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Azo-amide palladium(II) complexes: synthesis, characterization and application in C-C cross-coupling reactions

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Abstract

The newly designed tridentate ligands bis-2,2'-(N-alkylamino)azobenzene, **1a-1d**, have been prepared by the reaction between 2,2'-diaminoazobenzene and alkyl halides in the presence of K₂CO₃. These ligands were reacted with Na₂[PdCl₄] in a 1:1 ratio in methanol to give the new Pd(II) complexes **2a-2d**. All the compounds were characterized by ¹H NMR, IR spectroscopy and elemental analysis. Furthermore, the solid-state structures of the ligand **1a** and two complexes (**2a** and **2c**) were determined using single crystal X-ray diffraction analysis. The diffraction analysis revealed that the ligands bind with the Pd(II) ion in a monoanionic tridentate (N,N,N) fashion, offering a distorted square planar geometry where the fourth position is occupied by one chloride ligand. The air/moisture stable complex **2a** was employed as an efficient catalyst for the Suzuki and Heck reactions under mild conditions. The catalyst exhibits high catalytic activities for the coupling of several aryl halides with phenyl boronic acid and styrene, providing excellent yields. Further, the catalyst can be easily recovered by simple chromatographic separation and reused up to three times without significant loss of its catalytic activity.

Keywords: Bis-2,2'-(N-alkylamino)azobenzene, Crystal structure, Palladium(II), Catalysis, Suzuki and Heck Reaction

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