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Synthesis and reactivity of rhenium carbonyl thiolate complexes, $Et_4N[Re_2(\mu\text{-}SR)_3(CO)_6]$

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Abstract

Compounds of the formula $Et_4N[Re_2(\mu\text{-}SR)_3(CO)_6]$, (R = Me, Bu^t, Ph, C_6F_5) may be prepared in good yields from reactions between various halocarbonylrhenate anions and either the thiolate anion or $(n\text{-}Bu)_3SnSR$. These stable, white or off-white, crystalline species undergo reactions with $[Me_3O]BF_4$ in the presence of phosphines to form *cis*-$Re_2(\mu\text{-}SR)_2(CO)_6(L)_2$ (L = PMe_3, PPh_3, 1/2 dppm). When heated in benzene, these compounds are converted to the *trans* isomers; in contrast, when they are heated in tetrahydrofuran, carbon monoxide is lost and the species $Re_2(\mu\text{-}SR)_2(\mu\text{-}CO)(CO)_4(L)_2$ are isolated as products.

Infrequent references to organometallic rhenium complexes with thiolate ligands are found in the literature over the last 20 years [1*]. The most common of the known species have formulas $Re(SR)(CO)_5$, $Re_2(\mu\text{-}SR)_2(CO)_8$ and $Re_4(\mu\text{-}SR)_4(CO)_{12}$ (R = alkyl, aryl groups). These compounds have been prepared by what are now fairly standard methods in metal carbonyl thiolate chemistry, reactions of $ReX(CO)_5$ (X = Cl, Br, I) or $ReH(CO)_5$ with thiols or organotin thiolates. Structures and properties are those anticipated for 18 electron metal species. Anionic thiolate-bridged complexes of the formula given in the title of this paper were unknown prior to this work, although a number of structurally analogous complexes with different bridging groups have been described including the species $Et_4N[Re_2(\mu\text{-}X)_3(CO)_6]$, with X = Cl, Br, I [2], H, EtO, i-PrO [3].

In 1985, we reported on the synthesis and reactivity of complexes of the formula $Et_4N[Mn_2(\mu\text{-}SR)_3(CO)_6]$ (R = Me, t-Bu, Ph) in this journal [4]; this work on the rhenium analogues of these compounds is a continuation of this earlier research project. In this paper, we describe first the synthesis of several compounds of the formula $Et_4N[Re_2(\mu\text{-}SR)_3(CO)_6]$, (R = Me, t-Bu, Ph, C_6F_5), and then a study of

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* Reference number with asterisk indicates a note in the list of references.

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several reactions of these species leading to compounds of the formulas $Re_2(\mu\text{-}SR)_2(CO)_6(L)_2$ and $Re_2(\mu\text{-}SR)_2(\mu\text{-}CO)(CO)_4(L)_2$ (L = PMe_3, PPh_3, 1/2 dppm).

Experimental

Infrared data were obtained using a Beckman 4230 spectrometer. ^1H NMR spectra were recorded on an IBM-WP-200 spectrometer. Melting points were obtained in open capillaries and are uncorrected. Elemental analyses were performed by Galbraith Laboratories, Knoxville, TN.

All solvents were dried before use, and all manipulations were done under N_2 as a precautionary measure. Preparations of $Re_2(\mu\text{-}Br)_2(CO)_8$ [5], $Et_4N[ReBr_2(CO)_4]$ [6], $Et_4N[Re_2(\mu\text{-}Br)_3(CO)_6]$ [2], $[Me_3O]BF_4$ [7] and $(n\text{-}Bu)_3SnSR$ (R = Me, Bu^t, Ph, C_6F_5) [8] were carried out according to literature directions.

