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RECENT SYNTHESIS AND APPLICATIONS OF N- ARYL AMINES: A MINI REVIEW

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ABSTRACT

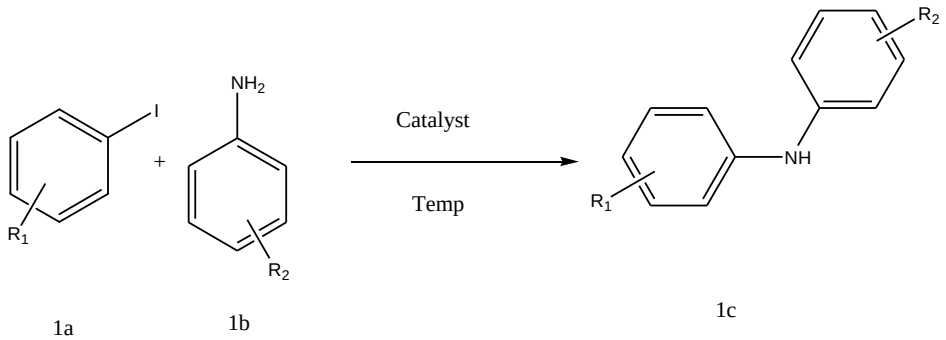
N- Aryl amines and heterocycles are an important class of compounds and are widely used as medicinal[1] as analgesic drugs, biological[2] and NHC chemistry. Some of the compounds have also been reported as antibacterial. N-Aryl heterocycles like imidazole, benzimidazole, benzotriazole, pyrazole and indazole serves as important building blocks in pharmaceuticals. Some of the N-heterocyclic moiety is also found in many bioactive natural products. In addition 1-aryl benzotriazole are useful intermediates in the synthesis of pyridoacridine and carbolin synthesis. Among the heterocyclics, N-aryl imidazole is widely studied because of its distinct biomedical properties like AMPA receptor antagonists. Transition metal mediated C-N bond formations are important fundamental transformations and are commonly used for N-arylation reactions by metals such as Pd, Cu/CuI, and Fe. Mostly C-N bond formed by copper mediated (Ullmann reaction) and Palladium catalyzed (Buchwald-Hartwig reaction) has received much attention. Recently, iron catalyzed N-arylation of N-nucleophiles with various copper-chelating ligands such as 1,10-phenoanthroline and vicinal diamines. N-arylation with excess of bases (K_2CO_3, K_3PO_4, KOH) in polar solvents (DMF, DMSO) was used in various literatures.

KEYWORDS: N- Aryl amines, Heterocyclic Compounds, Pyridoacridine, C-N bond formations, Lewis acid.

INTRODUCTION

Formation of (aromatic) C-N sigma bond plays important role in organic synthesis. The aniline moiety appears as subunits in a broad range of biologically active and medicinally significant molecules. Thus, the synthesis of these compounds has been of longstanding interest. In recent years, several versatile and widely employed copper-catalysed cross coupling methods of amines with aryl halides have been developed to construct aryl C-N bonds.[2a-d]

Diphenylamine 1c is an organic compound with the formula $(C_6H_5)_2NH$. The compound is a derivative of aniline1b,[3] with aryl halides 1a. Diphenylamine is consisting of an amine bound (C-N) to two phenyl groups as shown in Scheme-1. The compound is a colourless solid, but commercial samples are often yellow due to oxidized impurities.[4] Diphenylamine dissolves well in many common organic solvents, and is moderately soluble in water.[5] It is used mainly for its antioxidant properties. Diphenylamine is used as a pre- or post harvest scald inhibitor for apples applied as an indoor drench treatment. Its anti-scald activity is the result of its antioxidant properties, which protect the apple skin from the oxidation products of alpha-farnesene during storage,[6] physical injury that manifests in brown spots after fruit is removed from cold storage. Alkylated diphenylamines function as antioxidants in lubricants, approved for use in machines, in which contact with food is not ruled out.[7]



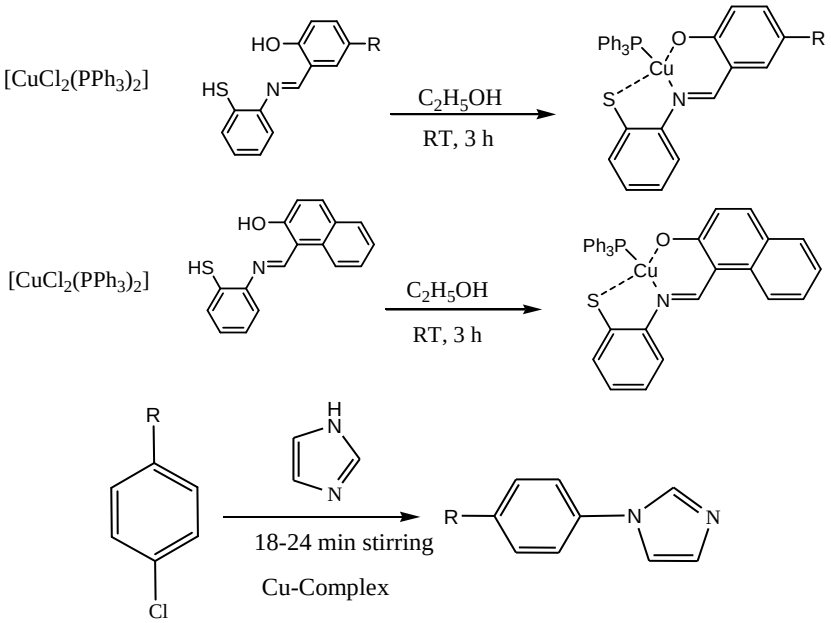
1a 1b 1c

Scheme 1: General Scheme for N-arylation.

