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The Reactivity of $(C_5Me_5)_2Sm(THF)_2$ with Bis(2-pyridyl)ethene Including the Synthesis of $[(C_5Me_5)_2Sm]_2(\mu\text{-}\eta^2{:}\eta^2\text{-pyCHCHpy})$ from $[(C_5Me_5)_2Sm]_2[\mu\text{-}\eta^3{:}\eta^3\text{-}1,2,3,4\text{-}(py)_4C_4H_4]$ by Reductive C–C Bond Cleavage

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Summary: $(C_5Me_5)_2Sm(THF)_2$, *1, reacts with 1,2-bis(2-pyridyl)ethene to form* $[(C_5Me_5)_2Sm]_2(\mu\text{-}\eta^2{:}\eta^2\text{-pyCHCHpy})$, *2, in 80% yield if the reaction stoichiometry is 2:1. Combination of the same reagents in a 1:1 ratio forms* $[(C_5Me_5)_2Sm]_2[\mu\text{-}\eta^3{:}\eta^3\text{-}1,2,3,4\text{-}(py)_4C_4H_4]$, *3, in 78% yield. Both 2 and 3 have been characterized by X-ray crystallography. 2 and 3 can be interconverted by reacting pyCH=CHpy with 2 and by reacting* $(C_5Me_5)_2Sm(THF)_2$ *with 3. 2 crystallizes from hexane with one hexane per bimetallic unit in the orthorhombic space group Pbcn with* $a = 14.693(5)$ Å, $b = 15.415(3)$ Å, $c = 50.173(7)$ Å, $V = 11364(5)$ Å^3, *and* $Z = 8$ *for* $D_{calcd} = 1.295$ g cm^{-3}. *3 crystallizes from hexane/toluene with one hexane per bimetallic unit in the monoclinic space group C2/c with* $a = 17.397(4)$ Å, $b = 29.338(9)$ Å, $c = 15.063(3)$ Å, $\beta = 123.810(13)°$, $V = 6388(2)$ Å^3, *and* $Z = 4$ *for* $D_{calcd} = 1.34$ g cm^{-3}.

$(C_5Me_5)_2Sm(THF)_2$ reacts with a variety of substrates containing multiple bonds, including alkenes,[1–3] alkynes,[4,5] phosphaalkynes,[6] imines,[7] nitriles,[8] isonitriles,[9] azo compounds,[10,11] carbon monoxide,[12] and even dinitrogen,[13] to form products containing Sm^{3+} ions and the reduced substrate. The products of these reactions typically fall into two categories: (1) $[(C_5Me_5)_2Sm]_2$(substrate) complexes are isolated which contain the doubly-reduced substrate or (2) reductive coupling occurs to form $[(C_5Me_5)_2Sm]_2$(substrate–substrate) species which contain a dianionic, coupled-substrate moiety. We report here that $(C_5Me_5)_2Sm(THF)_2$ reacts with bis(2-pyridyl)ethene to form *both* types of complexes and that $(C_5Me_5)_2Sm(THF)_2$ can cleave the C—C bond in the coupled product. Although recent studies of the reactivity of $(C_5Me_5)_2Sm(THF)_2$ and $(C_5Me_5)_2Sm$ have shown that these divalent organosamarium complexes can cause a variety of bond cleavage reactions to occur, including C≡O,[12] N=N,[11] C=C,[1] C—N,[8,9] N—N,[14] Bi—C,[15] Sb—C,[16] Al—C,[17] C—H,[18–20] and C—X[21,22] bond scission, this is the first example of a direct C—C single bond cleavage reaction in organosamarium(II) chemistry.

Experimental Section

The compounds described below were handled under nitrogen with the rigorous exclusion of air and water using Schlenk, high-vacuum, and glovebox (Vacuum Atmospheres HE-553 Dri-Lab) techniques. Solvents were dried and physical measurements were obtained as previously described.[21] 1,2-Bis(2-pyridyl)ethene (Aldrich) was degassed before use. $(C_5Me_5)_2Sm(THF)_2$, **1**, was prepared as previously described.[23]