The syntheses of several other (known) starting materials are described briefly below:

$Et_4N[Re_2(\mu\text{-}Br)_2(Br)(CO)_7]$. This compound was made using an adaption of the literature procedure [10] which achieved significantly higher yields of the product. A suspension of 0.55 g (1.35 mmol) $ReBr(CO)_5$ and 0.30 g (1.35 mmol) Et_4NBr in 60 ml of n-BuOH was heated to reflux. As the temperature rose, the suspension dissolved to give a pale-yellow solution. After refluxing for 12 h, the solution was cooled causing a white solid to precipitate. The solid was filtered, washed with hexane, and air dried. The addition of hexane to the filtrate caused precipitation of a second crop of product which was isolated as described above; yield 86%.

$Re_2(\mu\text{-}SR)_2(CO)_8$ (R = Ph, Me). A suspension of 0.38 g (0.50 mmol) of $Re_2(\mu\text{-}Br)_2(CO)_8$ in 30 ml of THF was treated with a slight excess of the appropriate tri-n-butyltinthiolate (0.5 ml, ~ 1.5 mmol). The suspension was stirred at room temperature; during 6 h, the white color turned to light yellow and the white $Re_2(\mu\text{-}Br)_2(CO)_8$ dissolved. Solvent reduction to 10 ml, addition of 10 mL Skelly B and cooling to −20 °C led to precipitation of a light yellow powder, the product, which was separated by filtration. The yield generally exceeded 90%. The methyl and phenyl complexes were characterized by comparison with m.p. and IR data in the literature [11].

Syntheses of $Et_4N(Re_2(\mu\text{-}SR)_3(CO)_6]$ complexes.

Samples of $Et_4N[Re_2(\mu\text{-}Br)_3(CO)_6]$ (0.23 g, 0.25 mmol) and $(n\text{-}Bu)_3SnSR$ (0.50 mmol) were dissolved in 25 ml of methanol. The solution was heated at reflux for 2 h and then allowed to cool to ambient temperature. Addition of hexane caused precipitation of a yellow-to-white solid product which was separated by filtration. The solid was dissolved in a minimum of CH_2Cl_2; after filtration, an equal volume of diethyl ether was added. Chilling the solution at −20 °C led to precipitation of the pure product, a crystalline solid. This was separated by filtration and dried in vacuo.

The following compounds were isolated by this procedure:

$Et_4N[Re_2(\mu\text{-}SPh)_3(CO)_6]$. 85%, m.p. 229 °C. Anal. Found: C, 38.60; H, 3.57. $C_{32}H_{35}NO_6S_3Re_2$ calcd.: C, 38.50; H, 3.63%. IR (CH_2Cl_2): ν(CO) 2004s, 1905s, br. ^1H NMR (acetone-d_6): 1.37t (int. 12); 3.45 q (int. 8); 7.18m, 7.45m (int. 15).

$Et_4N[Re_3(\mu\text{-}SBu^t)_3(CO)_6]$. 79%, m.p. 251 °C(d). Anal. Found: C, 33.23; H,

4.68. $C_{26}H_{47}NO_6S_3Re_2$ calcd.: C, 33.28; H, 5.05%. IR (CH_2Cl_2): ν(CO) 1988s, 1885s, br. ^{1}H NMR (acetone-d_6): 1.40t (int. 12), 1.42s (int. 27), 3.49 q (int. 8).

$Et_4N[Re_2(\mu\text{-}SMe)_3(CO)_6]$. 65%, m.p. 225 °C. Anal. Found: C, 25.57; H, 3.85. $C_{17}H_{29}NO_6S_3Re_2$ calcd.: C, 25.15; H, 3.60%. IR (CH_2Cl_2): ν(CO) 1994s, 1892s, br. ^{1}H NMR (CD_2Cl_2): 1.43t (int. 12), 2.16s (int. 9), 3.25 q (int. 8).

$Et_4N[Re_2(\mu\text{-}SC_6F_5)_3(CO)_6]$. 73%, m.p. 160 °C. Anal. Found: C, 30.80; H, 2.22. $C_{32}H_{20}NO_6F_{15}S_3Re_2$ calcd.: C, 30.26; H, 1.59%. IR (CH_2Cl_2): ν(CO) 2020s, 1921s, br. ^{1}H NMR (acetone-d_6): 1.35t (int. 12); 3.41 q (int. 8).

