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CS$_2$ Reductive Coupling to Acetylenedithiolate by a Dinuclear Ytterbium(II) Complex

Davide Toniolo,[a] Aurélien R. Willauer,[a] Julie Andrez,[a] Yan Yang,[b] Rosario Scopelliti, [a] Laurent Maron,*[b] and Marinella Mazzanti*[a]

Abstract: *The activation of CS$_2$ is of interest in a broad range of fields and more particularly in the context of creating new C-C bonds. The reaction of the dinuclear ytterbium(II) complex [Yb$_2$L$_4$], 1, (L = (O^{t}Bu)$_3$SiO$^-$) with carbon disulphide led to the isolation of unprecedented reduction products. In particular the crystallographic characterization of complex [Yb$_2$L$_4$(μ-C$_2$S$_2$)], 2 provides the first example of an acetylenedithiolate ligand formed from metal reduction of CS$_2$. Computational studies indicate that this unprecedented reactivity can be ascribed to the unusual binding mode of CS$_2^{2-}$ in the isolated "key intermediate" [Yb$_2$L$_4$(μ-CS$_2$)], 3 which is resulting from the dinuclear nature of 1.*

The activation of CS$_2$ by transition metals is of interest for the development of catalysts that can convert the CS$_2$ pollutant, largely used as feedstock in viscose industrial production, into less toxic or useful products.[1] Numerous studies have also been driven by its similarity to CO$_2$, a potential abundant and renewable source of carbon and the possibility of creating new C-C bonds.[2] Metal complexes of the d[2b, 2c, 3] and 5f block[4] [2d, 5], were shown to promote a wide range of reactions such as CS$_2$ reduction to CS$_2^{2-}$, disproportionation to CS$_3^{2-}$ and CS, head-to-head reductive C-C coupling to afford tetrathioxalate, head-to-tail reductive C-S coupling, or cleavage of one C-S bond leading to CS and sulphide complexes. Complete disassembly of the CS$_2$ molecule has also been reported in three cases.[1b] [6]

Divalent lanthanide ions display a broad range of reactivity with small unreactive molecules,[7] but only few examples of CS$_2$ activation have been reported. CS$_2$ activation by lanthanide ions so far resulted in CS$_2$ disproportionation or reductive C-S or C-C coupling to afford thiocarbonate, thiooxalate and thioformyl carbonotrithioate ((SCSCS$_2$)$^{2-}$) respectively.[8] Very recently the first example of a rare CS$_2^{2-}$ dianion trapped between two Yb(III) ions was also reported.[9]
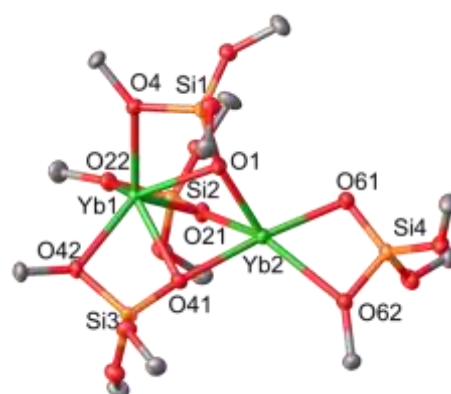
CO reductive coupling by samarium(II) and uranium(III) complexes was reported to afford ketene carboxylate and ynediolate O-C≡C-O^{2-} (that act as a linear κ^2-O,O' bridging ligand) respectively.[10] In contrast the formation of the acetylenedithiolate dianion analogue (S-C≡C-S)$^{2-}$ from CS$_2$ or CS reduction was never reported. Acetylenedithiolate is of interest as bridging ligand because of its potential to support electronic communication between metal centres, but, due to its low stability, only a few acetylenedithiolate complexes were so far reported.[11]

All the reported examples of CS$_2$ reduction by lanthanides involve two one-electron transfers by two metal complexes, but experimental and computational studies suggest that cooperative binding of the substrate by bimetallic intermediate is crucial in the outcome of the reduction.[8c, 12] As such, the reaction of polynuclear lanthanide complexes with CS$_2$ is of interest because it may allow to trap reactive intermediates or stabilize unusual reaction products.

