Chemistry & Biology Interface, 2018, 8, 4, 194-204

REVIEW PAPER ISSN: 2249 –4820

CHEMISTRY & BIOLOGY INTERFACE

An official Journal of ISCB, Journal homepage; www.cbijournal.com

Recent advances in synthesis of sulfonamides: A review

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Received 23 April 2018; Accepted 29 July 2018

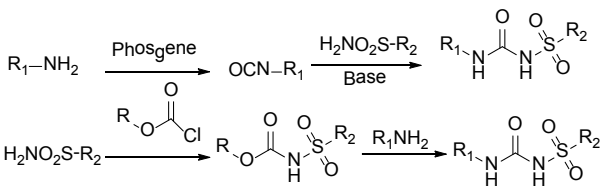
Abstract: Sulfonamides belong to an important class of compounds which show wide ranges of biological activities. Over the last few decades various pharmacological activities of sulfonamide conjugates were published. Moreover, Currently many lead compounds with sulfonamide functionality are ~~also~~ in clinical trial for the treatment of various medical conditions. For these reasons, development of an efficient process for the synthesis of sulfonamides has always been in the focus for research in organic synthesis. Researchers published numerous articles over the years for demonstrating effectiveness of sulfonylation and *N*-alkylation procedures for the synthesis of sulfonamide. The most typical method for the synthesis involves reaction between primary or secondary amines and sulfonyl chloride in presence of organic or inorganic bases. Although this method is effective, but the nucleophilicity of amines may vary depending on the groups attached to it. In general, primary amines are highly reactive, whereas secondary amines show very low to almost nil reactivity. In this study, we have reviewed recent advances related to the efficient synthetic procedures for different types of sulfonamides.

Keywords: Synthesis, review, sulfonamide, thiol, primary amine, secondary amine, heteroaryl amine, sulfonic acid, metal catalyst.

Introduction

Drugs containing sulfonamides of primary amines

Sulfonamides are used as a core structural moiety or an important fragment in many marketed drugs. Several antimicrobial drugs were prepared mainly by coupling between heterocyclic primary amines and aromatic sulfonyl chlorides e.g. sulfacetamide, sulfadiazine, sulfamethoxazole, sulfamoxole etc. Another important use of sulfonamide was noticed in synthesis of sulfonylureas (**scheme 1**), which were evolved as good anti-diabetic agents. Few important drugs in this series are glipizide, acetohexamide, carbutamide.



Scheme 1: Synthesis of sulfonyl urea

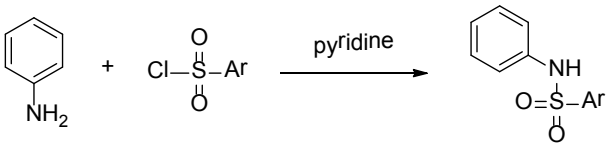
Sultiame, also known as sulthiame is a sulfonamide which was prepared from simple starting material p-aminobenzenesulfonamide. It is an inhibitor of enzyme carbonic anhydrase and currently in use as an anticonvulsant. Dorzolamide, acetozolamide and brinzolamide are a few other important series of drugs where primary sulfonamide side chains are attached to heterocyclic ring. These are used to treat glaucoma or ocular hypertension. Zonisamide has been evolved as a vital drug where methanesulfonamide group is attached with benzoisoxazole moiety. The drug has approved in many countries for various critical diseases. In US and UK zonisamide has received approval for adjunctive treatment of partial seizures. In Japan, the same molecule has marketed for the treatment of Parkinson's disease. In recent years, it is also being investigated for other diseases like migraine, obesity and bipolar depression.

Drugs containing sulfonamides of secondary amines

Several essential drugs containing sulfonamide moiety have also been marketed where coupling reaction between sulfonyl chloride and secondary amine was considered as the key synthetic step. Two important drugs in this category are amprenavir and darunavir. Amprenavir (original brand name agenerase) launched by Glaxo-Smithkline acts as a protease inhibitor. It is already in use to treat HIV infection. Darunavir is another drug in this category which is developed by Tibotec, sold under the brand name prezista. It is being used for antiretroviral medication to treat and prevent HIV/AIDS patient. Secondary sulfonamide side chain is attached with benzoic acid in the structure of probenecid which is used in treating gout and hyperuricemia. Various novel and efficient strategies for the synthesis of sulfonamides containing primary and secondary amine groups have been identified and discussed in this review.

