6H_3 2.26 s, 9H, CH_3	2.52 s, 6H, CH_3	1.62 d, 9H, PMe_3 $^{2}J(PH) = 10.7$ Hz	3.5
3b (76)	5.91 s, 4H, C_6H_4 2.73 m, 1H, $CHMe_2$ 2.12 s, 3H, CH_3Ar 1.25 dd, 6H, $(CH_3)_2C$ $^{3}J(HH) = 6.8$ Hz	2.56 s, 6H, CH_3	1.64 d, 9H, PMe_3 $^{2}J(PH) = 11.0$ Hz	4.2
3c (77)	2.05 d, 18H, CH_3 $J(PH) \approx 1.0$ Hz	2.23 s, 6H, CH_3	1.55 d, 9H, PMe_3 $^{2}J(PH) = 10.3$ Hz	1.9
3d (47)	5.93–5.48 m, 4H, C_6H_4 2.16 s, 3H, CH_3Ar 1.30 d, 3H, $CH_3(^{i}Pr)$ $^{3}J(HH) = 6.8$ Hz 1.28 d, 3H, $CH_3(^{i}Pr)$ $^{3}J(HH) = 7.1$ Hz	3.08 m, 4H, SCH_2 2.23 m, 4H, $(CH_2)_2$	1.68 d, 9H, PMe_3 $^{2}J(PH) = 10.7$ Hz	3.0
4a (50)	5.21 s, 3H, C_6H_3 2.13 s, 9H, CH_3	2.21 s, 6H, CH_3	7.34 s, 5H, Ph 4.85 s, 5H, $OCH_3 + CH_2$	

[a] Complexes **1a–1f**, **3a** and **3b** in $CDCl_3$; complexes **2a–2e**, **3c–3d** and **4a** in CD_2Cl_2; 297 K; 80 MHz (^{1}H) and 32.38 MHz (^{31}P) (Resonances of the PF_6 anion are omitted).

Owing to the lability of the sulfide ligand, phosphines such as PMe_3 react readily with complexes **1** by ligand exchange to give the adducts (η^6-arene)$Cl_2Ru(PMe_3)$, which are usually obtained directly by addition of the phosphine to [(η^6-arene)$Cl_2Ru]_2$ complexes [7]. Consequently, the unsymmetrical cationic derivatives **3a–3b** were prepared from (arene)$RuCl_2PR_3$ complexes by addition of a slight excess of the sulfide in the presence of NH_4PF_6, according to equation 3.

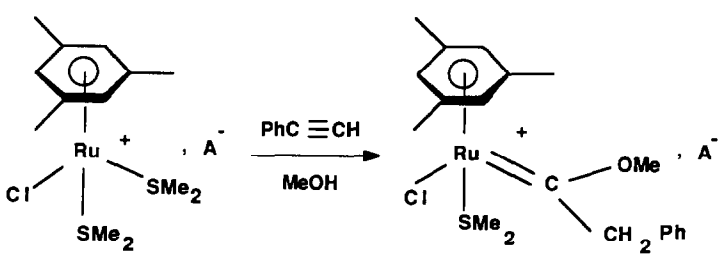
$$(\eta^6\text{-arene})Cl_2Ru(PMe_3) + SR_2 + NH_4PF_6 \rightarrow \underset{(3)}{\left[(\eta^6\text{-arene})ClRu(PMe_3)(SR_2)\right](PF_6)} + NH_4Cl \qquad (3)$$

	arene	SR_2
3a	1,3,5-$Me_3C_6H_3$	SMe_2
3b	*p*-$MeC_6H_4CHMe_2$	SMe_2
3c	C_6Me_6	SMe_2
3d	*p*-$MeC_6H_4CHMe_2$	$S(CH_2)_4$

The derivatives **2** and **3** were obtained in good yields (60–80%) as bright orange crystals after crystallization from dichloromethane upon addition of diethyl ether, and were found to be stable in solution and in the solid state. They were characterized by elemental analysis (Table 1) and ^1H NMR spectroscopy (Table 2). In addition, complexes **3** exhibit in their ^{31}P NMR spectra (Table 2) a single resonance in the range $\delta = 1.9$–4.2 ppm coming from PMe_3 group.

It has been shown previously that (η^6-C_6Me_6)$RuCl_2PR_3$ complexes can activate terminal alkynes via a ruthenium-vinylidene intermediate to produce carbene-ruthenium complexes [10], and this was attributed to the lability of Ru–Cl bond and the electron richness of the ruthenium center. The reaction of **2a** containing the SMe_2 ligand with phenylacetylene in the presence of methanol was thus been investigated.