Aromatic amines are organic nitrogen containing compounds 1b that may be considered derivatives of ammonia (NH_3) with at least one of the hydrogen atoms replaced by an aryl group. The nitrogen must be attached directly to the aromatic ring and so be able to interact with the aromatic π-electron system. The amine can be primary, secondary or tertiary depending on whether one, two or three of the protons are replaced by alkyl or aryl groups. The simplest aromatic amine is derived from benzene and is called aniline or benzenamine ($C_6H_5NH_2$). The amines from toluene are referred to as toluidines and from naphthalene as naphthylamines. Pyridine, pyrrole, and others in which nitrogen forms part of the ring are treated not as aromatic amines but as heterocyclic compounds. Aromatic amines most often are named by adding the suffix ‘‘amine’’ to the name of the radical (or radicals) replacing one or more of the hydrogen atoms in ammonia, except when some trivial name exists. According to IUPAC nomenclature, the amino ($-NH_2$) or modified amino group (NHR, NRR^1) is considered a substituting group of the aromatic hydrocarbon. Therefore, these compounds are named as derivatives of benzene, toluene, naphthalene and others, e.g., N,N-dimethylaminobenzene $C_6H_5N(CH_3)_2$. Aniline is generally used as the parent structure for its derivatives unless a carbon atom is attached to the benzene ring or unless a function of higher seniority than amino is present in the molecule. Important derivatives of aniline are named as such, e.g., N-methylaniline, p-nitroaniline, chloroaniline, whereas the sulfonic acids are named as derivatives of benzene (aminobenzenesulfonic acid) or by their trivial names (metanilic acid for m-aminobenzenesulfonic acid). If two of the hydrogen atoms of ammonia are replaced by aryl groups, the compounds are generally described as diarylamines, with diphenylamine (DPA) as the parent compound. Aromatic amines with two amino groups on the same benzene ring are named phenylenediamines. Chemical Abstracts called these compounds benzenediamines in 1972. The general reaction is shown in Scheme-1 where catalysts are used for increase rate of reaction. However, there are several novel catalyst are reported which enhance the yields and modern concept of green chemistry.

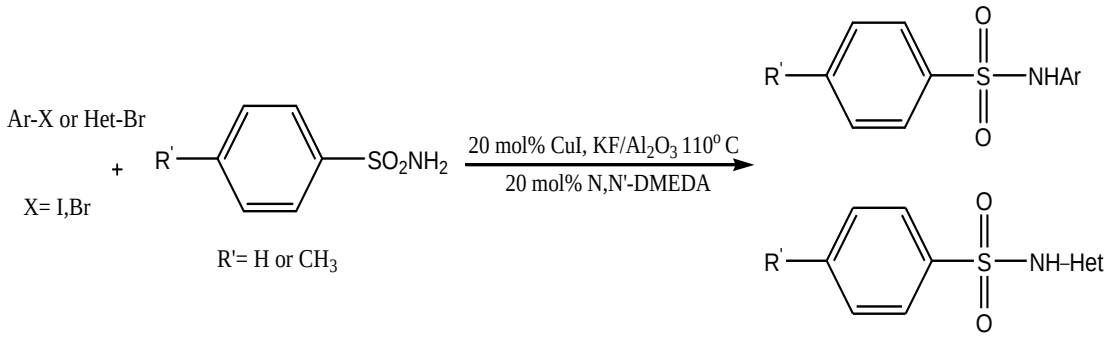
Copper (Cu) Catalysed N-arylation Reactions

Copper-mediated arylation of aromatic substituted amines, originally diarylamines, has history of 100 years as the traditional Ullmann reaction.[8] Cu is a transition metal and has good conducting property and is capable to form complexes with ligands with variety of combinations. The Cu(II) complexes of Schiff’s bases derived from the condensation of o- aminothiophenol with 2-hydroxy-1-napthaldehyde or salicylaldehyde or its derivatives (Br, Cl, NO_2, or CH_3) have been reported by S. Priyarega and others.[9] The Cu(II) complex have been utilized as N-Arylation of Imidazole as shown in Scheme-2.



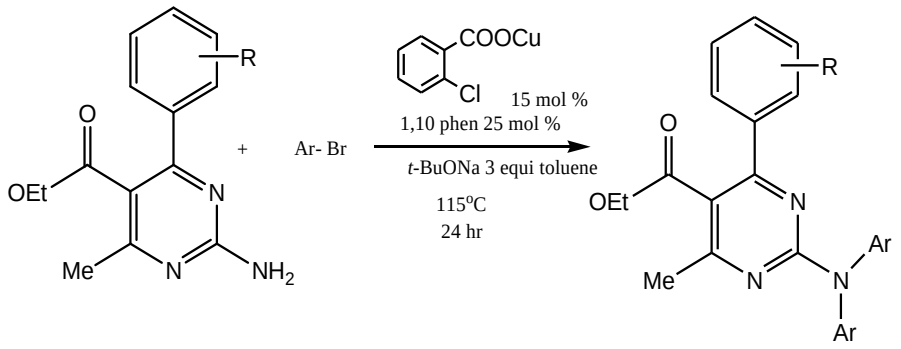
Scheme 2: Catalytic N-Arylation of Imidazole by Cu-(II) Complex.

