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2	Submitted on 6 th August, 2021 and Accepted on 8 th January 2022	Recent Advances in Copper-Catalyzed Carbon Chalcogenides Cross-Coupling Reactions	Totan Roy, Atanu Mahata and Debasish Kundu, <i>Curr. Org. Synth.</i> , 2023, 20(3), 267-277 https://benthamsience.com/public/article/121839
3.	Submitted on 9 th January 2023 and Accepted on 15 th March 2023	Transition Metals Catalyzed Direct C-H Chalcogentaion of Arenes and Heteroarenes	Atanu Mahata, Madhusudan Garain, Totan Roy, Dilip Gorai and Debasish Kundu, <i>Curr. Org. Synth.</i> , 2024, 21(6), 764-795 https://www.eurekaselect.com/article/131327

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MINI-REVIEW ARTICLE

Synthesis of Aryl/Heteroaryl Selenides Using Transition Metals Catalyzed Cross Coupling and C–H Activation

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Abstract: Aryl and heteroaryl selenides are an important class of organic compounds and their synthesis has been widely studied all over the world in the last two decades. Transition metals catalyzed cross-coupling and directed C–H activation in unactivated arenes in the presence of diselenides/aryl selenols are found to be the most important tools for their synthesis. In recent years different transition metal catalysts were found to be effective to perform C–Se cross-coupling in both aryl and heteroaryl rings. The present review article covers all the recent advances made in the last ten years in the field of the synthesis of aryl and heteroaryl selenides through homogeneous and heterogeneous transition metals catalyzed cross-coupling reactions and directed selenylation via C–H bond activations.



Dr. Debasish Kundu

Keywords: Cross-coupling, C–H activation, transition metal, selenides, homogeneous and heterogeneous catalysis, organic compounds.

1. INTRODUCTION

In the last two decades, researchers have witnessed the different transition metals catalyzed cross-coupling reactions as powerful organic synthetic methods for the generation of several biologically important molecules and natural products based on Carbon-heteroatom bonds. The diaryl diselenide compounds are attracting considerable attention from organic chemists due to their potential biological activities (e.g., anticancer, antitumor, antiviral, antimicrobial, antioxidant and antithyroid) [1-9]. They also exhibit tremendous catalytic activity in modern organic synthesis [10]. Diselenides, which mostly serve as a valuable source of organoselenium reagents, allows the inclusion of selenium moieties into the aromatic rings of organic molecules for the successful implementation in the generation of C–Se bonds [11]. A number of sustainable, new, and greener methodologies have been reported in the literature to prepare unsymmetrical diaryl selenides. Although C–Se cross-coupling reactions under conventional pathways suffer from several drawbacks such as the use of expensive and toxic solvents, longer reaction times, stoichiometric or greater quantity of metallic reagents, and high reaction temperature [12-19]. Selenation via C–H activation has been introduced as a sustainable alternative for this purpose.

Due to the much lower toxicity, availability, and acceptable stability of selenium, diselenides have received significant attention because they have been postulated as one of the most attractive sources of selenium as well as important selenylating agents in the cross-coupling reactions with pre-functionalized reactants like haloarenes, aryl boronic acids, diazonium salts and aryl amines to

synthesize unsymmetrical selenides [20-26]. The preparation of diaryl selenides from organoboron compounds is traditionally less explored compared to aryl halides. Based on the earlier reports of an ever-increasing number of groups, the researchers were intrigued by the chemistry of Se–Se bond in diselenides because it can generate nucleophiles (RS^-), electrophiles (RS^+) or radicals (RS^\cdot) which underwent various nucleophilic, electrophilic as well as photochemical transformations.