After only 1 h at room temperature the new carbene-ruthenium complex **4a** containing a SR_2 group was isolated in 50% yield (Scheme 1).



2a **4a**

A = PF_6

Scheme 1

The formation of **4a** shows that one Ru–SMe_2 bond in **2a** is labile and that the remaining SR_2 ligand is sufficiently electron-releasing to stabilize the Ru–carbene bond. The formation of the carbene ligand probably results from a (η^2-PhC≡CH)Ru → (η^1-PhCH=C=Ru) rearrangement followed by addition of methanol to the electrophilic vinylidene carbon. This reaction shows that the $(R_2S)Ru^+$ moiety is able to activate terminal alkynes toward alcohol, in contrasts with the behaviour of the thiolato-metal species [η^2-Ph_2PCH=C(R)S]M^+, which undergoes coupling of the terminal alkyne with the sulfur (M=Fe [11]) or a carbon (M=Ru [12]) atom of the chelating ligand.

The reaction of the cationic complex **3c** with phenylacetylene under similar conditions also leads to the displacement of the SR_2 ligand to afford the carbene complex **4b** (65%) (Eq. 4). The latter was obtained previously from (η^6-C_6Me_6)-$RuCl_2PMe_3$ [10].

$$[(C_6Me_6)ClRu(PMe_3)(SMe_2)](PF_6) + PhC{\equiv}CH + MeOH \rightarrow$$

$$\underset{\textbf{(4b)}}{\{(C_6Me_6)ClRu(PMe_3)[C(OMe)CH_2Ph]\}(PF_6)} + SMe_2 \qquad (4)$$

The methoxycarbene-ruthenium complex **4b** was identified by comparison with an authentic sample (^1H and ^{31}P NMR, IR) [10]. The methoxycarbene-ruthenium complex **4a** is an example of a stable Ru(carbene)(dialkyl sulfide) derivative containing the rather poor (relative to a phosphine) electron donor sulfide ligand.

The ^1H NMR spectrum of **4a** (Table 2) shows at room temperature singlets for the SMe_2 protons and the methylene protons. At low temperature (190 K, CD_2Cl_2, 300 MHz) the two methyl groups of the sulfide ligand have become non-equivalent, and two resonances are observed at $\delta = 2.47$ and 1.87 ppm, and the protons of the methylene group give an AB system ($\delta = 5.01$ and 4.75 ppm, $^2J(HH) = 16.3$ Hz). Both non-equivalencies are due to the chirality at the ruthenium center.

Table 3

$^{13}C\{^1H\}$ NMR [a] data for the complexes **1a**, **2a** and **4a**

Complex	T (K)	δ		
		Arene	Sulfide ligand	Carbene ligand
1a	297	101.3 s, CMe 80.5 s, CH 18.5 s, CH_3	22.8 s, CH_3	
2a	297	105.5 s, CMe 85.9 s, CH 18.7 s, CH_3	24.1 s, CH_3	
4a	297	116.6 s, CMe 87.0 s, CH 19.1 s, CH_3		329.3 s, CRu 133.0–128.3 m, C_6H_5 70.1 s, OCH_3 63.8 (broad) CH_2
4a	193	116.3 s, CMe 86.1 s, CH 19.5 s, CH_3	28.1 s, CH_3 22.2 s, CH_3	327.8 s, CRu 134.0–128.1 m, C_6H_5 70.5 s, OCH_3 65.4 s, CH_2

[a] Complex **1a** in $CDCl_3$, complexes **2a** and **4a** in CD_2Cl_2; 75.469 MHz.

The $^{13}C\{^1H\}$ NMR spectrum of **4a** can be compared with those of **1a** and **2a** (Table 3). There are only broad resonances for the carbon nuclei of the sulfide ligand and the methylene group at 297 K indicating fluxionality of the $RuSMe_2$ moiety. The $^{13}C\{^1H\}$ spectrum at a lower temperature (190 K) is well resolved, and shows two inequivalent methyl groups for the sulfide ligand. These observations suggest that these two modes of rotation are linked.

The present study of dialkylsulfide-ruthenium derivatives shows that the lability of the R_2S–Ru bond can be used for the easy generation of 16 electron ruthenium species under very mild conditions and that the R_2S–Ru^{II} species is as able as a R_3P–Ru^{II} intermediate [10,13] to activate terminal alkynes.