KF/Al_2O_3 as a base in the presence of CuI was used recently by chemists for C–N bond formation in arylation of amide, alcohols, phenols and diazoles.[10-11] Rahman Hosseinzadeh and its co-workers[12] in year 2010 developed a simple and efficient method for the copper-catalysed coupling of a variety of aryl iodides, aryl bromides, aryl chlorides and heteroaryl bromides with benzene and p-toluene sulphonamides using KF/Al_2O_3, CuI and N,N′-DMEDA as shown in Scheme-3a. Potassium fluoride supported on alumina (KF/Al_2O_3) provides a possible choice to bases such as Cs_2CO_3.[13]



Scheme 3a: N-Arylation of arylsulfonamides catalysed by CuI.

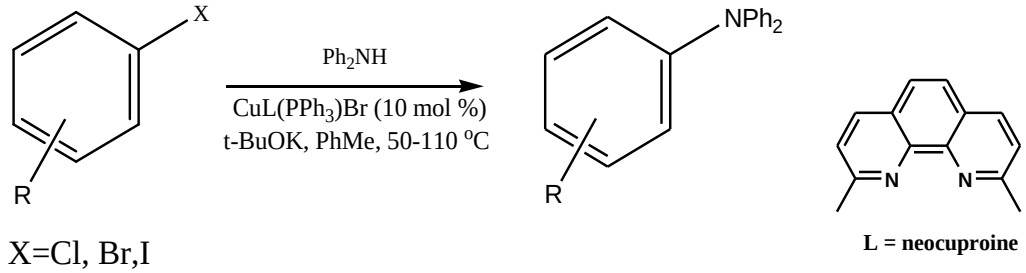
Yue Zhang and it's co-workers developed a novel move towards of selective arylation of 2-aminopyrimidines which leads in secondary and tertiary amines with pyrimidine scaffold oriented by different bases, explains the value of cuprous catalyst, (2- chlorobenzoyloxy) copper(I),[14] which was introduced has perform excellent role as catalyst which is shown in Scheme-3b.



Scheme 3b: Arylation of 2-aminopyrimidines.

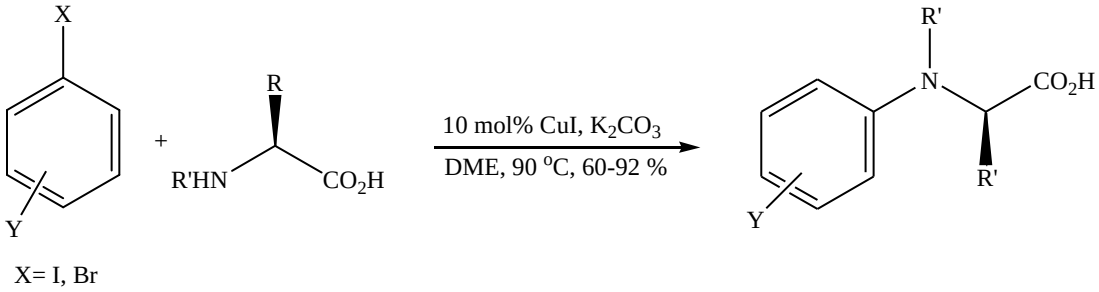
In the year 2005, cross-coupling takes place mainly using Pd catalysis, and utilized in C-C and C-heteroatom bond formation methods which was also reviewed by Irina P. Beletskaya and Andrei V. Cheprakov.[15a] They studied Cross-coupling found a generic term which denote a σ–bond metathesis reaction between a nucleophilic and electrophilic reagent[15b] and thus can be regarded as a simplification of nucleophilic replacement. The equations give idea that some reactions can take place only in catalyst.

R.K. Gujadhur studied the activity of such complexes, which allowing use not only for aryl iodides, but also for aryl bromides to carry out arylation of diphenylamine in the presence of t-BuOK as a base. Moreover, chlorobenzene as arylating agent, in which yield was up to 50%. The t-BuONa and Cs_2CO_3 are less effective, while weaker bases are altogether ineffective[16] as shown in Scheme-4.



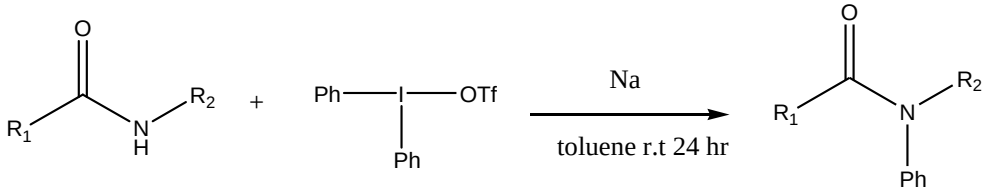
Scheme 4: N- Arylation of diphenylamine.

Qian Cai and co-worker's demonstrated N,N- or N,O-bidentate compounds as the powerful reaction promoters of Ullmann-type coupling reactions and led to N-arylation reaction. The α-amino acid in Cu-catalyzed Ullmann reaction, leading to achievement of the coupling reactions of aryl halides and α-amino acids at 80–90 °C as shown in Scheme-5 .