Synthesis of sulfonamide using primary amines and aryl sulfonyl chloride:

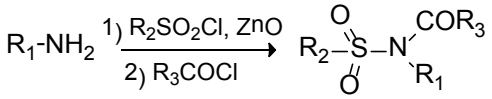
Youn *et al.* [1] reported preparation of sulfonamide using aryl primary amine and aryl sulfonyl chloride (**scheme 2**) employing pyridine as a base at 0-25 °C. They have observed 100% yield when aniline is used as a primary amine and benzene sulfonyl chloride or 4-nitrobenzyl sulfonyl chloride as sulfonylation agent. Quantitative yield also reported for reaction between p-toluidine and tosylchloride. The aim of the study was regioselective synthesis of 3-Arylindoles from *N*-Ts-Anilines and styrenes.



Scheme 2: Synthesis of sulfonamide using primary amine and sulfonyl chloride

Rattanburi *et al.* [2] reported Fe_3O_4-DIPA catalyzed sulfonamide preparation with excellent yield (98%) where the reactant and Fe_3O_4-DIPA in dichloromethane (DCM) were shaken at room temperature (RT) for reaction completion. Catalyst was separated by magnetic separation and reused. Chemoselective solvent free synthesis of sulfonamide (**scheme 3**) using zinc oxide-nanoparticle was reported by Tamaddon *et al.* [3] with 95% yield. Synthesis started with primary amines, sulfonylation followed by acylation produced N-acylsulfonamides. Reusability of environmental-friendly catalyst

was the main advantage of the protocol. Tamaddon *et al.* [4] also reported invention of highly efficient catalyst CsF-Celite for sulfonylation reaction. Chemoselective solvent free neat reaction has been demonstrated for the preparation of various sulfonamides with high yield.



Scheme 3: Solvent free synthesis of sulfonamide using ZnO-nanoparticle

Over the decades researchers reported use of neat pyridine or its combination with polar solvents for the preparation of sulfonamides of primary amines *e.g.,* Raju *et al.* [5] filed a patent on ramoplanin derivatives having antibacterial activity in 2006 where they have reported synthesis of N-phenylbenzenesulfonamide with 90% yield. Neat pyridine was added to amine substrate at 0 °C and reaction was carried out at room temperature (RT) after addition of sulfonyl chloride. Kato *et al.* [6] reported synthesis of achiral aromatic sulfonamides which are further undergone spontaneous rapid resolution to produce chiral crystal. Polymer supported pyridine was used as a base and dichloromethane as a solvent to afford 92% yield. Preparations of mono-sulfonamide and bis-sulfonamide were reported by Alba *et al.* [7] with 90% yield where sulfonamide prepared using pyridine as a base in THF-solvent. The synthesized sulfonamides were used as an organo-catalyst i.e. hydrogen-bond donors in organo-catalyzed ROP of lactones.

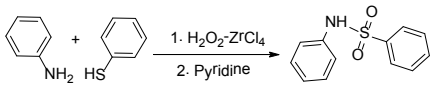
Moderate to good yield was reported in several articles for the sulfonylation of primary amines using another commercially abundant organic base triethylamine (TEA). Kurkin *et al.* [8] reported 86% yield using TEA as a base in THF solvent. Synthesis procedure was simple, TEA was added dropwise to solution of aniline in THF, the mixture was stirred in ice bath. Benzenesulfonyl chloride was added dropwise and stirred at RT for 6 h for reaction completion. Conway *et al.* [9] reported 85% yield of N-phenylbenzenesulfonamide by reaction between aniline and benzene sulfonyl chloride in diethyl ether at 0 °C. Qui *et al.* [10] reported reaction between diamino aryne precursor and sulfonamide where sulfonamides were prepared with 85% yield using TEA as a base in DCM.