Cross-coupling reactions via transition metals catalysis have emerged as an interesting and appropriate technique for the synthesis of organoselenides [27-34]. Synthesis of highly potent diaryl selenides through such a concise and convenient catalytic route has motivated the area of research at its vital stage of development. Over the past few years, significant developments in C–Se cross-coupling chemistry have been successfully achieved by employing transition metals like copper, silver, iron, nickel, palladium, indium, etc., but catalytic systems consisting of some transition metals are not highly desirable and anticipated due to their tremendous cost, toxicity, and low turnover number. Although, the metal-assisted transformations are highly efficient, regio- and stereoselective leading to a decrease in the production of waste, the costly and toxic heavy metal complexes go through difficulties in the separation of soluble metal catalysts from the reaction centre. A number of specialized works toward the metal-free synthesis of diaryl selenides are available in the literature [35-39], but our current research focuses on the applicability of transition metals to catalyze their synthesis.

In recent times, selenylation reactions of activated $C(sp^2)-H$ bonds by transition metals catalysis have gained growing importance regarding photochemical transformations in modern organic chemistry. Transition metals-assisted aromatic C–H oxygenation [40-42], amination [43-45], and halogenation [46-48] reactions have been successfully achieved by researchers in synthetic

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MINI-REVIEW ARTICLE

Recent Advances in Copper-Catalyzed Carbon Chalcogenides Cross-Coupling Reactions

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Abstract: Cu-catalyzed carbon-heteroatom bond formation is a powerful tool in the field of organic synthesis. In the past two decades, numerous Cu-based catalytic systems are developed in both homogeneous and heterogeneous forms. Important developments have been reported on Cu-based catalytic systems in the field of C-Chalcogenide cross-coupling in the last few decades. Where homogeneous Cu/L-based catalytic systems are found to perform reactions with high selectivity, heterogeneous supported-Cu and Cu-based nanoparticles are found to perform the reactions under sustainable conditions and high recyclability of catalytic systems. This present overview mainly focuses on the recent advances and applications in this fast-growing research field with an emphasis on copper-catalyzed cross-coupling generations of carbon-chalcogenide (S/Se/Te) bonds.

Keywords: Copper, selenium, sulfur, tellurium, cross-coupling, organo chalcogenides.

1. INTRODUCTION

The formation and necessity of carbon-heteroatom bond for the synthesis of organo-sulphides, selenides, and tellurides attract attention due to their profound applications in the field of catalysis [1], material science [2], biological, environmental, and pharmaceutical chemistry [3]. There has been an increasing interest in selenium and tellurium chemistry from the effective Se- and Te-based organocatalysts in several functional group transformations under sustainable conditions for the synthesis of bioactive molecules [4].

Transition metal-catalyzed cross-coupling reactions provide a powerful tool for the synthesis of a wide array of organic compounds via C-C and C-heteroatom cross-coupling within unsaturated carbon centres [3, 5-8]. However, the progress in carbon-chalcogen bond formation via copper-mediated or catalyzed cross-coupling reactions covers a variety of functional groups as well as the stability of starting materials and products due to the essentially required harsh reaction conditions, such as high temperatures-typically 150-200°C and for the extended reaction time from several hours to few days [9]. Recently, environment-friendly nanocrystals were successfully applied for C-Se cross-coupling under benign conditions [10]. Although the chemistry of C-Te bond formation is still in its early stages, the catalytic C-S and C-Se

cross-coupling reactions via decyanative cross-coupling or nucleophilic addition of ArSe to aryl halides, aryldiazonium salts, and arylboronic acids have received a lot of attention in recent years [3, 8, 11-14]. Due to the catalyst poisoning nature of sulphur-containing compounds, it becomes the most challenging factor for C-S cross-coupling, whereas a small number of thiols or disulphides can destroy the catalytic activity [15]. However, the recent development of efficient catalytic systems has successfully ruled out the mentioned problems to perform C-S coupling. Catalytic investigation was further extended in performing C-Se and C-Te cross-coupling.

