

## Synthesis and molecular structures of the first phosphoranylidene complexes of rare earth metals

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### Experimentals

All experiments were performed in an atmosphere of dry, oxygen-free nitrogen or argon using Schlenk technique and solvents dried over sodium/benzophenone and distilled prior to use over NaAlEt<sub>4</sub>. Lu(CH<sub>2</sub>SiMe<sub>3</sub>)<sub>3</sub>(THF)<sub>2</sub> and Y(*o*-Me<sub>2</sub>NCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>)<sub>3</sub> were synthesized according to literature methods.<sup>1,2</sup> Ph<sub>3</sub>P and *o*-CH<sub>3</sub>OC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>Cl were used as supplied (Aldrich, Fluka). NMR spectra were recorded using the Bruker ARX 200 and 300 spectrometers. Lu was determined complexometrically against xylenol orange after digestion by 60% HClO<sub>4</sub> at pH 6 to 7.<sup>3</sup>

*Triphenylphosphonium-ortho-methoxybenzylidene 1*.<sup>4</sup> To a solution of Ph<sub>3</sub>P (5.25 g, 20 mmol) in 1:1 Et<sub>2</sub>O-acetone mixture (200 mL) *o*-CH<sub>3</sub>OC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>Cl (3.91 g, 25 mmol) was added *via* syringe. The reaction mixture was allowed to stir at room temperature for 3 days followed by evaporation of the solvent to dryness and washing of the *o*-methoxybenzyl-triphenylphosphonium chloride with several portions of pentane. Yield after drying under vacuum: 3.25 g (40%). To a suspension of thus obtained white crystalline material (2.91 g, 7 mmol) in dry THF (40 mL) NaH (0.48 g, 20 mmol) was added at once. In few minutes the reaction mixture became orange in colour. It was allowed to stir under argon at room temperature for 2 days resulting in formation of deep-orange solution and white suspension of NaCl and an excess of NaH. After filtration and evaporation of THF 1.81 g (98% yield) of **1** as an orange powder was obtained.

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$^1\text{H}$  NMR (200 MHz,  $\text{C}_6\text{D}_6$ ,  $+23^\circ\text{C}$ ):  $\delta$ , 7.74 – 7.64, 7.03 – 6.64 (m, 19H, Ph, Aryl), 3.21 (s, 3H,  $\text{OCH}_3$ ), 3.08 (d, 1H,  $\text{P}=\text{CH}$ ,  $J_{\text{PH}} = 20\text{ Hz}$ );  $^{31}\text{P}$  NMR (81 MHz,  $\text{C}_6\text{D}_6$ ,  $+23^\circ\text{C}$ ):  $\delta$ , 10.8. EA for  $\text{C}_{26}\text{H}_{23}\text{OP}$ : calcd. C 81.66%, H 6.06%, observed C 81.58%, H 5.90%.

## References

- 1 H. Schumann, J. Müller, J. Organomet. Chem. **1978**, 146, C5.
- 2 M. Booiij, N.H. Kiers, H.J. Heeres. J.H. Teuben, J. Organomet. Chem. **1989**, 364, 79.
- 3 M. Glanz, S. Dechert, H. Schumann, D. Wolff, J. Springer, Z. Anorg. Allg. Chem. **2000**, 626, 2467.
- 4 L.K. Johnson, S.C. Virgil, R.H. Grubbs, J.W. Ziller, J. Am. Chem. Soc. **1990**, 112, 5384.