Use of inorganic base like potassium carbonate was also explored in sulfonylation reaction by Pranab *et al.* [11] where the researchers achieved up to 78% yield when reactions were carried out in PEG-400 solvent. The developed protocol was convenient due to heterogeneous reaction mass where base can be easily separated from reaction mass by filtration. Recovery and reuse of PEG-400 is another advantage of the reported method from economical and environmental aspect. Rebecca and coworkers [12] reported poor yield (only 44%) when strong inorganic base like sodium hydroxide was employed for the sulfonamide synthesis reaction. In a magnetic stirrer acid chloride and amine were mixed together. Addition of 10% NaOH was done in portions and the reaction mixture stirred for 1 h at RT to produce *N*-phenylbenzenesulfonamide. The main intension of the study was to prepare unalkylated benzene sulfonanilides to check rearrangement of the same to sulfones. Sulfonamide synthesis by reaction between aqueous solution of primary amine and sulfonyl chloride at RT using sodium carbonate as a base was reported by Soukaina *et al.* [13] The research group prepared series of sulfonamide-4-substituted-1, 2, 3-trizolyl nucleosides and evaluated their activity against tumor cell lines RCC4 and MDA-MB-231.

Synthesis of sulfonamide using amines and thiol:

Another strategy for the synthesis of

sulfonamide of primary amines was explored by the researchers where SH group of aromatic thiol was in-situ oxidized by oxidizing agent and chlorinated before the amide bond formation. Bahrami *et al.* [14] reported stage wise reaction between aniline and benzene thiol (**scheme 4**) using hydrogen peroxide as an oxidizing and zirconium chloride as a chlorinating agent. In stage-2, pyridine used as a base and overall 98% yield was achieved. They have claimed environmentally green and economical protocol where the novel H_2O_2-$ZrCl_4$ reagent system offered fast reaction at room temperature to furnish excellent yield (92-98%).

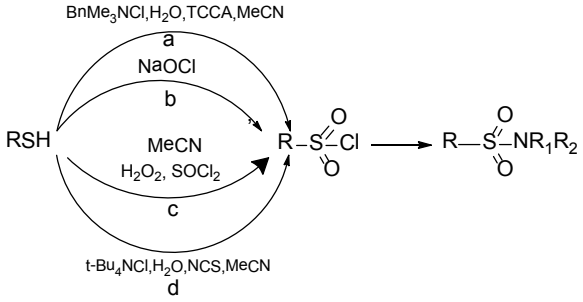


Scheme 4: Synthesis of sulfonamide using primary amine and thiol

The same research group also reported [15] a stage wise process (**scheme 5, path c**) where in stage 1, a combination of hydrogen peroxide and thionyl chloride reagent system were used for direct oxidative chlorination of various thiol derivatives to the corresponding sulfonyl chloride intermediates, which were further converted to sulfonamide in stage 2 by reacting with different amines using pyridine as a base in acetonitrile or water solvent to afford 94-98% overall yield.

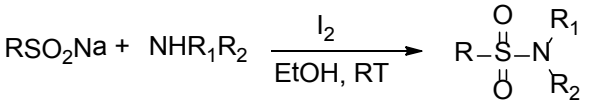
Thiol can be converted to sulfonyl chloride in many ways (**scheme 5**). Apart from the oxidative chlorination of thiol to sulfonyl chloride as reported by Bahrami et al, in-situ conversion of heteroaromatic thiols to sulfonyl choride using sodium hypochlorite as an oxidizing agent (**path b**) was reported by Wright *et al.* [16] to prepare sulfonyl chloride at low temperature which was immediately trapped with benzyl amine to produce sulfonamide in almost quantitative yield. Frank *et al.* [17] prepared organic chloramines by mixing sodium hypochlorite and aq. solution of amine which has reacted rapidly with sodium arenesulfinate to form arene sulfonamide.

Veisi *et al.* [18] developed a novel method where an oxidizing chlorinating system was developed to synthesize sulfonyl chloride from various thiols by mixing N-Chlorosuccinamide and tertbutylammonium chloride-water system in acetonitrile solvent (**path d**). The sulfonyl chloride is in situ converted to the corresponding sulfonamide in one pot. Bonk *et al.* [19] invented novel system for controlled liberation of chlorine in combination of trichloroisocyanuric acid and benzyltrimethylammonium chloride in MeCN solvent (**path a**). The sulfonyl chloride was generated and in-situ reacted with various amines to diversify the scope of the protocol.