These complexes could also be prepared using $Et_4N[ReBr_2(CO)_4]$ as a starting material. Conditions of the reaction, and isolation and purification of these products, were identical to those described above. Yields of the products, $Et_4N[Re_2(\mu\text{-}SR)_3(CO)_6]$, were: R = Ph, 71%; R = Bu^t, 66%; R = Me, 43%; R = C_6F_5, 57%.

The compound $Et_4N[Re_2(\mu\text{-}SPh)_3(CO)_6]$ was the sole organorhenium compound obtained from a reaction between excess NaSPh and $(Et_4N)_2[ReBr_3(CO)_3]$ in methanol (ambient temperature, 3 h; 65% yield of product isolated as described above).

A reaction between $Et_4N[Re_2(\mu\text{-}Br)_2(Br)(CO)_7]$ (0.12 g, 0.13 mmol) and excess $(n\text{-}Bu)_3SnSPh$ (0.25 g, 0.62 mmol) in CH_2Cl_2 led to the same product (ambient temperature, 0.5 h; 68% yield using similar isolation procedures). In reactions using a 1/1 ratio of these reagents a mixture of four possible compounds $Et_4N[Re_2(\mu\text{-}SPh)_n(\mu\text{-}Br)_{3-n}(CO)_6]$ was determined to be present according to IR data. We did not choose to separate these species. Similar results were obtained using NaSPh as the thiolate containing reagent.

Reactions of $Et_4N[Re_2(\mu\text{-}SPh)_3(CO)_6]$ with $[Me_3O]BF_4$ and phosphines (PMe_3, dppm)

Preparation of cis-$Re_2(\mu\text{-}SPh)_2(CO)_6(Phos)_2$. A sample of $Et_4N[Re_2(\mu\text{-}SPh)_3(CO)_6]$ (0.25 g, 0.25 mmol) was dissolved in 50 ml CH_2Cl_2. Solid $[Me_3O]BF_4$ (0.040 g, 0.27 mmol) was then added to the flask. After the mixture was stirred for 20 min (a slight darkening being observed), an approx. 0.5 mmol sample of the phosphine was added. Stirring was continued for an additional 30 min; the solvent was then removed in vacuo. The solid residue was extracted with several portions of diethyl ether in which Et_4NBF_4 was insoluble. The diethyl ether solution was filtered; cooling at −20 °C led to formation of light yellow crystals of the product, *cis*-$Re_2(\mu\text{-}SPh)_2(CO)_6(Phos)_2$.

The following were characterized:

cis-$Re_2(\mu\text{-}SPh)_2(CO)_6(PMe_3)_2$, yield 38%, m.p. 173 °C (dec.). Anal. Found: C, 31.69; H, 3.06. $C_{24}H_{28}O_6P_2S_2Re_2$ calcd.: C, 31.58; H, 3.09%. IR ($CHCl_3$): ν(CO) 2032s. 2014s, 1935sh, 1918s, 1987s. ^{1}H NMR (acetone-d_6): 1.82d, J_{PH} 8.0 Hz (int. 9); 7.30 m, 7.62m (int. 5).

$Re_2(\mu\text{-}SPh)_2(CO)_6(dppm)$. Yield 43%, m.p. 187 °C (dec.). Anal. Found: C, 45.34; H, 2.76. $C_{43}H_{32}O_6P_2S_2Re_2$ calcd.: C, 45.10; H, 2.82%. IR ($CHCl_3$): ν(CO) 2040s, 2025s, 1950s, 1925sh, 1909s. ^{1}H NMR (acetone-d_6): 4.63t, J(PH) 10.8 Hz (int. 1); 7.30m, 7.67m (int. 15).