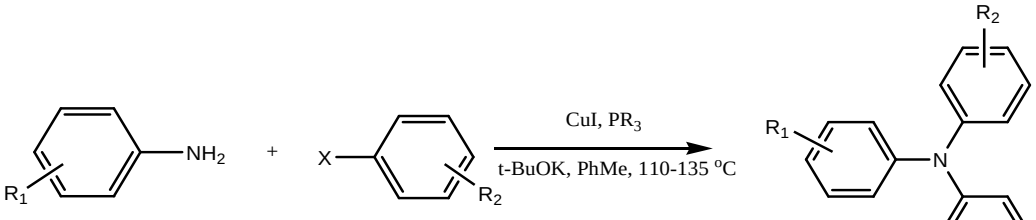
Scheme 5: CuI-catalyzed coupling of aryl halides with α-amino acids.

Fredrik Tinnis gave a high reactivity of diaryliodonium salts which often requires copper catalysis utilized in metal-free arylations of some amides and amide derivatives yielding tertiary acyclic amides at ambient temperature[17a] as shown in Scheme-6. Same work was done by Modha, S. G., & Greaney arylation using diaryliodonium salts which generates one equivalent of an iodoarene as a side-product. Here, they conclude the result which shows that diaryliodoniums salts can undergo Cu-catalyzed tandem C−H/N−H arylation.[17b]



Scheme 6: N-arylation using diaryliodonium salts.

Some monodentate phosphines, such as trio-tolylphosphine P(o-tol)$_3$ and tri- n-butylphosphine PBu$_3$ are highly effective ligands for the arylation of anilines by aryl halides such as iodides or chlorides, in sharp contrast to PPh$_3$, which is practically ineffective even for aryl iodides as shown in below Scheme-7.



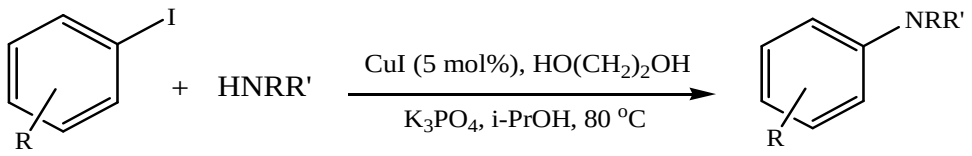
R_2,R_1 = H, o-Me, p-Me, NO_2
X = Cl, Br, I
PR_3 = PBu_3 (X= Cl), P(o-Tol)$_3$ (X= Br, I)

R_2,R_1 = H, o-Me, p-Me, NO_2
X = Cl, Br, I
PR_3 = PBu_3 (X= Cl), P(o-Tol)$_3$ (X= Br, I)

Scheme 7: Copper-catalyzed protocol using PBu$_3$ as ligand.

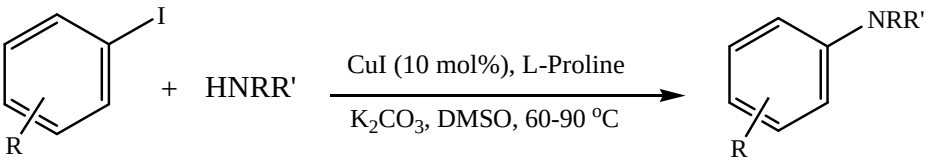
In the above Scheme-7 N.M. Patil and its Co-workers studied the copper-catalyzed protocol PBu$_3$ used as ligand of choice for the most challenging aryl chloride substrates.[17c]

Enguehard in 2003 discovered arylation of aliphatic amines which is achieved in hydroxylic solvent in the presence of K_3PO_4.[18] CuI is the best copper source, though other Cu(I) and Cu(II) compounds are reactive. The chelating effect of glycols is likely to be a critical factor, as both monoatomic alcohols, and remote diols are ineffective as shown in Scheme-8.



Scheme 8: Arylation of aliphatic amines.

Some synthesis carried out by Ma and Zhang conclude that about self-arylation of ligand such as aminoacids with secondary amino-group should be selected, e.g. N-methylglycine or proline. While N,N-dimethylaminoacids are not so much effective as ligands[19] as shown in Scheme-9.

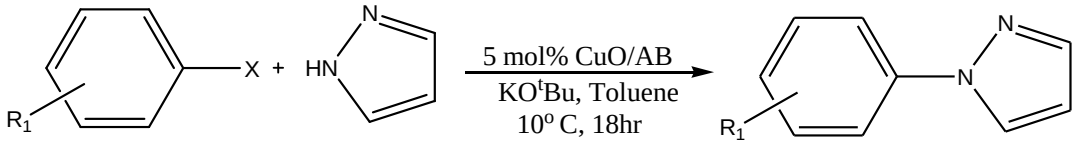


R' = H, R = alkyl, allyl, benzyl,etc
or HNRR' = morpholine, piperidine

Scheme 9: CuI-catalyzed coupling in presence of L-proline.

