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Synthesis, Characterization and Electrocatalytic H₂ Production of Diiron

Complexes Containing SeNC Donors

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Abstract: The three-component reactions of ArNHC(=Se)OCH₃, Et₃N and Fe₃(CO)₁₂ afford the [Et₃NH]⁺ salt of the (μ-ArN=COCH₃)Fe₂(CO)₆(μ-Se⁻) cluster anion. The Se-centered cluster anion reacts with CH₃I to give two pairs of isomers (μ-k²N,C-ArN=COR)Fe₂(CO)₆(μ-SeCH₃) (**1**-e, **1**-a, Ar = C₆H₅) and (μ-k²N,C-ArN=COR)Fe₂(CO)₆(μ-SeCH₃) (**2**-e, **2**-a, Ar = 4-ClC₆H₄). Furthermore, trapping the Se-centered cluster anion (μ-ArN=COR)Fe₂(CO)₆(μ-Se⁻) with PPh₂Cl offers (μ-k²N,C-ArN=COR)Fe₂(CO)₆(μ-k²Se,P-SePPh₂) (**3**, Ar = C₆H₅, **5**, Ar = 4-ClC₆H₄, R = CH₃; **4**, Ar = C₆H₅, **6**, Ar = 4-ClC₆H₄, R = CH₂Ph). Reactions of **1** with monophosphine L lead to the corresponding substituted complexes (μ-k²N,C-PhN=COCH₃)Fe₂(CO)₅(L)(μ-SeCH₃) (**7**-e, **7**-a, L = PPh₃; **8**-e, L = PPh₂Py). All the new complexes have been completely characterized by elemental analyses, IR, ¹H NMR, ¹³C NMR, and ³¹P NMR spectroscopy and structurally determined by X-ray crystallography. Electrochemical studies reveal that when using HOAc or TFA as a proton source, all of the complexes can catalyze H₂ production.

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