Here we report the reduction of CS$_2$ by the dinuclear Yb(II) complex [Yb$_2$L$_4$], 1, (L = (O^{t}Bu)$_3$SiO$^-$)[13] which allowed to trap for the first time a product of CS coupling from CS$_2$ reduction in the complex [Yb$_2$L$_4$(C$_2$S$_2$)], 2 together with the CS$_2^{2-}$ bridged key intermediate [Yb$_2$L$_4$(CS$_2$)], 3.

Crystals of the homoleptic complex 1, were obtained by recrystallization from *n*.hexane at -40 °C of the [Yb$_2$L$_4$(DME)] complex prepared as previously described for [Sm$_2$L$_4$(DME)].[14] The solid-state structure of 1 is presented in Figure 1 and shows the presence of a dimer where two Yb(II) ions are bridged by three oxygen atoms from three different siloxide ligands in a non-symmetric fashion. The Yb-O$_{siloxide}$ bond lengths in 1 range from 2.265(2) – 2.570(3) Å, and are consistent with the values previously reported for the divalent ytterbium complex [YbL$_4$K$_2$] (2.251(6) – 2.571(6) Å).[1b]

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Figure 1. Solid-state molecular structures of 1 (50% probability ellipsoids). Hydrogen atoms, methyl groups and solvent molecule are omitted for clarity.

The addition of 1 - 2 equivalents of ^{13}CS$_2$ to a C$_6$D$_{12}$ solution of 1 at room temperature, resulted reproducibly in the immediate complete conversion of 1 to new species as

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indicated by ^{1}H NMR spectroscopy studies (Scheme 1). The ^{1}H NMR spectrum of the reaction mixture shows the presence of the same species in the same ratios independently of the conditions used.
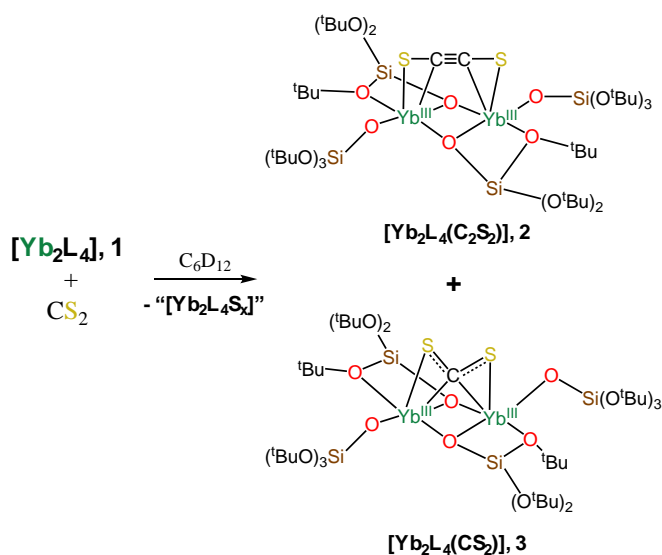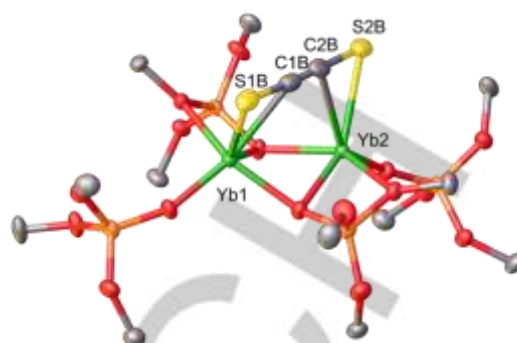

Scheme 1. Reaction of **1** with carbon disulphide in cyclohexane.