Two other procedures have been used to synthesize the $Re(\mu\text{-}SR)_2(CO)_6(Phos)_2$ species. Both were originally described in the literature by Abel and coworkers [12]. The first involves reactions between $ReBr(CO)_3(PR_3)_2$ and organotin thiolates, run in THF at the reflux temperature. The second procedure, involving reactions between Re$_2$(μ-SR)$_2$(CO)$_8$ and phosphines, has been further studied by Vahrenkamp and Kullmer [13]. We have also used both procedures to make the compounds described above and several others. The second method is briefly described below.

Reaction of the Re$_2$(μ-SR)$_2$(CO)$_8$ and phosphines

A benzene solution of these reactants, in a 1/2 molar ratio, was heated at reflux for 3 h. After being allowed to cool, the solution was evaporated to dryness in vacuo and the solid residue was purified by crystallization from CH$_2$Cl$_2$/hexane. Good yields of the following compounds were obtained:

cis-Re$_2$(μ-SMe)$_2$(CO)$_6$(PPh$_3$)$_2$. Yield, 62%; m.p. 170 °C (dec.). Anal. Found: C,45.78; H, 3.18. $C_{44}H_{36}O_6P_2S_2Re_2$ calcd.: C, 45.52; H, 3.13%. IR (CH$_2$Cl$_2$): ν(CO) 2024s, 2007s, 1940m, 1913s, 1904s cm^{-1}. ^{1}H NMR (CDCl$_3$): 2.21s (int. 1); 7.30m, 7.65 (int. 5).

cis-Re$_2$(μ-SMe)$_2$(CO)$_6$(PPh$_3$)$_2$. Yield, 60%; m.p. 165 °C (dec.). Anal. Found: C, 31.69; H, 3.06. $C_{54}H_{30}O_6P_2S_2Re_2$ calcd.: C, 31.58; H, 3.09%. IR (CH$_2$Cl$_2$): ν(CO) 2036s, 2018s, 1952m, 1923s, br cm^{-1}. ^{1}H NMR (acetone-d_6): 7.38 (int. 2); 7.65m (int. 3).

Also using the procedure described above, *cis*-Re$_2$(μ-SPh)$_2$(CO)$_6$(PMe$_3$)$_2$ and *cis*-Re$_2$(μ-SPh)$_2$(CO)$_6$(dppm) were obtained in 83 and 76% yields, respectively.

Among the products in the synthesis of *cis*-Re(μ-SPh)$_2$(CO)$_6$(PPh$_3$)$_2$, a small amount of the *trans*-isomer was detected by IR. We verified in an independent experiment that heating the *cis* isomer in benzene (at reflux) causes its conversion to the *trans*-isomer. In a typical experiment a 0.25 g (0.27 mmol) sample of the *cis*-isomer was heated in benzene at reflux for 30 h; *trans*-Re$_2$(μ-SPh)$_2$(CO)$_6$(PPh$_3$)$_2$ was recovered upon evaporation of the solvent (0.25 g) as the sole product.

Anal. Found: C, 31.77; H, 3.07. $C_{54}H_{34}O_6P_2S_2Re_2$ calcd., C, 31.58; H, 3.09%. IR (CHCl$_3$): ν(CO) 2012s, 1931s, 1906s cm^{-1}. ^{1}H NMR (CD$_2$Cl$_2$): 1.43d, J_{PH} 9.0 Hz (int. 9); 7.10m, 7.52m (int. 5), m.p. 197 °C.

In contrast, a different reaction occurs if *cis*-Re$_2$(μ-SPh)$_2$(CO)$_6$(PPh$_3$)$_2$ is heated in tetrahydrofuran. During 48 h at reflux, an orange solution was formed. After the solution was allowed to cool, the solvent was removed, in vacuo, and the solid residue crystallized from CH$_2$Cl$_2$ giving Re$_2$(μ-SPh)$_2$(μ-CO)(CO)$_4$(PPh$_3$)$_2$, 30% yield; m.p. 243 °C.

Anal. Found: C, 51.28; H, 3.08. $C_{53}H_{40}O_5P_2S_2Re_2$ calcd.: C, 50.71; H, 3.21%. IR (CHCl$_3$): ν(CO) 1998s, 1960s, 1927s, 1810s, br cm^{-1}. ^{1}H NMR (CDCl$_3$): 7.28m (int. 3); 7.80m (int. 2).