$\mathbf{[(C_5Me_5)_2Sm]_2(\mu\text{-}\eta^2{:}\eta^2\text{-pyCHCHpy}), 2.}$ A solution of 1,2-bis(2-pyridyl)ethene (53 mg, 0.29 mmol) in 5 mL of toluene was added dropwise to a stirred toluene solution of $(C_5Me_5)_2Sm(THF)_2$ (330 mg, 0.58 mmol) in 10 mL of toluene. The solution quickly turned red and was stirred for 1 h. The toluene and THF were removed by rotary evaporation to give a red solid which was dissolved in hexane. The solution was filtered, and the hexane was removed by rotary evaporation to yield **2** (240 mg, 80%). Crystals of **2** were obtained by slow evaporation of a hexane solution of **2** at room temperature. Anal. Calcd for $C_{26}H_{35}NSm$: C, 61.00; H, 6.89; N, 2.74; Sm, 29.38. Found: C, 60.77; H, 6.71; N, 2.64; Sm, 29.65. ^{1}H NMR (C_6D_6): δ 2.48 (m, 2H), 1.79 (s, C_5Me_5, 60H), −6.88 (br $\Delta\nu_{1/2} = 60$ Hz, 2H), −9.62 (d, $J = 8$ Hz, 2H), −11.79 (m, 2H). ^{13}C NMR (C_6D_6): δ 183.9 (s), 143.8 (d, $J_{CH} = 170$ Hz), 132.6 (d, $J_{CH} = 159$ Hz), 119.3 (s, C_5Me_5), 111.6 (d, $J_{CH} = 101$ Hz), 105.2 (d, $J_{CH} = 152$ Hz), 99.1 (d, $J_{CH} =$

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164 Hz), 19.3 (q, J_{CH} = 125 Hz, C_5Me_5). IR (KBr): 2900 (s), 2860 (s), 1605 (m), 1470 (m), 1435 (s), 1390 (m), 1325 (w), 1300 (m), 1270 (m), 1190 (w), 1160 (m), 1100 (w), 1020 (w), 855 (m), 780 (s), 760 (w), 730 (m), 690 (w) cm^{-1}.

A crystal of **2** measuring approximately 0.20 × 0.35 × 0.65 mm was immersed in Paratone oil, manipulated onto a glass fiber, and placed in the cold stream of a Siemens P3 diffractometer equipped with a modified LT-2 low temperature system. Although the crystallographic analysis identified the overall structure of the molecule, accurate bond distances and angles could not be obtained. Details are given in the supplementary material.[24]

$[(C_5Me_5)_2Sm]_2[\mu\text{-}\eta^3\text{:}\eta^3\text{-}1,2,3,4\text{-}(py)_4C_4H_4]$, 3. A solution of $(C_5Me_5)_2Sm(THF)_2$ (235 mg, 0.42 mmol) in 10 mL of toluene was added dropwise to a solution of 1,2-bis(2-pyridyl)ethene (76 mg, 0.42 mmol) in 10 mL of toluene. The solution turned dark red immediately and was stirred for 1 h. The solvent was then removed by rotary evaporation, and crude **3** was isolated as a red powder. Recrystallization from toluene gave **3** as red crystals (100 mg, 0.081 mmol, 78% based on 1,2-bis(2-pyridyl)ethene). Anal. Calcd for $C_{32}H_{40}N_2Sm$: C, 63.73; H, 6.69; N, 4.65; Sm, 24.94. Found: C, 63.55; H, 6.53; N, 4.58; Sm, 24.65. ^1H NMR (C_6D_6): δ 8.05 (d, J = 8 Hz, 2H), 7.01 (m, 2H), 6.32 (m, 2H), 6.26 (m, 2H), 5.70 (m, 2H), 5.60 (m, 2H), 5.34 (m, 2H), 4.52 (d, J = 4 Hz, 2H), 2.72 (br, $\Delta\nu_{1/2}$ = 40 Hz, 2H), 1.43 (s, C_5Me_5, 30H), 1.07 (s, C_5Me_5, 30H). Small peaks at δ 1.04 and 1.51 ppm may be due to an isomer. ^1H NMR (C_4D_8O): δ 7.95 (d, J = 6 Hz, 2H), 7.16 (s, 1H), 7.09 (dd, 2H), 6.29 (s, 2H), 5.67 (dd, 2H), 4.64 (d, J = 3 Hz, 2H), 1.29 (s, 15H, C_5Me_5), 0.94 (s, 15H, C_5Me_5). ^{13}C NMR (C_4D_8O): δ 170.9 (s), 152.2 (d, J_{CH} = 175 Hz), 149.4 (d, J_{CH} = 172 Hz), 137.6 (d, J_{CH} = 156 Hz), 124.2 (d, J_{CH} = 164 Hz), 121.2 (d, J_{CH} = 167 Hz), 113.2 (s, C_5Me_5), 111.6 (s, C_5Me_5), 104.3 (d, J_{CH} = 164 Hz), 17.0 (q, J_{CH} = 125 Hz, C_5Me_5), 15.8 (q, J_{CH} = 125 Hz, C_5Me_5). IR (KBr): 2960 (m), 2900–2860 (s), 2730 (w), 1600 (s), 1490 (m), 1475 (m), 1435 (s), 1405 (w), 1380 (w), 1290 (m), 1275 (m), 1155 (w), 1065 (w), 1020 (w), 1005 (w), 985 (w), 750 (s), 705 (m), 650 (m) cm^{-1}.