Storage of a concentrate solution of the reaction mixtures at room temperature resulted in the isolation of the complex [Yb$_2$L$_4$(C$_3$S$_5$)] as a minor product (5 % yield) (See supporting information). Storage of a concentrated *n*-hexane solution of the reaction mixture at -40 °C, after removal of [Yb$_2$L$_4$(C$_3$S$_5$)]**,** allowed the isolation of pale orange crystals of the co-crystallized species [Yb$_2$L$_4$(μ-C$_2$S$_2$)]**, 2** and [Yb$_2$L$_4$(μ-CS$_2$)]**, 3**, with **2/3** ratios of 0.24/0.76, 0.18/0.82 or 0.10/0.90 obtained from X-ray diffraction measurements on different crystals.

In one occasion, crystals containing only complex **3** were also isolated.

The structure of **2** (Figure 2) shows the presence of a dimer where the C$_2$S$_2$$^{2-}$ ligand bridges the two Yb(III) ions in an unprecedented linear μ–η^2-C,S: η^2-C',S' fashion. The observed binding mode is consistent with the formulation of the ligand acetylenediolate and differs significantly from the side-on bridging mode (μ–η^2-C,C') found in homo and heterobimetallic complexes of d-block metals.[11a-f]

The Yb-Yb distance (3.2460(2) Å) is slightly longer than in **1** (3.2303(3) Å). The Yb–O$_{siloxide}$ distances (2.068(2) – 2.337(2) Å) are consistent with those found in the homoleptic trivalent complex [YbL$_3$] (2.029 (3) – 2.339 (2) Å).[15] The value of the C–C distance in the C$_2$S$_2$$^{2-}$ moiety (1.16(2) Å) is characteristic of a triple bond (1.2 Å in acetylene) and is slightly shorter than what found in other Ln(III)-bound acetylides (1.202(4) Å).[16] The two C–S bonds (1.71(2) and 1.72(2) Å) are longer than the CS double bond in CS$_2$ (1.55 Å) and in the range of values previously reported for the C-S bond in ethylenedithionate complexes.[11a, 17] The mean value of the Yb(III)-C bond distance at 2.62(2) Å is shorter than the value found in the Yb(II) η^2-acetylene complex [(Me$_5$C$_5$)$_2$Yb(η^2-MeC≡CMe) (2.850(1) Å)[18] but consistent with the difference in ionic radii (0.16 Å).The C$_2$S$_2$$^{2-}$ moiety is nearly linear with C2B–C1B–S1B and C1B–C2B–S2B angles of 176(2)° and 177(2)°.

Figure 2. Solid-state molecular structure of **2** (50% probability ellipsoids). Hydrogen atoms, methyl groups and co-crystallized **3** were omitted for clarity. Selected bond lengths (Å): C1B–C2B = 1.16(4); C1B–S1B = 1.71(2); C2B–S2B = 1.72(2); Yb1-S1B = 2.566(6); Yb2-S2B = 2.664(6).

The complex **3** crystallizes in the monoclinic space group P2$_1$/c as a dimer with six-coordinated metal centre. The structure presented in Figure 3 shows that the reduced CS$_2$$^{2-}$ moiety bridges the two trivalent ytterbium ions in a symmetric μ–η^2(CS): η^2(CS') fashion. The two C–S bond distances have similar values (1.673(18) and 1.68(2) Å) and are longer than free CS$_2$ (1.55 Å), consistent with the two-electron reduction of carbon disulphide. The symmetrical binding mode adopted by the CS$_2$$^{2-}$ in complex **3** differs significantly from those found in the only other reported lanthanide complex of CS$_2$$^{2-}$ [Yb(DippForm)$_4$(CS$_2$)][9] (μ-η^3(S,C,S'):η^2(S,C)) or in the U(IV) complexes, [U{(OSi(O^{t}Bu)$_3$)$_3$}$_2$(μ-CS$_2$)],[4b] (μ-η^2(S,C):η^2(S,S'))) and [(CH$_3$C$_5$H$_4$)$_3$U]$_2$(μ-CS$_2$)],[4a] ((μ-η^2(S,C):η^1(S')) isolated from the reduction of CS$_2$ by two mononuclear Yb(II) and U(III) complexes respectively.