The coupling of aryl halides with primary amines, cyclic secondary amines, and N-containing heterocycles proceeded at 60–90 °C to give the corresponding amines and N-aryl heterocycles in good yields.[20]

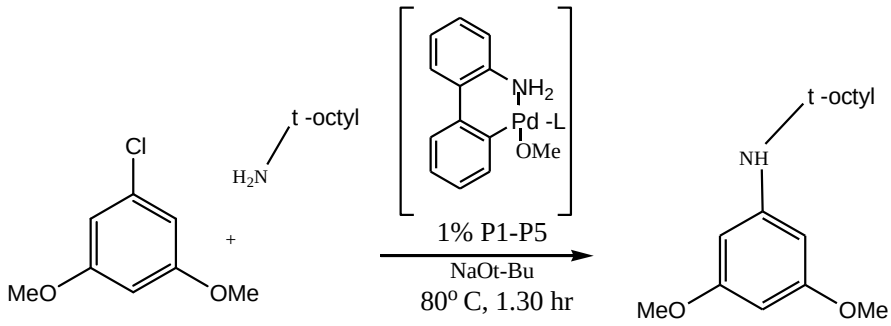
A variety of nitrogen-containing heterocycles on N-arylation catalyzed by CuO/AB with aryl halides studied by a Young Kim et. al. Scheme-10.



Scheme 10: CuO/AB catalyzed N-arylation of various N- heterocycles with aryl halides.

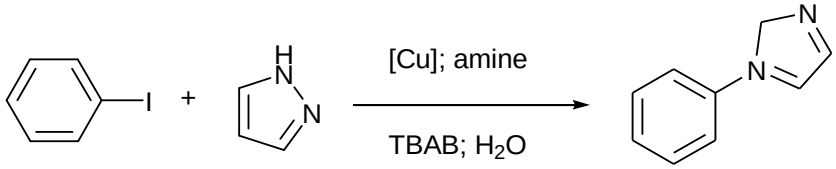
The CuO hollow nanospheres were synthesized by a controlled oxidation reaction of Cu_2O nanocubes. Typically, Cu_2O nanocubes were prepared by a polyol process in 1,5-pentanediol in the presence of poly(vinyl pyrrolidone) (PVP, Aldrich, Mw = 55,000).[21a]

Paula Ruiz-Castillo, Donna G Blackmond, and Stephen L. Buchwald have developed a general method for the cross-coupling of hindered primary amines with a range of (hetero)aryl chlorides and bromides. The coupling reaction was done on of 3,5-dimethoxychlorobenzene with t-octylamine[21b] as shown in Scheme-11.



Scheme 11: Coupling reaction of 3,5-dimethoxychlorobenzene with t-octylamine.

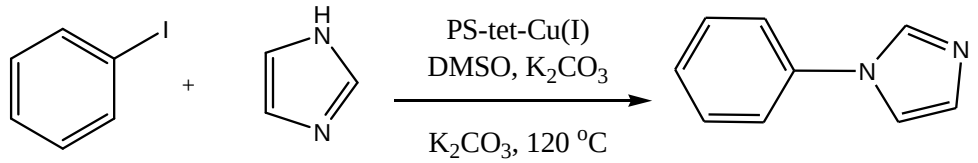
N-Arylheterocycles are ordinary and significant motifs in pharmaceutical and material fields,[22-23] thus many efforts have been devoted to the development in the formation of C-N bond catalyzed by transition metals by now. Among these various progressive strategies of direct N-arylation of heterocycles with aromatic halogen compounds, Pd-catalyst systems possess unavoidable drawbacks of high cost and toxicity,[24] so the modified copper-catalyzed Ullmann reaction comprise an smart alternative as shown in Scheme-12.



Scheme 12: Reaction for N-arylation of pyrazole with iodobenzene.

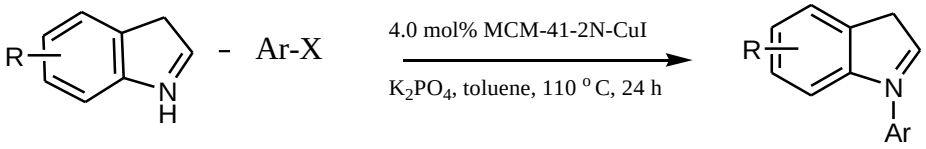
Tetraethylenepentamine (TEPA) and triethylenetetramine (TETA) are found to be efficient organic bases for the N arylation of pyrazole and imidazole with aryl and heteroaryl-iodides and -bromides catalyzed by CuI in water at moderate temperature. The cross-couplings proceed smoothly with good to excellent yields and a variety of functional groups are tolerate under this condition.

Mahmoud Nasrollahzadeh, Ali Zahraei and Eslam Pourbasheer gave novel application of copper complex supported nanopolymer and the ligand-free C–N coupling.[25a] The reactions between amines and N-containing heterocycles with aryl halides as given in Scheme-13a.



Scheme 13a: Application of copper complex supported nanopolymer.

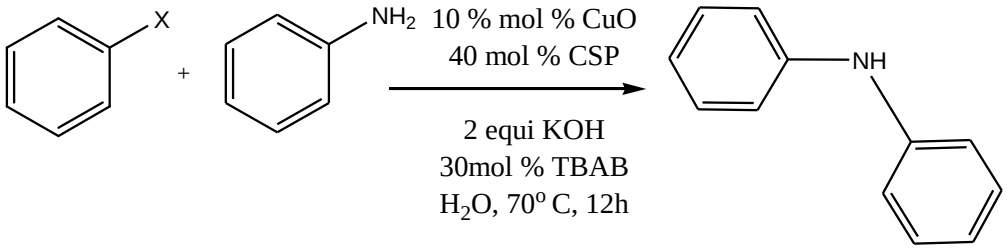
The N-aryl nitrogen heterocycle motif is present in numerous natural products and biologically active pharmaceutical products.[25b] Various strategies have been also developed for the N-arylation of heterocycles.