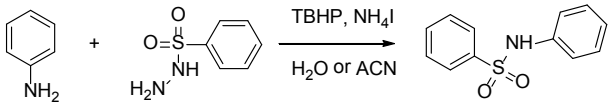
Scheme 5: Conversion of thiol to sulfonyl chloride using various reagents

Concept of green chemistry was applied for the first time in sulfonylation reaction where stage wise reaction using iodine and ethanol was investigated by Yang *et al.* [20] The reported procedure was novel and efficient since any metal, base, ligand or additive was not used in the reaction. The scope of the protocol was also very wide which had covered construction of variety of primary, secondary and tertiary sulfonamides.



Scheme 6: Metal free sulfonamide synthesis using sodium sulfinate and amines

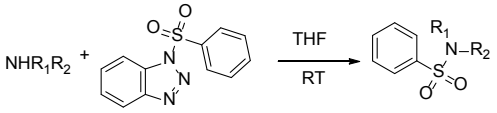
In a recently published article, [21] use of benzene sulfonyl hydrazide (**scheme 7**) was mentioned for the first time by Yu *et al.* for sulfonylation of various amines in presence of tert-butylhydroperoxide (TBHP) and ammonium iodide in water or acetonitrile with moderate to good yield. TBHP was used as an oxidant and ammonium iodide (NH_4I) as a catalyst to produce aryl sulfonyl hydrazide which is further reacted with amines to produce corresponding sulfonamides.



Scheme 7: Synthesis of sulfonamide using benzenesulfonyl hydrazide

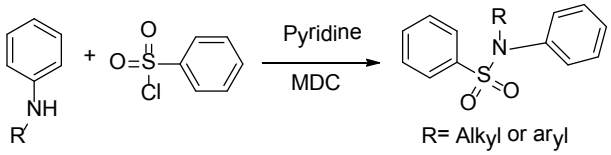
Synthesis of sulfonamide using secondary amines

Katritzky *et al.* [22] invented a novel reagent 1-phenylsulfonylbenzotriazole (**scheme 7** 8) for the conversion of various aliphatic and aromatic amines and phenols to their corresponding benzenesulfonamides and benzenesulfonates.



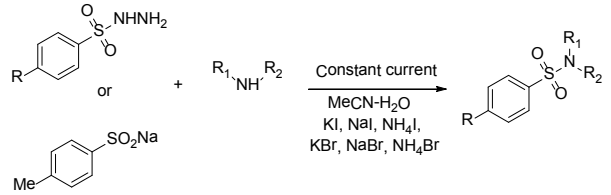
Scheme 8: Synthesis of sulfonamide using 1-phenylsulfonylbenzotriazole

Sulfonamide synthesis using secondary amines like *N*-methyl aniline or diphenyl aniline (**scheme 9**) was reported by Wang *et al.* [23] The aim of the study was to explore the hemolytic cleavage and intermolecular radical-radical coupling reaction mechanism of 1,3- and 1,5-sulfonyl migration of *N*-arenesulfonylphenothiazines and *N*-arenesulfonylphenoxazines. Excellent yield (97%) was achieved using pyridine as a base in chlorinated solvent by portion wise addition of benzenesulfonyl chloride.



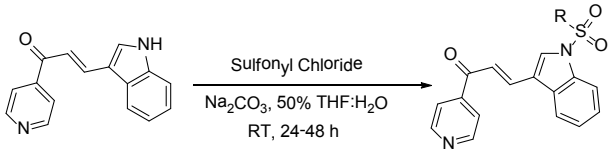
Scheme 9: Synthesis of sulfonamide using secondary amine

In 2016, Terent'ev *et al.* [24] reported an effective electrochemical synthesis of sulfonamides (**scheme 10**) from arenesulfonohydrazides or sodium *p*-methylbenzenesulfinate and amines. The reactions were carried out in a undivided cell using graphite anode and iron cathode at constant current density 35-40 mA cm^{-2} to efficiently produce fourteen various sulfonamides in 56-98%.