Only two examples of dinuclear complexes presenting a similar symmetrical coordination mode with two side-on bound C-S fragments were reported so far for d-block metals.[2c, 3c]
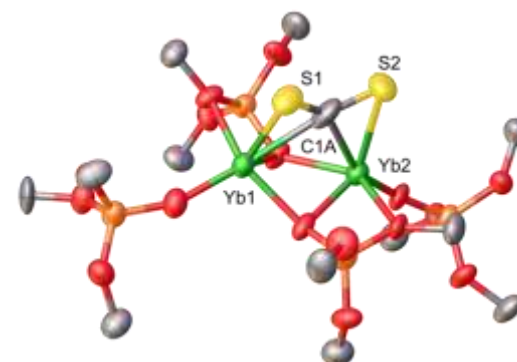
Figure 3. Solid-state molecular structure of **3** (50% probability ellipsoids). Hydrogen atoms and methyl groups were omitted for clarity. Selected bond lengths (Å): C1A–S1 = 1.673(18); C1A–S2 = 1.68(2), Yb1–S1 = 2.656(4); Yb2–S2 = 2.645(5); Yb1–C1A = 2.52(2); Yb2–C1A = 2.531(17).

The complexes **2** and **3** co-crystallize and could not be isolated separately for analytical purposes, but they can be isolated together in good yield from the other reaction

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products by crystallization. The ^1H NMR spectrum of the isolated mixtures of **2** and **3** shows only the presence of three NMR signals for the siloxide ligands of the two complexes.

The ^{13}C NMR signals of the bound $C_2S_2^{2-}$ and CS_2^{2-} dianions could not be observed in the isolated mixtures of **2** and **3** probably due to the paramagnetism of the Yb(III) cation. However, after addition of two equivalents of pyHCl, the quantitative ^{13}C NMR in DMSO-d_6 shows the presence of two signals at δ = 257.2 ppm and 246.3 ppm assigned to the $C_2S_2^{2-}$ and CS_2^{2-} dianions in **2** and **3** respectively (see supporting information).

The reactivity of **1** with CS_2 differs significantly from that reported for the mononuclear ytterbium complex [YbL$_4$K$_2$], that resulted in the formation of $C_2S_4^{2-}$, $C_3S_5^{2-}$, CS_3^{2-}.[8a] Due to the sterically encumbered environment of the Yb(II) centre in [YbL$_4$K$_2$], the CS_2 reduction products were quickly released and metal bound intermediates could not be isolated. In spite of the lower number of siloxide supporting ligands and anticipated lower reducing power, the dinuclear complex [Yb$_2$L$_4$] promotes the reduction of CS_2 and allowed to trap the reduction products. The high stability of the CS_2^{2-} intermediate **3** and the formation of the acetylenediolate reduction product may be ascribed to the unusual binding mode imposed in the CS_2^{2-} intermediate by the dinuclear nature of the [Yb$_2$L$_4$] complex and the cooperative reduction by the two metal centres. The formation of $C_2S_2^{2-}$ is accompanied by the formation of a sulphide or polysulphide complex that could be identified by ^1H NMR studies (by performing the reaction of **1** with elemental sulphur), but that could not be isolated. Previous studies showed that the reaction of Ln(II) with sulphur can yield dinuclear sulphide, disulphide, trisulphide and polysulphide complexes or fascinating sulphide bridged cluster compounds.[9, 19]

The reduction of CS_2 to acetylenediolate is unprecedented both in f- and d-block metal chemistry. Therefore, DFT calculations (B3PW91) were carried out in order to get some insights on this peculiar reactivity (Figure 4).
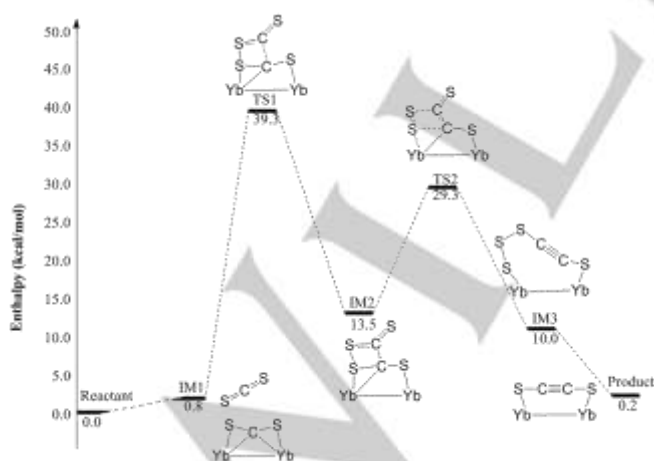
Figure 4. Computed enthalpy profile for the reaction of **1** with CS_2 at room temperature.