A single crystal measuring approximately 0.15 × 0.40 × 0.20 mm was sealed under nitrogen in a glass capillary, mounted on a Siemens P3 diffractometer, and analyzed by X-ray crystallography at 296 K. Although the connectivity of the atoms was established, the data were not good enough to provide detailed information on bond distances and angles. Details are given in the supplementary material.[25]

Reaction of $[(C_5Me_5)_2Sm]_2(\mu\text{-}\eta^2\text{:}\eta^2\text{-pyCHCHpy})$ with pyCH=CHpy. In the glovebox, pyCH=CHpy (22 mg, 0.12 mmol) was added to a stirred solution of **2** (120 mg, 0.12 mmol) in toluene. The reaction mixture darkened and was allowed to stir for 1/2 h. Solvent was removed under vacuum to leave, in near quantitative yield, solid **3** which was identified by ^1H NMR spectroscopy.

Reaction of $[(C_5Me_5)_2Sm]_2[\mu\text{-}\eta^3\text{:}\eta^3\text{-}1,2,3,4\text{-}(py)_4C_4H_4]$ with $(C_5Me_5)_2Sm(THF)_2$. In a manner similar to that above, $(C_5Me_5)_2Sm(THF)_2$ (70 mg, 0.12 mmol) was added to a stirred toluene solution of **3** (75 mg, 0.062 mmol). The color of the reaction mixture became lighter. After the reaction was stirred for 1/2 h, solvent was removed under vacuum to leave **2** as a solid in nearly quantitative yield identified by ^1H NMR spectroscopy.

Results and Discussion

$[(C_5Me_5)_2Sm]_2(pyCHCHpy)$, 2. $(C_5Me_5)_2Sm(THF)_2$, **1**, reacts rapidly with 1,2-bis(2-pyridyl)ethene to form red products.[1] If **1** is present in excess either by stoichiometry or by dropwise addition of the alkene to a solution of **1**, the $[(C_5Me_5)_2Sm]_2$(substrate) complex **2** is formed in 80% yield (eq 1). **2** was characterized by ^1H and ^{13}C NMR spectroscopy, IR spectroscopy, and complete elemental analysis. Although these data were consistent with a trivalent samarium complex with the formula shown in eq 1, they were not structurally definitive and an X-ray diffraction study was initiated. X-ray crystallography established the structure shown in Figure 1, although the quality of the data was not sufficient to discuss bond distances and angles.

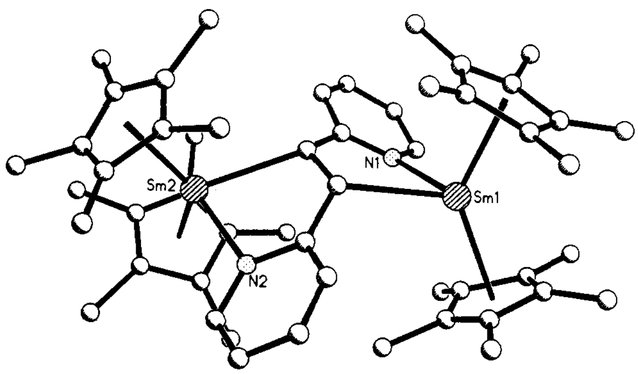
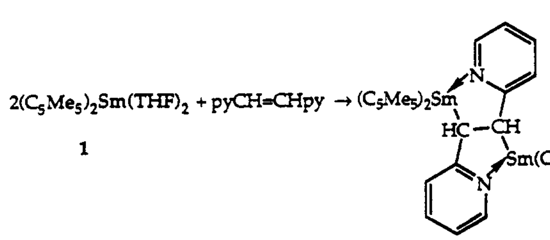