Experimental

All manipulations were performed under an inert atmosphere by Schlenk techniques. Solvents were dried by conventional methods. NMR spectra were recorded on Bruker WP 80 and AM300 spectrometers, and analyses were performed by the "Service de Microanalyse du CNRS" Vernaison, France.

The starting products $[(\eta^6\text{-arene})RuCl_2]_2$ (arene = *p*-$MeC_6H_4CHMe_2$; C_6Me_6 [14]; 1,3,5-$Me_3C_6H_3$ [9]) were prepared by published methods, and $RuCl_3 \cdot 3H_2O$ was purchased (Johnson–Matthey).

Complexes $(\eta^6\text{-arene})Cl_2Ru(SR_2)$ (1)

In a typical run, 3.00 g (5.14 mmol) of $[(1,3,5\text{-}Me_3C_6H_3)RuCl_2]_2$ and 1.0 mL (13.6 mmol) of dimethylsulfide were stirred with 40 mL of CH_2Cl_2 for 10 h at room temperature. The resulting dark red solution was filtered and the filtrate covered with a layer of 170 mL of diethyl ether. The red crystals of **1a** which resulted from the slow diffusion of ether were decanted, washed twice with 20 mL of diethyl ether, and dried under vacuum (2.69 g, 74%).

This procedure gave the red crystalline complexes **1b**–**1f** in 60–80% yields.

Complexes $[(\eta^6\text{-arene})ClRu(SR_2)_2](PF_6)$ (2)

In a typical run, 1.00 g (1.71 mmol) of $[(1,3,5\text{-}Me_3C_6H_3RuCl_2]_2$, 0.60 g (3.57 mmol) of $NaPF_6$ and 1.0 mL (13.6 mmol) of dimethyl sulfide were stirred with 40 mL of acetone for two days at room temperature. The resulting yellow mixture was evaporated to dryness and the residue extracted with 20 mL of CH_2Cl_2. The extract was filtered and the orange filtrate covered with a layer of 80 mL of diethyl ether. The orange crystals resulting from the slow diffusion of ether were separated by decantation of the solvent, washed twice with 20 mL of diethyl ether, and dried under vacuum (0.74 g, 70%).

This procedure give the orange crystalline complexes **2b**–**2e** in 60–90% yields.

Complexes $[(\eta^6\text{-arene})ClRu(PMe_3)(SR_2)](PF_6)$ (3)

In a typical procedure, 0.70 g (1.90 mmol) of $(1,3,5\text{-}Me_3C_6H_3Cl_2Ru(PMe_3)$, 0.31 g (1.90 mmol) of NH_4PF_6 and 0.30 mL (4.1 mmol) of dimethyl sufide were stirred with 25 mL of methanol for 24 h at room temperature. The resulting yellow slurry was evaporated to dryness and the residue extracted with 20 mL of CH_2Cl_2. The solution was filtered and the orange filtrate covered with 80 mL of diethyl ether. The orange crystals resulting from the diffusion of ether were isolated by decanta-

tion of the solvents, washed twice with 20 mL of diethyl ether, and dried under vacuum (0.46 g, 55%).

This procedure gave the orange crystalline complexes **3b–3d** in 50–80% yields.

Complexes {(η^6-arene)Cl(L)Ru[C(OMe)CH_2Ph]}(PF_6) (4) and {(1,3,5-$Me_3C_6H_3$)-ClRu(SMe_2)[C(OMe)CH_2Ph]}(PF_6) (4a)

A mixture of 0.53 g (1.0 mmol) of [(1,3,5-$Me_3C_6H_3$)ClRu(SMe_2)$_2$](PF_6) (**2a**) and 0.20 mL (1.8 mmol) of phenylacetylene were stirred for 1 h with 50 mL of methanol. (Decomposition occurs at longer reaction times.) The solution was reduced in volume to 20 mL and cooled to −20 °C. The yellow precipitate was fillered off on a sintered-glass frit and dried under vacuum. Recrystallization from dichloromethane (20 mL)/diethyl ether (60 mL) mixture at −20 °C afforded orange crystals of **4a** (0.30 g, 50%).

{(C_6Me_6)ClRu(PMe_3)[C(OMe)CH_2Ph]}(PF_6) (4b)

Complex **4b** was obtained in 65% yield by the procedure used for **4a**, by reaction of **3c** with phenylacetylene and $NaPF_6$ in methanol.

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