This type of complex is known for manganese [14] but has not been seen before in rhenium chemistry.

Alternative synthesis of Re$_2$(μ-SPh)$_2$(μ-CO)(CO)$_4$(PPh$_3$)$_2$

A solution of 0.58 g (0.66 mmol) ReBr(CO)$_3$(PPh$_3$)$_2$ and 0.3 mL (0.84 mmol) (n-Bu)$_3$SnSPh in 40 mL of THF was refluxed for 24 h. After cooling, the resulting orange solution was reduced in volume to 5 ml and cooled (−20 °C). The orange crystals that formed were collected by filtration and washed three times with diethyl ether. This sample of Re$_2$(μ-SPh)$_2$(μ-CO)(CO)$_4$(PPh$_3$)$_2$ was recrystallized from CH$_2$Cl$_2$/hexane (35% yield).

The combined filtrate and washings were reduced in volume to a yellow oil. The yellow oil was dissolved in a minimum of diethyl ether. Hexane was layered on, and the solution was cooled giving yellow crystals which were collected by filtration and washed with hexane. Recrystallization from $CHCl_3$/hexane afforded analytically pure *cis*-$Re_2(\mu\text{-}SPh)_2(CO)_6(PPh_3)_2$ in 52% yield.

If the solution had been allowed to reflux in THF for a longer period of time, one may presumably expect complete conversion to the former product to occur.

Discussion

The rhenium complexes $Et_4N[Re_2(\mu\text{-}SR)_3(CO)_6]$ are obtained in high yield; with no evidence of other organorhenium products or decomposition the yields are apparently limited only by workup and purification techniques. As in the synthesis of the analogous manganese complexes, a variety of starting materials may be employed. In this study we chose either the thiolate anion (as the sodium salt, NaSR) or a tributyltin thiolate compounds, $(n\text{-}Bu)_3SnSR$, to provide the thiolate ligand; rhenium precursors included the halocarbonyl species $Et_4N[Re_2(\mu\text{-}Br)_3(CO)_6]$, $Et_4N[ReBr_2(CO)_4]$, or $(Et_4N)_2[ReBr_3(CO)_3]$, and $Et_4N[Re_2(\mu\text{-}Br)_2(Br)(CO)_7]$. The equation below is representative of reactions described:

$$Et_4N[Re_2(\mu\text{-}Br)_3(CO)_6] + 3(n\text{-}Bu)_3SnSR \rightarrow$$

$$Et_4N[Re_2(\mu\text{-}SR)_3(CO)_6] + 3(n\text{-}Bu)_3SnBr$$

(R = Me, Ph, t-Bu, C_6F_5)

The products are white or off-white, crystalline species, soluble in polar solvents and insoluble in hydrocarbons. They are not sensitive to air or atmosphere moisture for at least moderate periods of time. Two strong carbonyl stretches are observed in infrared spectra of these compounds. The frequencies of these IR absorptions range from 2020(s) and 1921(s, br) cm^{-1} for the SC_6F_5 derivative to 1994(s) and 1885(s, br) cm^{-1} for the t-BuS derivative; the variation in frequencies is predictable based on the electron donor-acceptor character of the organic substituent groups. Diamagnetism is indicated by the proton NMR spectra containing the resonances expected for the thiolate and tetraethylammonium units. As with structurally similar compounds, only a single proton resonance is seen for the thiomethyl groups in $Et_4N[Re_2(\mu\text{-}SMe)_3(CO)_6]$. From this evidence, it appears that only one conformer is present in which all the thiolate groups are equivalent.

