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Pd-NHC PEPPSI Complexes: A Breakthrough in Green and Efficient Cross-Coupling Catalysis

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Description

Modern organic synthesis heavily relies on palladium-catalyzed cross-coupling reactions to form carbon-carbon and carbon-heteroatom bonds, which are important for producing pharmaceuticals, natural products and agrochemicals. These reactions have become popular at both the laboratory and industrial levels due to their simplicity and versatility. Early catalytic systems used palladium(II) sources like $PdCl_2$ and $Pd_2(dba)_3$, often combined with optional ligands such as triphenylphosphine (PPh_3) and inorganic bases like K_2CO_3. Over time, advancements in catalyst design have led to the development of more sophisticated precatalysts capable of handling more challenging substrates with enhanced selectivity and milder reaction conditions. A significant advancement in this field was the 2006 development of Pyridine-Enhance Precatalyst Preparation, Stabilization and Initiation (Pd-PEPPSI) complexes by organ and colleagues. These robust palladium(II) complexes are characterized by an N-Heterocyclic Carbene (NHC) ligand and a labile 3-chloropyridine ligand. The 3-chloropyridine ligand is a throw-away ligand, not involved in the catalytic cycle, allowing pyridine to be used as a less expensive and less toxic alternative. These 16-electron species are stable in air and water until they are reduced to Pd(0) during the catalytic cycle. Due to their stability and effectiveness in cross-coupling reactions, Pd-PEPPSI complexes have since become commercially available and widely utilized.

palladium catalysis

In 2022, researchers began examination more sustainable and environmentally friendly approaches to palladium catalysis by using biosourced NHC ligand precursors derived from natural and abundant methylxanthine alkaloids like caffeine and theophylline. Our group, along with others, successfully synthesized Pd-NHC PEPPSI complexes using these natural products. For example, the alkylation of caffeine with methyl iodide produced tetramethylxanthinium iodide, which was then converted into the (PdI_2(NHC)(Py)) complex in high yield. Parallel efforts by employed similar strategies, showing that these complexes could also be synthesized with various ligand environments, including 3-chloropyridine and N-methylimidazole. The catalytic activity of these Latest Pd-NHC PEPPSI complexes was tested in cross-coupling reactions, one of the most widely used palladium-catalyzed processes. The results were promising, with complexes 1-3 demonstrating high catalytic efficiency. The reactions were optimized for environmental impact reduction by using low catalyst loadings, operating at 40°C in green, water-based solvents and using innocuous bases like K_2CO_3 in the presence of air. Under these conditions, a wide range of biaryl derivatives were successfully produced by coupling phenylboronic acids with aryl bromides and iodides, including those with electron-donating and electron-withdrawing substituents. Even aryl chlorides, typically less reactive, could be activated under more forcing conditions, especially when electron-withdrawing groups were present on the aromatic ring.

Hirshfeld surface

Building on the victory of these initial tests, we expanded the use of Pd-NHC PEPPSI complexes to more challenging catalytic applications, such reactions and C(sp^2)-H arylation reactions. These further demonstrated the versatility and effectiveness of Pd-NHC PEPPSI complexes, even under mild and environmentally friendly conditions. Structural studies, including X-ray crystallography and Hirshfeld surface analysis, provided deeper insights into the properties of these complexes, enhancing our understanding of their catalytic behavior. In summary, the development of Pd-NHC PEPPSI complexes represents a signi icant advancement in palladium-catalyzed cross-coupling reactions. Their successful application in C(sp^2)-H arylation reactions indicates their potential for broader use in both academic and industrial settings. As research continues, these environmentally friendly catalysts could play a key role in developing novel methodologies for the efficient and sustainable synthesis of complex molecules.