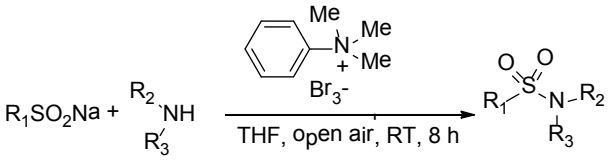
Scheme 10: Synthesis of sulfonamide using arenesulfonohydrazides or sodium *p*-methylbenzenesulfinate and secondary amine

In 2018, Peerzada *et al.* [25] prepared various tertiary sulfonamide derivatives (**scheme 11**) of pyridyl-indole based heteroaryl chalcone in 60-90% yield. Reactions were carried out room temperature (RT) using weak inorganic base Na_2CO_3 in 50% THF:H_2O solvent mixture. All the compounds were evaluated for carbonic anhydrase IX inhibitors and anticancer agents.



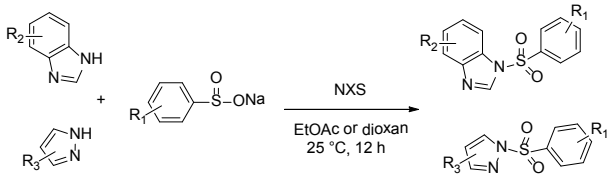
Scheme 11: Synthesis of sulfonamide derivatives of pyridyl-indole based heteroaryl chalcone

An open air, metal free oxidative coupling for the synthesis of sulfonamide (**scheme 12**) mediated by phenyl trimethyl ammonium tribromide (PTAB) has been reported by Sarkar *et al.* [26] Initially benzene sulfinate reacted with PTAB to produce forms the corresponding sulfonyl bromide, then nucleophilic reaction of amines with sulfonyl bromide produced the desired products in 54-82% yield.



Scheme 12: Synthesis of sulfonamide mediated by PTAB

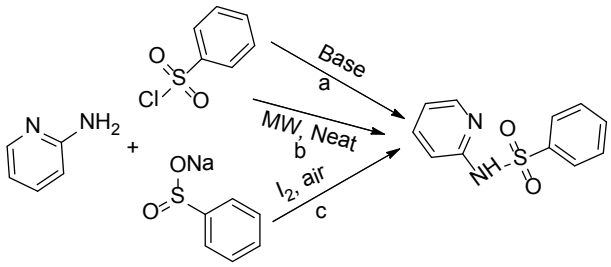
Metal free direct *N*-sulfonylation of azoles or benzimidazoles by sodium sulfinates (**scheme 13**) was reported by Fu *et al.* [27] A diverse range of azoles and pyrazoles were converted to sulfonamides following simple and green procedure. Initially, aromatic sodium sulfinate was reacted with *N*-Iodo or *N*-bromosuccinimide (NIS) to produce sulfonyl bromide or iodide which was finally converted to sulfonamides by nucleophilic attack of azoles or benzimidazoles.



Scheme 13: Synthesis of sulfonamide using substituted azoles or benzimidazoles and sodium sulfinates

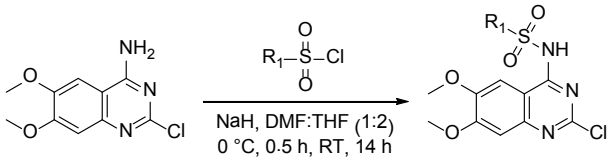
Synthesis of sulfonamide using heteroaryl amine

Reaction between 2-aminopyridine and benzenesulfonyl chloride (**scheme 14, path a**) was reported by Kumar *et al.* [28, 29] using organic base pyridine in 63% yield. Comparatively low yield (29%) obtained when same sort of reactions were carried out applying microwave irradiation (**scheme 14, path b**) by Sharma *et al.* [30] Iodine catalyzed reaction between heteroaryl amine and sodium salt of benzenesulfonate (**scheme 14, path c**) was reported by Wei *et al.* [31] in 52% yield.