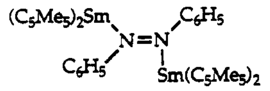
Figure 1. Ball and stick plot of crystallographically characterized $[(C_5Me_5)_2Sm]_2(\mu\text{-}\eta^2\text{:}\eta^2\text{-pyCHCHpy})$, **2**.



2

(1)

The structure of **2** is similar to the structure shown below of $[(C_5Me_5)_2Sm]_2(\mu\text{-}\eta^1\text{:}\eta^1\text{-PhNNPh})$, **4**, a complex formed from **1** and PhN=NPh.[10,11] In each case, two trivalent $(C_5Me_5)_2Sm$ units form single bonds to the two atoms originally involved in the double bond of the substrate. In **2**, each samarium atom is also coordinated by the nitrogen atom of a pyridine ring such that each samarium is formally eight-coordinate. In **4**, extra coordination in the solid state is achieved through long distance interactions between the metal centers and two ortho-hydrogen atoms on the same arene ring.[10,11] Complexes **2** and **4** are similar in that both react with 2 equiv of CO to form products in which the two CO molecules have inserted into the double bonds of the original organic substrate (eqs 2[11] and 3[1]). Hence, **2** is a typical example of a $[(C_5Me_5)_2Sm]_2$(substrate) organosamarium(II) reaction product.



4

$[(C_5Me_5)_2Sm]_2[\mu\text{-}\eta^3\text{:}\eta^3\text{-}1,2,3,4\text{-}(py)_4C_4H_4]$, 3. When $(C_5Me_5)_2Sm(THF)_2$ is added dropwise to pyCH=CHpy with a final Sm:alkene ratio of 1:1, complex **2** is not formed. Instead, another red product, **3**, is isolated in 78% yield. Complex **3** was characterized in the same manner as **2** and was identified by X-ray crystallography as $[(C_5Me_5)_2Sm[\mu\text{-}\eta^3\text{:}\eta^3\text{-}1,2,3,4\text{-}(py)_4C_4H_4]$ (Figure 2). In this case, a

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[24] 2 crystallizes from hexane with one hexane per bimetallic unit in the orthorhombic space group *Pbcn* with a = 14.693(5) Å, b = 15.415(3) Å, c = 50.173(7) Å, V = 11364(5) $Å^3$, and Z = 8 for D_{calcd} = 1.295 g cm^{-1} at 183 K. See supplementary material for details.

[25] 3 crystallizes from hexane/toluene with one hexane per bimetallic unit in the monoclinic space group *C2/c* with a = 17.397(4) Å, b = 29.338(9) Å, c = 15.063(3) Å, β = 123.810(13)°, V = 6388(2) $Å^3$, and Z = 4 for D_{calcd} = 1.34 g cm^{-1}. See supplementary material for details.

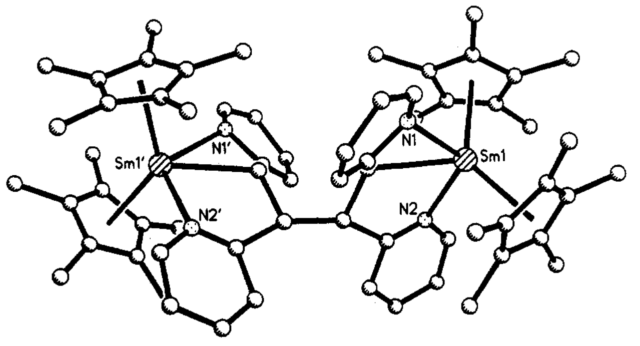
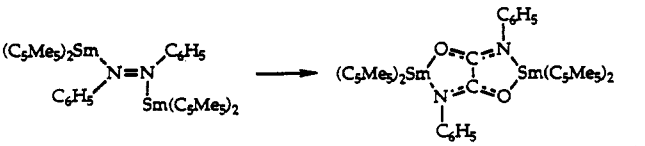
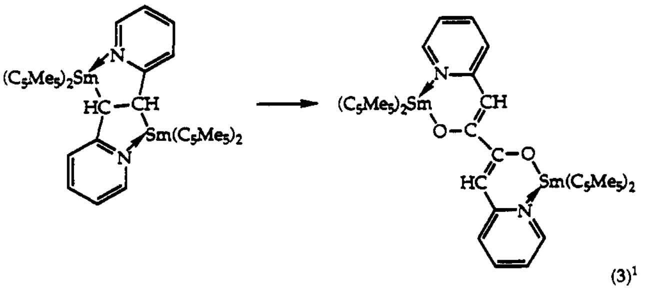