Scheme 13b: N-arylation of heterocycles.

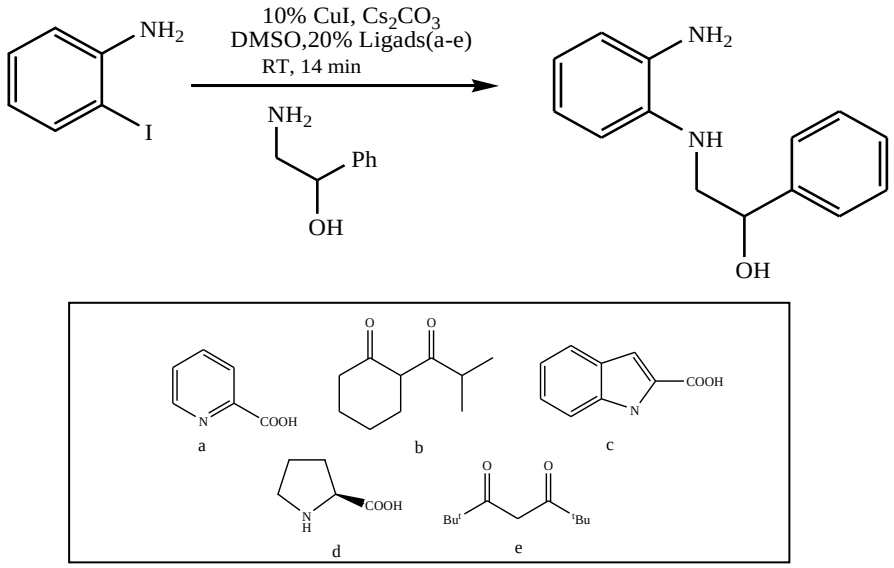
The heterogeneous N-arylation reaction of indoles with aryl halides was achieved in toluene at 110^0C by using 4 mol % MCM-41-immobilized bidentate nitrogen copper(I) complex [MCM-41-2N-CuI] as catalyst and K_3PO_4 as base, yielding a variety of N-arylindoles.[26a] Ruian Xiao have developed a novel, practical, and environmentally affable method for the synthesis of N-arylindoles through the reaction of indoles with aryl halides by using an MCM- 41-immobilized bidentate nitrogen copper complex as catalyst under mild reaction conditions as shown in above Scheme-13b.

Bo Yang, Zuxing Mao, Xinhai Zhu, and Yiqian Wan has developed functionalised Chitosan which was synthesised and combined with CuO to gave a green and recyclable catalytic properties and they have also done study of eco-friendly nature of heterogeneous catalytic system leading to the Ullmann C-N coupling reaction[26b] as shown in Scheme-14.



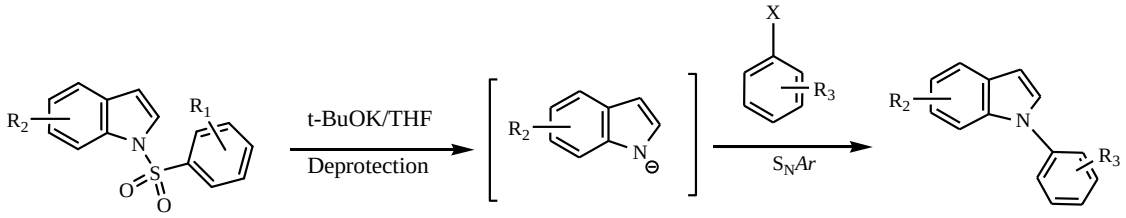
Scheme 14: CuO/CSP-catalysed coupling reactions of aryl halides with aniline.

Priyabrata Das and Jef K. De Brabander has developed copper catalyzed N-selective arylation of β-amino alcohols with 2,2,6,6- tetramethylheptane-3,5-dione or indole-2-carboxylic acid as supporting ligands arylation.[27] The synthesis of N-(2-aminophenyl)-2-hydroxyethylamines via a copper catalyzed N-selective arylation of β-amino alcohols with iodoanilines[28] as shown in Scheme-15.



Scheme 15: The Cu-catalyzed arylation of amino alcohol with iodoaniline.

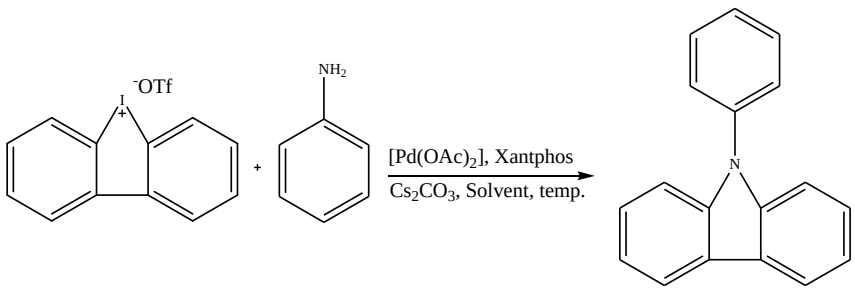
Hui XU and Ling-Ling Fan have developed t-BuOK mediated method for the synthesis of N-arylindoles in moderate to good yields. The protocol involves the consecutive deprotection of N-arylsulfonylindoles as latent indoles and subsequent S_NAr reactions with activated aryl halides[29] as shown in Scheme-16.