Scheme 14: Synthesis of sulfonamide using heteroaryl amine

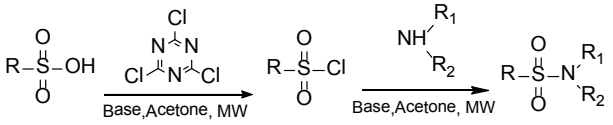
In 2017, Poudapally *et al.* [32] synthesized various quinazoline sulfonamide derivatives (**scheme 15**) and evaluated them as anticancer agent against SKOV3, DU145, THP1, U937 and COLO205 cell lines. Few compounds were found to be active against THP1 and U937 cell lines. Different aryl, heteroaryl, alkyl and cyclopropyl sulfonyl chlorides were reacted with 2-chloro-6,7-dimethoxyquinazolin-4-amine in DMF and THF solvent mixture using sodium hydride as a base to produce the corresponding sulfonamide in 72-96% yield.



Scheme 15: Synthesis of substituted quinazoline sulfonamide derivative

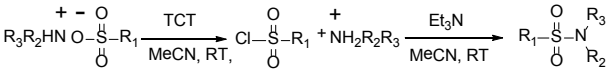
Synthesis of sulfonamide of secondary amines starting from sulfonic acid

Microwave assisted preparation of sulfonamide starting from sulfonic acid was explored by De Luca *et al.* [33] in high yield. The reaction proceeded via sulfonyl chloride intermediate (**Scheme 16**). 2, 4, 6-trichloro-[1, 3, 5]-triazine was added to a solution of p-toluenesulfonic acid in acetone, followed by TEA. The reaction mass was irradiated at 80 °C for 20 min, the sulfonyl chloride is isolated by filtration and further reacted with allylamine to produce corresponding sulfonamide in 95% yield.



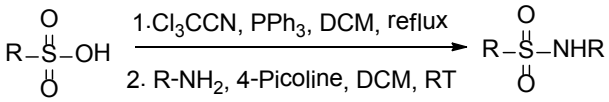
Scheme 16: Microwave assisted preparation of sulfonamide from sulfonic acid

Rad *et al.* [34] reported one pot synthesis of sulfonamide from primary or secondary amine by preparing amine sulfonate salt using cyanuric chloride (**scheme 17**) using classical heating method with excellent yield. The reaction mechanism has also been explained by the research group. The reaction proceeds via S_NAr-type reaction between sulfonate anion and cyanuric chloride. The chloride ion attacks the sulfer atom to produce corresponding sulfonyl chloride. In next step, the amine liberates from ammonium in presence of TEA, which reacts with sulfonyl chloride to afford sulfonamide product.



Scheme 17: Preparation of sulfonamide from amine sulfonate salt using cyanuric chloride

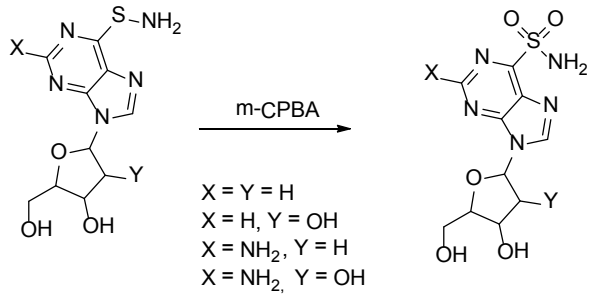
Chantarasriwong *et al.* [35] discovered a novel method for sulfonamide preparation (**scheme 18**) where scope of the study was widened by using aliphatic, aromatic and heterocyclic amines. In first step, sulfonic acid was treated with trichloroacetonitrile, triphenylphosphine in dichloromethane to produce corresponding sulfonyl chloride, which is subsequently transformed to sulfonamide by reacting with amine and base.



Scheme 18: Synthesis of sulfonamide from sulfonic acid using trichloroacetonitrile

Synthesis of sulfonamide by oxidation of sulfenamide

Revankar *et al.* [36] reported amination of 2'-deoxy-6-thioinosine and 9-*β*-D-arabinofuranosyl-6-thiopurine with chloramine to prepare corresponding 6-sulfenamides which on oxidation by excess meta-chloroperbenzoic acid (mCPBA) produced corresponding sulfonamide (**scheme 19**) in 48% yield. mCPBA was used as an oxidizing agent for the oxidation of sulfur to produce the corresponding sulfone.