Figure 2. Ball and stick plot of crystallographically characterized $[(C_5Me_5)_2Sm]_2[\mu\text{-}\eta^3{:}\eta^3\text{-}1,2,3,4\text{-}(py)_4C_4H_4]$, **3**.

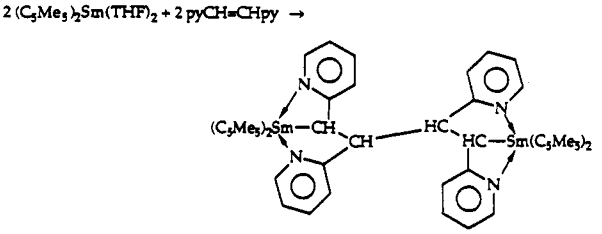


(2)[11]

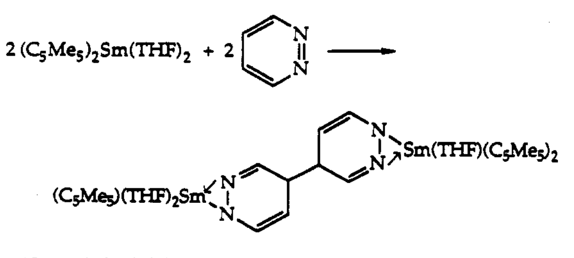


(3)[1]

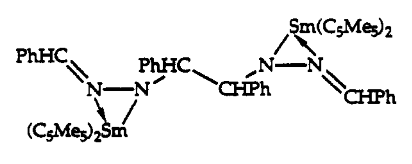
reductive coupling was induced by $(C_5Me_5)_2Sm(THF)_2$ (eq 4). Reductive coupling of pyridazine and benzaldehyde azine by $(C_5Me_5)_2Sm(THF)_2$ has previously been observed (eqs 5 and 6).[7]



3 (4)



(5)



(6)

The samarium atoms in **3** are formally nine-coordinate, a coordination number that is not as common as eight for $(C_5Me_5)_2Sm$-containing complexes, but one for which there are now at least ten examples.[26] $(C_5Me_5)_2Sm$ complexes with this coordination number often have chelating ligands as is found in **3**. Unfortunately, the crystallographic data on **3** were not good enough for a detailed analysis of bond distances and angles.

Interconversion of 2 and 3. The reaction chemistry of $(C_5Me_5)_2Sm(THF)_2$ and bis(2-pyridyl)ethene described so far is consistent with the single electron reduction chemistry of this divalent Sm(II) reagent.[27] Reduction of pyCH=CHpy by one Sm(II) center would generate a radical, $(C_5Me_5)_2Sm(pyCHCHpy)$, which could either react with another $(C_5Me_5)_2Sm(THF)_2$ unit to form **2** or dimerize to form **3**.

A more remarkable feature of this Sm(II)/bis(2-pyridyl)ethylene system is that **2** and **3** can be readily interconverted. As shown in eqs 7 and 8, addition of $(C_5Me_5)_2Sm(THF)_2$ to **3** forms **2** and addition of pyCH=CHpy to

$$\underset{\mathbf{3}}{[(C_5Me_5)_2Sm]_2[\mu\text{-}\eta^3{:}\eta^3\text{-}1,2,3,4\text{-}(py)_4C_4H_4]} + 2(C_5Me_5)_2Sm(THF)_2 \rightarrow 2\underset{\mathbf{2}}{[(C_5Me_5)_2Sm]_2(\mu\text{-}\eta^2{:}\eta^2\text{-}pyCHCHpy)} \quad (7)$$

$$\underset{\mathbf{2}}{[(C_5Me_5)_2Sm]_2(\mu\text{-}\eta^2{:}\eta^2\text{-}pyCHCHpy)} + pyCH{=}CHpy \rightarrow \underset{\mathbf{3}}{[(C_5Me_5)_2Sm]_2[\mu\text{-}\eta^3{:}\eta^3\text{-}(py)_4C_4H_4]} \quad (8)$$