Scheme 16: t-BuOK mediated construction of N- arylindoles.

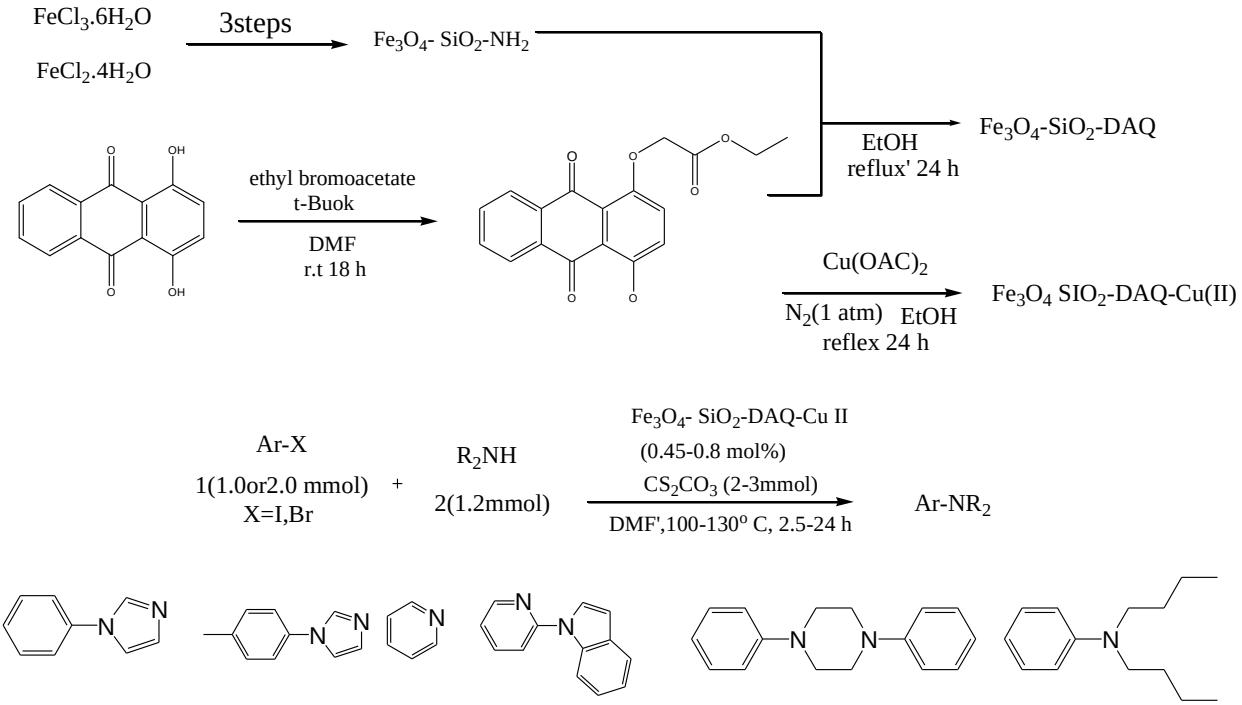
A series of new analogues of trifluralin (TFL)[30] were synthesized and characterized in view of changing the unfavorable properties that limits its use as antileishmanial agent. Twelve TFL analogues were synthesized and tested for their antileishmanial activity and their cytotoxicity to human cells as compared with the parent compound.

Stefan Riedmüller and Boris J. Nachtsheim gave method for direct synthesis of N-arylated carbazoles through a palladium-catalyzed amination of cyclic iodonium salts with aromatic amines. In scrupulous N-arylcarbazoles have promising electroluminescent properties and have consequently found various applications as hole-transport or as host or luminescent-materials in electronic devices. They reported an alternative Pd-catalyzed method for the construction of N-substituted carbazoles based on a stable, cyclic iodonium salt and electron-deficient anilines[31] Scheme -17.



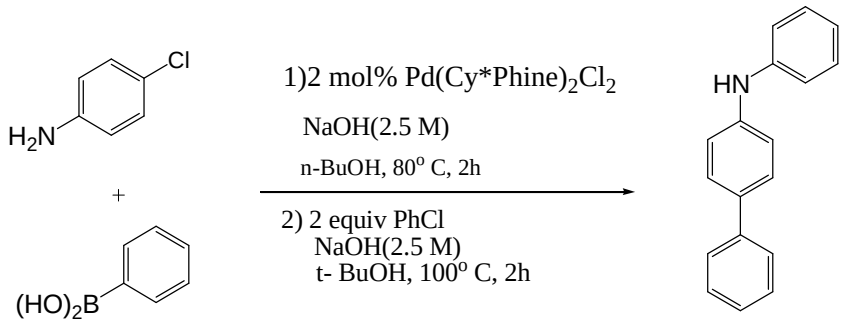
Scheme 17: Synthesis of N- arylcabazole using Pd salts.

S. Zahmatkesh gave method for synthesis of 1,4-Dihydroxyanthraquinone–Copper(II) Supported on Superparamagnetic Fe_3O_4-SiO_2 an efficient Catalyst for N-Arylation of Nitrogen heterocycles and alkylamines with aryl halides and Click Synthesis of 1-Aryl-1,2,3-triazole derivatives[32] as Scheme-18.

