Supporting Information

Rare-Earth Silylamide-Catalyzed Selective Dimerization of Terminal Alkynes and Subsequent Hydrophosphination in One-Pot

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Procedure. ^{1}H, ^{13}C, and ^{31}P NMR spectra were recorded at 400, 100, and 160 MHz, respectively. IR spectra were taken on an FT-IR spectrophotometer. Mass spectra (EI) were obtained at 70 eV on a GC-MS apparatus. Melting points are uncorrected. All reactions were carried out under argon.

Materials. Toluene was distilled from sodium/benzophenone ketyl immediately prior to use. $Ln[N(SiM_3)_2]_3$ were prepared from anhydrous $LnCl_3$ and $NaN(SiMe_3)_2$[1] in THF, according to the literature.[2] The alkynes **1b, 1c, 1e**,[3] **1d**,[4] and **1f**[5] were prepared by the reported methods. Diphenylphosphine was prepared by reduction of triphenylphosphine with lithium metal in THF. All other materials were commercially available and were used after drying and distillation.

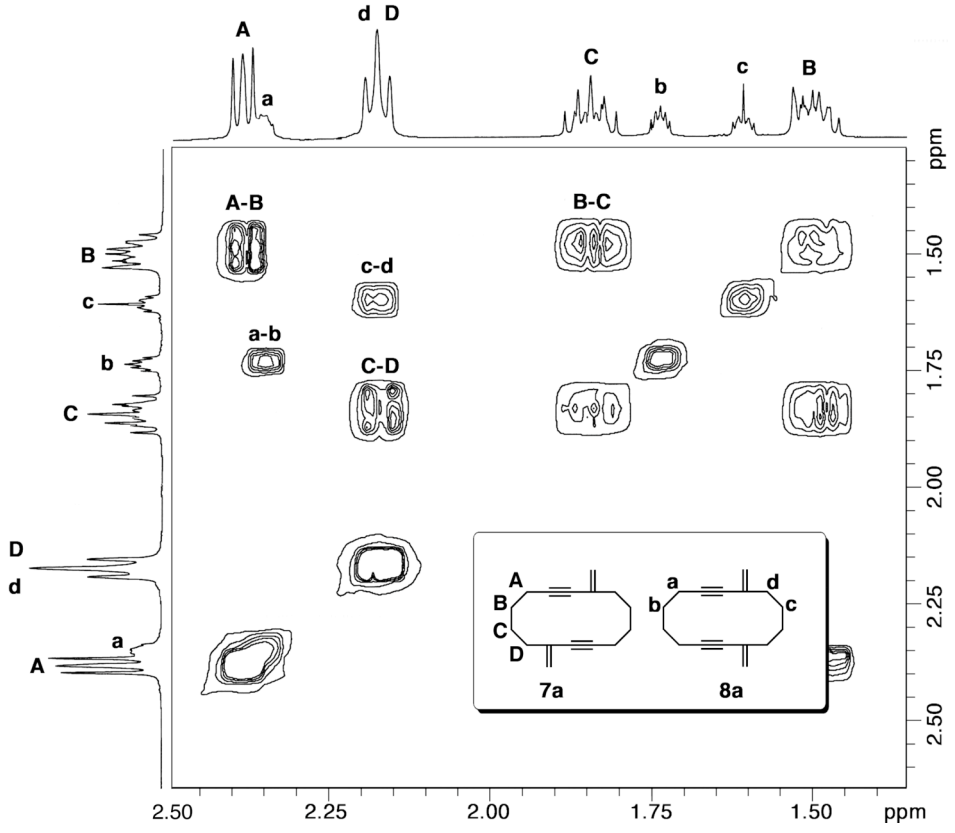

Figure S1. H-H COSY Spectra of **7a** and **8a**

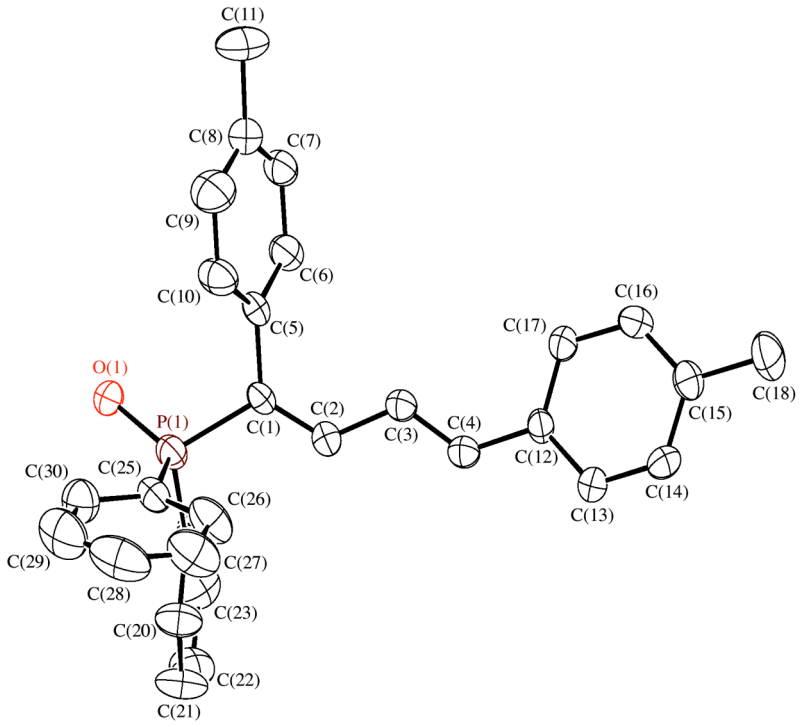

Figure S2. ORTEP drawing of **10c**.

References

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(4) Jiang, X. K.; Ji, G. Z.; Wang, D. Z. R. *J. Fluorine. Chem.* **1996**, *79*, 173-178.

(5) Hommes, H.; Verkruijsse, H. D.; Brandsma, L. *J. Chem. Soc., Chem. Commun.* **1981**, 366-367.