2 forms **3**. Equation 7 is the first example of the reductive cleavage of a carbon—carbon single bond by an organometallic samarium(II) complex and adds to the list of bond cleavage reactions which can be effected by $(C_5Me_5)_2Sm(THF)_2$. The reaction is particularly surprising considering the steric demands of $(C_5Me_5)_2Sm(THF)_2$ and the relatively protected environment of the carbon—carbon bond which is cleaved. The pyridyl substituents may be important in this regard.

Reaction 8 is also surprising. Although **2** is clearly reactive with small molecule substrates such as CO (eq 3),[1] one would not expect formal insertion of a molecule as large as pyCH=CHpy into one of the Sm—C bonds of **2** to be sterically allowed. The closest reaction in the literature is shown in eq 9.[11] In this case, the $(C_5Me_5)_2Sm(N_2Ph_2)$ precursor is sterically unsaturated and can accommodate additional ligands.

$$[(C_5Me_5)_2Sm]_2(N_2Ph_2) + PhN{=}NPh \xrightarrow{THF} 2(C_5Me_5)_2Sm(N_2Ph_2)(THF) \quad (9)$$

The interconversion chemistry observed here suggests that the $(C_5Me_5)_2Sm$ unit has considerable flexibility in these complexes. We have previously noted that the $(C_5Me_5)_2Sm$ unit is sterically flexible since in both divalent and trivalent systems it can form stable crystallographically characterizable compounds with a variety of formal coordination numbers, even with very similar ligand sets.[26–30] It appears that the $(C_5Me_5)_2Sm$ unit is electronically flexible as well. For example, there is a growing list of trivalent complexes which revert under mild conditions to the divalent complex, $(C_5Me_5)_2Sm(THF)_2$:

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[26] Evans, W. J.; Foster, S. E. *J. Organomet. Chem.* **1992**, *433*, 79–94.

[27] Evans, W. J. *Polyhedron* **1987**, *6*, 803–835.

[28] Evans, W. J.; Drummond, D. K.; Hughes, L. A.; Zhang, H.; Atwood, J. L. *Polyhedron* **1988**, *7*, 1693–1703.

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[30] Evans, W. J.; Kociok-Köhn, G.; Foster, S. E.; Ziller, J. W.; Doedens, R. J. *J. Organomet. Chem.* **1993**, *444*, 61–66.

the trivalent species, $[(C_5Me_5)_2Sm]_2(N_2)$,[13] $[(C_5Me_5)_2Sm]_2(PhCCPh)$,[4] $[(C_5Me_5)_2Sm]_2(PhCHCH_2)$,[2] and $[(C_5Me_5)_2Sm]_2(PhCHCHPh)$,[2] all form the divalent $(C_5Me_5)_2Sm(THF)_2$ upon addition of THF. Hence, the $(C_5Me_5)_2Sm$ moiety may be able to accommodate changes in both oxidation state and coordination environment which allow interconversions of the type reported here to occur.

Conclusion

The formation of **2** or **3** from $(C_5Me_5)_2Sm(THF)_2$ and pyCH=CHpy demonstrates that with some unsaturated substrates both the reductive coupling and the double reduction pathways can be traversed with divalent organosamarium(II) reagents. Obviously, in these cases, careful control of stoichiometry and the order of addition of reagents is critical. The reductive cleavage of the carbon—carbon bond in **3** by $(C_5Me_5)_2Sm(THF)_2$ shows that organolanthanide reagents should be included in efforts to develop facile, selective, carbon—carbon activation reactions. The reaction of **2** with pyCH=CHpy to form **3** raises interesting questions regarding organolanthanide reactivity in complexes which superficially appear to be sterically and electronically saturated.

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Supplementary Material Available: Textual description of X-ray data, details of the refinement and tables of crystal data, positional parameters, bond distances and angles, and thermal parameters, and diagrams of conformations 1 and 2 of **3** (26 pages). Ordering information is given on any current masthead page.

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