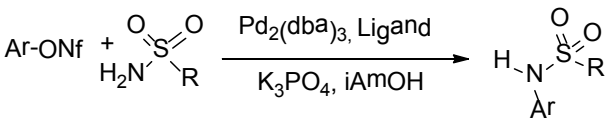


Scheme 19: Synthesis of sulfonamide from sulfenamide

Preparation of secondary and tertiary sulfonamides using metal catalyst

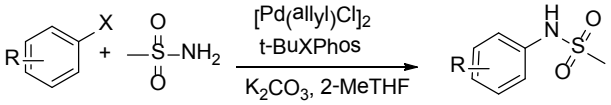
Pd catalyzed sulfonamidation of aryl nonafluorobutanesulfonates (**scheme 20**) have been investigated by Shekhar *et al.* [37] The optimal reaction condition was established for N-arylation where combinations of $Pd_2(dba)_3$

and binary phosphine ligand, t-BuXPhos were used as highly active catalyst and K_3PO_4 in tert-amyl alcohol was found as optimal base-solvent combination for the reaction. Though the instability of di-substituted aryl nonaflates is major limitation, but the wide substrate scope is the advantage of the protocol.



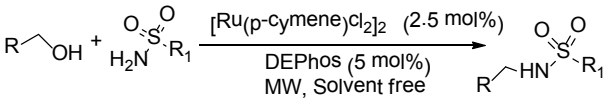
Scheme 20: Pd catalyzed synthesis of sulfonamide

Pd-catalyzed cross coupling reaction between substituted aryl halide and methane sulfonamide (**scheme 21**) was studied by Rosen *et al.* [38] with high yield.



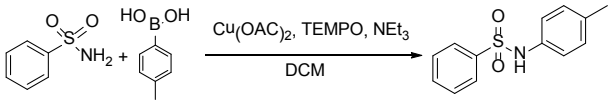
Scheme 21: Sulfonamide synthesis using borrowing Hydrogen catalyst

Solvent free microwave assisted *N*-alkylation of primary sulfonamide with alcohol to produce secondary sulfonamide in very good yield (**scheme 22**) was reported by Watson *et al.* [39] Ru-complex was used as a catalyst in the reaction to borrow the hydrogen from alcohol. The intermediate aldehyde reacted with amine to generate imine which is eventually reduced by the catalyst to produce sulfonamide of secondary amine.



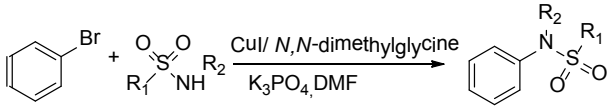
Scheme 22: Synthesis of secondary amine-sulfonamide by hydrogen auto transfer

Cu catalyzed *N*-arylation of benzenesulfonamide using aryl boronic acid (**scheme 23**) was reported by Lam *et al.* [40] in quantitative yield. Diversity of cross-coupling between arylboronic acid with wide range of primary sulfonamide using catalytic copper system is the main advantage of the invented protocol. Catalytic $Cu(OAc)_2$/TEMPO in air and catalytic $Cu(OAc)_2/O_2$ system worked well for majority of the substrate.



Scheme 23: Cu-catalyzed N-arylation of sulfonamide using boronic acid

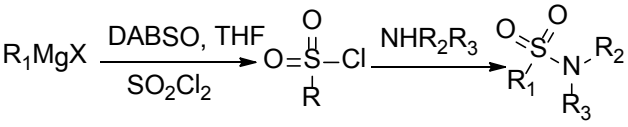
Another example of Cu catalyzed N-arylation of sulfonamide was disclosed by Deng *et al.* [41] where coupling between aryl bromide or iodide and sulfonamide was carried out in excellent yield. DMF used as a solvent, K_3PO_4 and amino acid as a base and ligand (**scheme 24**). Invention of less expensive and more environmentally benign Cu(I)/amino acid catalyst system for C-N cross-coupling reaction is the novelty of the protocol.



