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Synthesis, Characterization and Electrocatalytic H₂ Production of Base-containing Fe/S and Fe/Se Clusters

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Abstract: Reaction of base-containing diphosphine ligand $\text{NP}_2^{\text{S}_2}$ ($\text{NP}_2 = (\text{Ph}_2\text{PCH}_2)_2\text{NCH}_3$) with $[\text{Fe}_3(\text{CO})_{12}]$ in THF at room temperature forms $[\{\text{Fe}_2(\text{CO})_6(\mu\text{-PPh}_2)\}_2(\mu_4\text{-S}_2)]$ (**1**) and $[\text{Fe}_3(\text{CO})_8(\mu_3\text{-S})_2\{\text{kP-PPh}_2\text{CH}_2\text{N}(\text{CH}_3)\text{CH}_2\text{P}(=\text{S})\text{Ph}_2\}]$ (**2**) resulting from P=S bond cleavage and P-Fe bond formation. Similarly, $\text{NP}_2^{\text{Se}_2}$ reacts with $\text{Fe}_3(\text{CO})_{12}$ to provide $[\{\text{Fe}_2(\text{CO})_6(\mu\text{-PPh}_2)\}_2(\mu_4\text{-Se}_2)]$ (**3**) and $[\text{Fe}_3(\text{CO})_7(\mu_3\text{-Se})_2\{\text{k}^2\text{P,P-PPh}_2\text{CH}_2)_2\text{NCH}_3\}]$ (**4**). All the compounds have been completely characterized by elemental analyses, IR, ^1H NMR, ^{13}C NMR and ^{31}P NMR spectroscopy and structurally determined by X-ray crystallography. Electrochemical studies reveal that when using HOAc or TFA as a proton source, **2** and **4** exhibit catalytic H₂ production.

Keywords: Fe/S Cluster; Fe/Se Cluster; Cyclic voltammetry; H₂ production

1. Introduction

The development of renewable energy is nowadays one of the most important challenges as global energy consumption is rising significantly. Molecular hydrogen is a carbon-free fuel that could become the energy carrier of the future, thus making the reversible interconversion of protons to molecular hydrogen a key process for future energy schemes. Nature is able to catalyze this process by a class of

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