The reaction begins with the double reduction of CS_2 by complex **1**, yielding the dinuclear "key intermediate" (complex **3**). This step is found to be exothermic by 22.4 kcal/mol as found in other reactions. From complex **3**, the

direct CS bond breaking to yield thiocarbonate is computed to be kinetically difficult (barrier of 46.6 kcal/mol, see figure S15). Complex **3** can then further react with another CS_2 molecule but in a very different way from what found for mononuclear U(III),[4c] or Sm(II)[8c] complexes. Indeed, rather than a nucleophilic addition, the system is undergoing a [2+2] cycloaddition. This peculiar reactivity is associated with the CS_2 binding mode in complex **3**. Indeed, unlike previously reported $(CS_2)^{2-}$ complexes, the HOMO indicates a bonding interaction between the carbon of CS_2 and the two Yb centres (Figure 4), reversing the bond polarity in the CS_2 molecule by relocalizing negative charge on the sulphur atoms (-0.13). Therefore, the best overlap of the incoming CS_2 molecule and complex **3** involves sulphur-sulphur and carbon-carbon interactions, as found in TS1 (cycloaddition TS). The cycloaddition product formation (IM2) appears to be endothermic by 13.7 kcal/mol so that the system evolves through the release of S_2 (TS2), yielding the final product whose formation is athermic from complex **3**. The trapping of S_2 by a Yb(II) dimer (leading to the experimentally observed sulfide complex) is necessary for the release to occur. Notably, direct addition of excess CS_2 to complex **3** in the absence of the precursor does not lead to further reactivity.
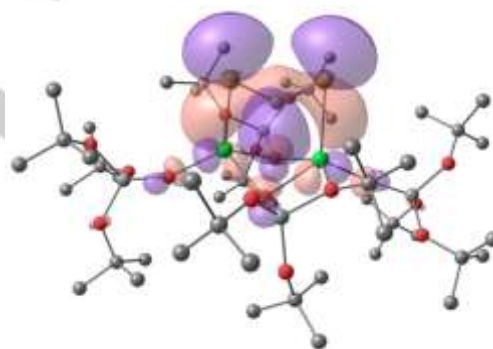
Figure 5. 3D representation of the HOMO of complex **3**

In conclusion, CS_2 reduction by a dinuclear Yb(II) complex supported by siloxide ligands, proceeds very differently from what previously found for other mononuclear Ln(II) or U(III) complexes highlighting the important effect of multimetallic cooperativity in the outcome of CS_2 activation. Notably, we isolated the first example of acetylenedithiolate complex formed from the metal reduction of CS_2. DFT studies suggest that this unusual reactivity arises from the rare symmetric double side-on binding mode found in the stable dinuclear Yb(III)-CS_2^{2-} intermediate.

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It takes two to couple:

The unusual symmetric binding of CS₂ by two Yb(II) centers in a dinuclear complex supported by siloxide ligands leads to the first example of reduction of CS₂ to acetylenedithiolate (C₂S₂²⁻) in a metal complex.
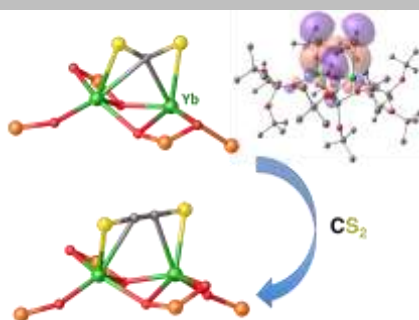
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