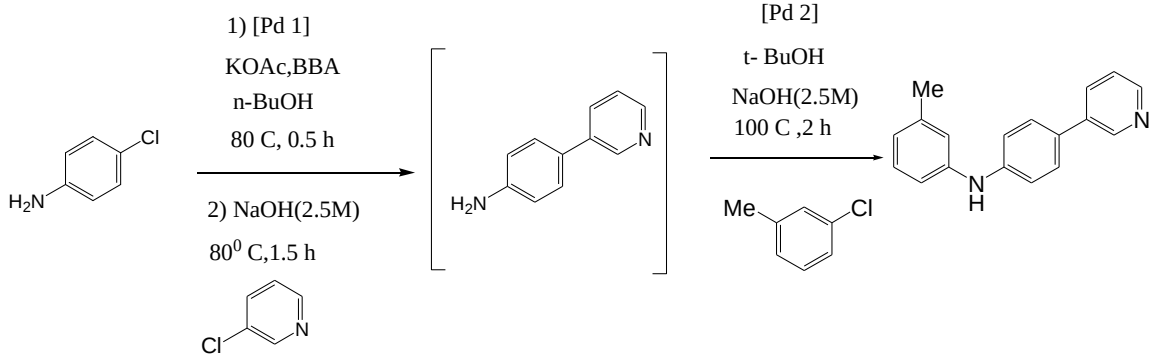


Scheme 18: Synthesis of 1,4-Dihydroxyanthraquinone–Copper(II).

Howard Jong gave methodology for a sequential palladium- catalyzed cross-coupling procedure consisting of borylation, the Suzuki reaction. Amination has been developed for the assembly of molecules with multi-aryl backbones. The linchpin of this development is the meta-terarylphosphine ligand, Cy*- Phine, which has been engaged as an air- and moisture-stable precatalyst, $Pd(Cy*Phine)_2Cl_2$, to improve the efficiency of one-pot borylation– Suzuki reactions[33] as shown in the below Scheme-19a and Scheme-19b.

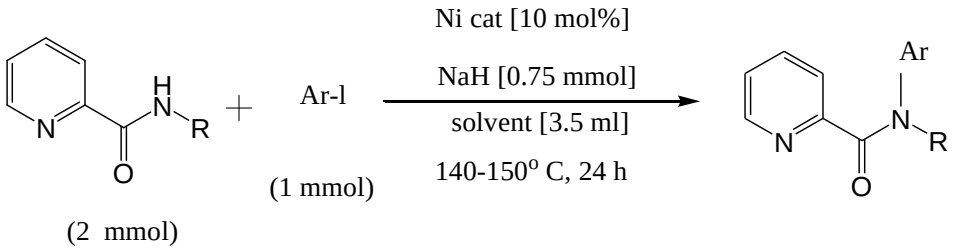


Scheme 19a: Suzuki- sequential amination coupling.



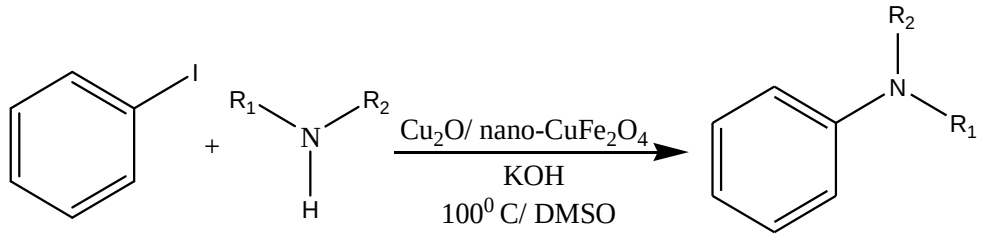
Scheme 19b: One-pot borylation– Suzuki reactions.

Rathinam Sankar gave Ni (II)-catalyzed, C-N cross coupling reaction of secondary acyclic amides (2-picolinamides, which were derived from 2-picolinic acid and amines) and aryl halides and construction of tertiary amides are reported.



Scheme 20: Ni (II)-catalyzed, C-N cross coupling reaction.

Firouzeh Nemati and Ali Elhampour have developed an effective method for the N- arylation of aliphatic amines and azoles with aryl halide using Cu_2O/ nano-$CuFe_3O_4$ magnetic composite as the catalyst and KOH as the base. This methodology found to be applicable to wide range of N- containing heterocycles as well as aliphatic amines in good to moderate yield[34] Scheme-21.



R_1,R_2= H, Alkyl,Aryl

Scheme 21: N-arylation using Cu_2O/nano-Cu-Fe_2O_4 catalyst.

Firouzeh Nemati and Ali Elhampour also investgated nano-magnetic Fe_3O_4-TiO_2/Cu_2O composite and KOH as the base for arylation of aromatic amines. This procedure does not require the use of expensive ligands. Notably, the catalyst is easily recoverable and reused by magnetic separation up to five times without appreciable loss of its catalytic activity.[35]

CONCLUSION

In summary, we enlist the synthesis, applications and its recent development. Among the catalyst system Copper and Lewis Acids has become most popular catalyst among all the N- arylation synthesis. In last five year's literature and publications showed its fast growing importance towards organic synthesis due to its ability to perform a variety of transformations. It is also complimented by their readily availability, nature friendly, stability, tolerance to moisture and water, easy handling. N-arylation has ability to form various derivatives and shows versatile application in heterocyclic and in asymmetric synthesis.

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