Catalytic systems with other transition metals like nickel [16], cobalt [17], and iron [18] have also been employed as catalysts; such systems suffer from serious drawbacks such as metal toxicity, low turnover numbers, etc. development of Cu-based heterogeneous catalytic systems were employed in such coupling reactions due to its low cost and ready accessibility. The use of Cu-based heterogeneous catalysts has the distinct benefit of decreasing the probability of Cu leaching from the surface. A large number of attractive Cu-catalyzed cross-coupling reactions have been reported by various research groups. In recent years, diarylchalcogenides-containing C-S and C-Se bonds occur as component of drugs like AZD4407 [19a, b] vortioxetine [19c], chlorpromazine [19d] etc. These are effective and biologically active molecules against various diseases like cancer, HIV, Alzheimer's, asthma, Parkinson's, etc., demanding the continuous development in the synthesis of carbon-chalcogen compounds to meet the growing demand in many pharmaceutical applications [19]. As shown in

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REVIEW ARTICLE

Transition Metals Catalyzed Direct C-H Chalcogenation of Arenes and Heteroarenes

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Abstract: Transition metals catalyzed C-H bond activation reactions have appeared as an emerging field to introduce different functional groups in the inactivated saturated and unsaturated C-H bonds. C-S and C-Se bond constructions in aromatic scaffolds are very interesting due to the important applications of organochalcogen reagents in pharmaceutical chemistry and the material world. The introduction of sulphur or selenium moiety to an inert C-H functionality of an arene under transition metal catalysis has become one of the prime challenges and targets in recent years. In this perspective, various transition metals such as Cu, Ni, Co, Pd, Rh, Ru *etc.* have been extensively studied. Aromatic arenes owning bearing suitable directing groups appeared as the most promising coupling partners to selectively synthesize differently substituted aryl sulfones and aryl sulfides/selenides. The synthetic strategies were highly convenient owing to the regioselectivity of products, broad substrate scope, mild reaction conditions and excellent functional group tolerance. The current review article comprehensively summarizes the extent of C-S/Se bond formation *via* transition metal-catalyzed C-H bond activation with the assistance of directing groups to govern the site selectivity.

Keywords: Transition metal catalysis, sulfenylation, selenylation, C-H activation, diaryl diselenides, directing groups.

1. INTRODUCTION

Transition metals promoted catalytic reactions have reached a paramount level of sophistication and have been enriched by the incorporation of chalcogen atoms into activated C-H bond functionalities, emerging as outstanding strategies in modern organic synthesis.[1-8] In addition to the significant advancements in organometallic chemistry, notable innovations have been achieved in the area of C-Z (Z = S, Se) bond constructions as these structural motifs demand massive applications in pharmaceutical chemistry, [9, 10] material science[11, 12] and fluorescence spectroscopy [13, 14]. Due to prevalent structural fragments, acceptable stability and excellent functionality, the C-H bond in the aromatic ring has been extensively analysed in organic chemistry [15-17]. In the past few years, researchers have successfully employed transition metal catalysis to achieve C(sp²)-H bond functionalization reactions such as C-H halogenation[18, 19] oxygenation [20, 21] acylation [22] and alkylation [23, 24]. Synthesis of aryl-substituted chalcogenides *via* traditional methods involves harsh reaction conditions, poor functional group tolerance, elevated reaction temperature and affording minimum yields. However, transi-

tion metal-catalyzed direct transformation of an unreactive C(sp²)-H functionality to a C-chalcogen framework has emerged as a powerful tool to realize enormous promising conversions as these methodologies require mild reaction conditions, step and atom economy, sustainable and environmentally benign green protocols.[25-27] The ease of selective cleavage of a particular C-H bond of arenes is overwhelmed by the presence of directing groups and assistance of coordinating ligands which directs a transition metal atom into the close proximity of a specific C-H centre.[28-30] Besides, the strong coordinating nature of the chalcogen atoms (S and Se) has enabled them to get incorporated into an inert C-H motif. The excellent regioselectivity, site-selectivity, wider substrate scope and outstanding functional group compatibility of the arenes undergoing C-H bond functionalization reactions under standard protocols have been at the forefront of our current investigations.

In the past few years, activated C-H bond functionalization reactions of arenes under metal- and solvent-free protocols have extensively been employed to highlight the significant contribution to the sustainable development of green chemistry [31-34]. It is worth mentioning that the advancement of novel and practical catalytic methods involving C-S/Se bond constructions through transition metal-promoted direct C-H activation is still less explored in comparison to C-C, C-N and C-O bond functionalization reactions. In con-

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