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Carboranes and metallacarboranes: Advances and new perspectives

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Most of the carborane derivatives of the icosahedral (C_2B_{10}) or small cage (C_2B_4) systems are formed, by varying the groups on the cage carbons. This is usually accomplished in the original carborane synthesis by reacting substituted acetylenes with either the $B_{10}H_{14}$ or B_5H_9 precursors. These reactions led directly to the carbons adjacent carboranes in which the carbon atoms occupy adjacent positions in the cage. The larger cages are obtained as *closo*-icosahedra, while the small cage, C_2B_4 -carboranes, have nido- structures. There is another geometry of the small cages in which the carbon atoms are separated by a boron atom. Although these carbons apart or *nido*-2,4-(CR)₂ B_4H_6 species are thermodynamically more stable and are more symmetric than the carbons adjacent isomers, they are not as well studied. The main reasons for the relative scarcity of information on the carbons systems lie in their method of preparation. They must be synthesized from their carbons adjacent analogues through sequential oxidative cage closure/reductive cage opening reactions. The historic perspective of the chemistry of carboranes and metallacarboranes with the latest findings in our research involving boron nanomaterials will be presented in detail.

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Stille reaction of β-nitro and β-tosyl substituted styryl bromides

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The palladium-catalyzed coupling of organostannanes with organic electrophile (Stille reaction) has become an important L synthetic tool in organic chemistry (eq. 1). Several years ago, we became interested in applying the Stille reaction to α -stannyl- α , β -unsaturated carboxylic esters. We found that these esters reacted with acid chlorides to afford good yields of coupling products (eq. 2). However, the reaction failed when it was attempted with vinylic bromides or iodides. We hypothesized that this result could be changed by reversing the roles of the reactants in the coupling process. Thus, a study of the Stille coupling of a series of functionalized styryl bromides with 1-propenyltributyltin was undertaken (eq. 3). This reaction afforded low to moderate yields of 1, 3-dienes. Interestingly, the best yield of product was obtained using methyl a-bromocinnamate, the styryl bromide substituted with the more electron-withdrawing group (R=CO₂Me). Consequently, we decided to examine the Stille reaction of β -nitro and β -tosyl (β -(p-MeC₆H₄SO₂)) substituted styryl bromides (eq. 4). The β -bromo- β -nitro-styrenes 1 and 2 reacted with a variety of organostannanes using bis (acetonitrile) dichloropalladium (II) as catalyst and n-methyl-pyrrolidinone (NMP) as solvent at room temperature. Good yields of 2-nitro-1, 3-dienes were obtained as a mixture of isomers. The Stille reaction of β -bromo- β -tosyl-styrenes 3 and 4 was also investigated. In this case, the best yields of coupling products were obtained using an excess of organotin compound (1.5 equivalents), a mixture of bis (acetonitrile) dichloropalladium (II) (5% molar) and copper iodide (10% molar) as catalysts and NMP as solvent at room temperature. Again, good yields of products, as a mixture of isomers, were obtained. The Stille reactions of β -nitro and β -tosyl substituted styryl bromides reported here constitute an useful synthetic tool towards the preparation of functionalized 1, 3-dienes. These unsaturated molecules are among the most versatile organic compounds which participate in a wide variety of applications, including fine chemical synthesis and polymer chemistry.

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