#### **Supplementary Information**

#### Coordination Chemistry of Trimethylsilylphosphaalkyne: A Phosphaalkyne Bearing a Reactive Substituent

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#### NMR spectra of crude reaction mixtures.





Figure S1. <sup>31</sup>P NMR spectrum of compound 2.



-1 -2 -5 5 4 f1 (ppm) -3 -4 

**Figure S2**. <sup>1</sup>H NMR spectrum of compound **2**. The peak at 0.31 is from silicon grease as an impurity.



**Figure S4**. <sup>1</sup>H NMR spectrum of compound **3**. The peak at 0.30 is from silicon grease as an impurity.



Figure S5. <sup>19</sup>F NMR spectrum of compound 3.





**Figure S7**. <sup>1</sup>H NMR spectrum of compound **4**. The peak at 0.30 is from silicon grease as an impurity.







Figure S9. <sup>1</sup>H NMR spectrum of compound 5. The resonance at 2.13 is from residual toluene.



**Figure S10.** <sup>31</sup>P NMR spectrum of the product formed from the reaction of 5 with TBAT. \* denotes  $[(N=N)_2Mo(dppe)_2]$  impurity.



Figure S12. <sup>1</sup>H NMR spectrum of compound 6. The resonance at 2.11 is from residual toluene.



**Figure S13**. <sup>31</sup>P NMR spectrum of compound **7** before a substantial amount of by-products had been formed by thermal reaction.



**Figure S14**. <sup>1</sup>H NMR spectrum of compound **7** before a substantial amount of by-products had been formed by thermal reaction.



Figure S15. <sup>31</sup>P NMR spectrum of thermal reaction products of compound 7.



Figure S17. <sup>1</sup>H NMR spectrum of compound 8. The resonance at 2.13 is from residual toluene.



Figure S19. <sup>1</sup>H NMR spectrum of compound 9. The resonance at 2.11 is from residual toluene.