Scheme 24: Synthesis of N-arylsulfonamide using CuI

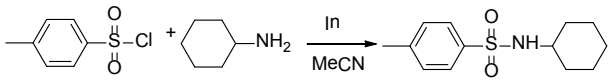
Woolven *et al.* [42] invented a stable complex DABSO, which was prepared by reaction between DABCO and gaseous sulfer dioxide. The complex was used as a safer source of sulfer dioxide which was combined with Grignard reagent to produce sulfinate. Sulfuryl chloride was used to convert sulfinates to sulfonyl chloride which was in-situ reacted with various amines to produce corresponding sulfonamides

(**scheme 25**).



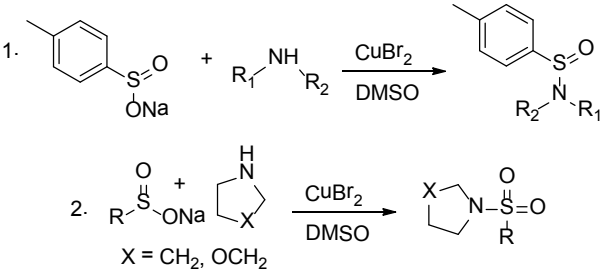
Scheme 25: Synthesis of sulfonamide using DABCO-Bis(sulfer dioxide complex).

Indium catalyzed sulfonylation of sterically hindered and less nucleophilic amines were reported by Kim *et al.* [43] in excellent yield. A wide range of sulfonamides were prepared using the new methodology. The research group claimed reusability of the catalyst up to five times. They proposed that, eletrophilic species RSO2+ InCl- might be generated by reaction between sulfonyl chloride and indium metal, which is further reacted with amines to furnish sulfonylated product and active indium metal (**scheme 26**).



Scheme 26: Synthesis of sulfonamide using catalytic Indium

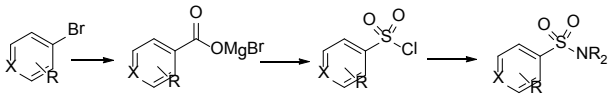
Cu catalyzed oxidative coupling between various amines and sodium sulfonates (**scheme 27**) were investigated by Tang et al. [44] Good yield and excellent chemoselectivity was observed since the transformation happened via single electron transfer pathway.



Scheme 27: Synthesis of sulfonamide via Cu-catalyzed oxidative coupling

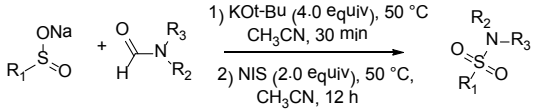
Synthesis of sulfonamide via Grignard

Barret *et al.* [45] discovered innovative method for the preparation of sulfonyl chloride followed by sulfonamide. Aryl and heteroaryl bromides were first converted into the corresponding Grignard reagents using isopropylmagnesium chloride, which were subsequently reacted with sulfuryl chloride and amines to produce sulfonamides (**scheme 28**).



Scheme 28: Synthesis of sulfonamide via Grignard

In 2018, Bao *et al.* reported two step sequential metal free process for the synthesis of sulfonamides using *N,N*-disubstituted formamides as an amine and sodium sulfinates as sulfone source. Initially, formamide was decarbonylated by KOt-Bu to produce the corresponding amine in acetonitrile solvent. At the same time sodium sulfinate reacted with *N*-Iodosuccinamide (NIS) to afford sulfonyl iodide which was eventually generated sulfonyl radical. Finally, nucleophilic attack of amine to sulfonyl radical provided desired sulfonamide in 24-91% yield.



Scheme 29: Synthesis of sulfonamide via Grignard

Conclusion: Sulfonamides not only constitute very important structural components of many approved drugs used in various medical conditions, but also their presence increases biological activity of several important pharmacores by many folds. Hence, over the decades researchers have been putting enormous

effort to prepare sulfonamide conjugates of existing drugs. In this review, we summarized a number of reports about synthetic pathways for primary, secondary and tertiary sulfonamides mostly starting from amines, sulfonic acids, thiols and alcohols**.**

Acknowledgment:

We acknowledge the management of Dr. Rafiq Zakaria College for women Aurangabad.

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