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Supporting Information

$\text{Tp}^*\text{Rh}(\text{C}_2\text{H}_4)(\text{PMe}_3)$ **1a**

A stirred solution of $\text{Tp}^*\text{Rh}(\text{C}_2\text{H}_4)_2$ (0.81 g, 1.75 mmol) in THF (40 ml) is cooled to $-30\text{ }^\circ\text{C}$ and PMe_3 is added (1.75 ml of a solution 1M in THF, 1.75 mmol). The initial orange color of the solution becomes pale yellow. After stirring for 15 min. at $-30\text{ }^\circ\text{C}$, the solution is warmed to room temperature and stirred during 1 h, after which time the solvent is removed in vacuo, yielding a microcrystalline solid. The NMR spectra of the solid obtained reveals quantitative formation of **1a**, pure enough for preparative purposes. The high sensitivity of this complex to oxygen, coupled with its high solubility in organic solvents prevents its recrystallization and, hence, the elemental analysis determination.

$\text{Tp}^*\text{Rh}(\text{H})(2\text{-C}_4\text{H}_3\text{S})(\text{PMe}_3)$ **2a**

0.05 g of **1a** (0.1 mmol) are dissolved in thiophene (30 ml) and heated at $90\text{ }^\circ\text{C}$ during 6h. The yellow solution changes to brown-orange. The thiophene is evaporated under reduced pressure and a brown solid is obtained which contains, as revealed by NMR, 85 % of the title compound, together with small amounts of the isomer $\text{Tp}^*\text{Rh}(\overline{\text{CHCHCHCHS}})(\text{PMe}_3)$. Extraction with petroleum ether gives a solution enriched in complex **2a**, although complete separation upon crystallization could not be achieved.

$\text{Tp}^*\text{Rh}(\overline{\text{CHCHCHCHS}})(\text{PMe}_3)$ **3a**

Complex **1a** (0.23 g, 0.45 mmol) is dissolved in thiophene (50 ml) and transferred to a photoreactor. The mixture is then irradiated, at room

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temperature, for 2 h and the resulting orange solution is evaporated to dryness. NMR monitoring of the crude solid obtained reveals the presence of three species: complex **3a** (60 %), complex **2a** (20 %) and another unidentified species (20 %). Extraction of the mixture with petroleum ether (*ca* 2 x 10 ml) leaves **3a** as a solid, which can be crystallized from THF.

Anal. Calcd.: C, 47.16; H, 6.30; N, 15.00. Found: C, 47.16; H, 5.96; N, 14.84.

Complexes **1b-3b** are prepared following procedures analogous than described for **1a-3a**.