Addition of trimethyloxonium tetrafluoroborate to a solution of any of these compounds, followed by addition of a phosphine, leads to formation of the compounds, *cis*-$Re_2(\mu\text{-}SR)_2(CO)_6(L)_2$ (I) as shown in the equation below. Such reactions are straightforward, easy to carry out, and result in good yields of products.

$$Et_4N[Re(\mu\text{-}SR)_3(CO)_6] + [Me_3O]BF_4 + 2L \rightarrow$$

$$cis\text{-}Re_2(\mu\text{-}SR)_2(CO)_6(L)_2 + Me_2O + MeSR + [Et_4N]BF_4$$

(R = Ph, L = PMe_3, PPh_3, 1/2 dppm; and R = Me; L = PPh_3)

In this reaction, the $[Me_3O]BF_4$ presumably acts as an alkylating agent, transfering a methyl group to a lone pair on one of the bridging thiolate groups and converting it to a bridging sulfide (MeSR) group. Sulfides (RSMe) are not expected to bond strongly, particularly as bridging ligands; likely they would be readily lost, replaced by better ligands such as phosphines if present in the solution. This type of reaction was also observed to occur with the manganese analogues [14]; in those examples, the incoming ligands have a *cis* configuration. Similar species have been characterized as products in two other reactions. Previously, it had been reported that *cis*-$Re_2(\mu\text{-}SR)_2(CO)_6(L)_2$ species are the products in reactions between $ReCl(CO)_3(PPh_3)_2$ and Me_3SnSR, and in reactions between $Re_2(\mu\text{-}SR)_2(CO)_8$ and phosphines [12,13].

In all, four new compounds of the indicated formula were obtained by these various reactions. Stable, white crystalline compounds, they have a characteristic four or five band infrared pattern and ^1H NMR spectra appropriate for the indicated stoichiometry; absorptions at 2036s, 1952m, and 1923s,br cm^{-1} are measured for *cis*-$Re_2(\mu\text{-}SPh)_2(CO)_6(PPh_3)_2$, for example.

In the course of this work we repeated the literature procedures for synthesis of several of these complexes. In the reaction between $Re_2(\mu\text{-}SPh)_2(CO)_8$ and PPh_3 (benzene, at reflux, 3 h) we determined that a second isomer of the expected product was also present. Upon further study, it was determined that the indicated *cis* isomer is, in fact, the initial product in this reaction but that this product was being converted thermally to *trans*-$Re_2(\mu\text{-}SR)_2(CO)_6(PPh_3)_2$ (II). Complete conversion from the *cis* to the *trans* isomer can be accomplished by heating a solution of the former in benzene (at reflux) for 30 hrs.

A similar conversion of a *cis* isomer to *trans* isomer had earlier been demonstrated for the species $Re_2(\mu\text{-}SH)_2(CO)_6(PPh_3)_2$ [13]. The *trans* isomers are easily identified in these reactions by a somewhat simpler three band carbonyl stretching pattern in their infrared spectra; the spectrum for $Re_2(\mu\text{-}SPh)_2(CO)_6(PPh_3)_2$, for example, contains absorptions at 2012s, 1931s, and 1906s cm^{-1}.

A different result is encountered when a tetrahydrofuran solution of *cis*-$Re_2(\mu\text{-}SPh)_2(CO)_6(PPh_3)_2$ is heated. Here, the compound $Re_2(\mu\text{-}SPh)_2(\mu\text{-}CO)(CO)_4(PPh_3)_2$ (III) is obtained as the sole product. Identification of this orange species can be made from the weak bridging carbonyl absorption at 1810w cm^{-1} in an infrared spectrum; additional carbonyl absorptions are seen at 1998s, 1960s, 1927s cm^{-1}. Analogous manganese compounds are known to be formed in similar reactions [14]. Indeed, such reactions in the manganese case are quite facile; for example, it is not actually possible to isolate $Mn_2(\mu\text{-}SPh)_2(CO)_6(PPh_3)_2$, the analogue to the rhenium compound described in this paper, because decarbonylation occurs so readily. Crowding of the phosphines in the precursor with *cis* stereochemistry appears to be a driving force for this reaction. Steric interactions in the rhenium system must be much less serious, since rather more severe conditions are required for this conversion.

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