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A Review on Synthesis of Azoles

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Abstract: Heterocyclic compounds form a major class of compounds in chemistry. There has been considerable interest in the development of novel heterocyclic compounds with varied biological activities. Azoles are famous and widespread scaffold in the pharmaceutical industry due to their range of activities. Diazocarbonyl compounds have found numerous applications in many areas of chemistry. Among the most developed fields of diazo compounds. This approach represents a useful alternative to more conventional methods of the synthesis of azoles. The present review discussed the different characteristics of the azoles of interest, to recognize the differences in pharmacology, safety, toxicity and potential drug interactions of these antifungal agents.

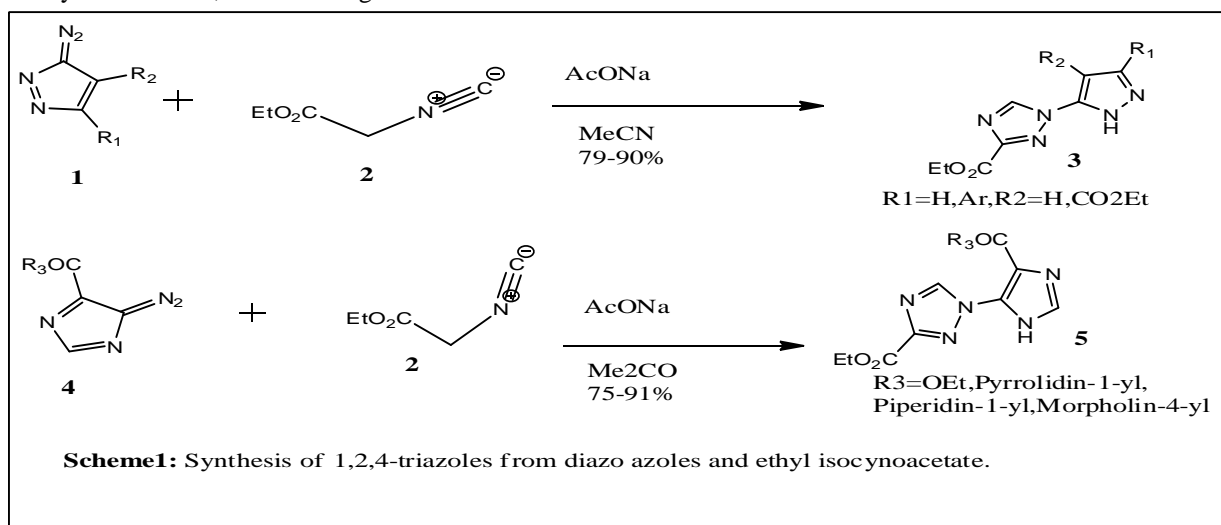
Keywords: Heterocyclic compound, biological activities, synthesis of azoles, Diazo compounds

I. INTRADUCTION

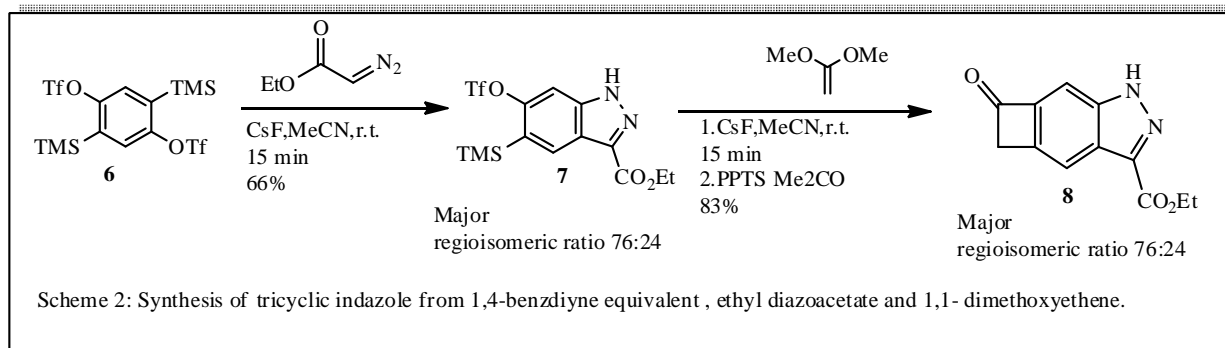
Azoles are the famous and widespread scaffold in the pharmaceutical industry due to their wide range of activities, high efficiency, good tolerability, and oral availability. Furthermore, azole derivatives have attracted attention as potent antimicrobial agents. The purpose of this review is to provide an overview of pharmacological aspects of the main scaffolds of azoles, including imidazole, triazole, and tetrazole, which possess antimicrobial activity. Diazo compounds are incredibly useful reagents in synthetic organic chemistry due to their extremely versatile and unique reactivity.

Recently, a range of new methods have been developed for the synthesis of various heterocycles from diazo compounds, some of which are described in review. Versatility and efficiency of diazo compounds are most clearly manifested in the synthesis of azoles—five-membered heterocycles containing a nitrogen atom and at least one other non-carbon atom of either nitrogen, oxygen, or sulfur. Diazo reagents allow synthesis of azoles of many types with two, three, and four heteroatoms. These heterocycles have found broad application in many fields such as organic electronics, functional materials, explosives, dyes, fluorophores, and especially medicine. This review aims to summarize all recent developments in this area covering all types of azoles available from diazocarbonyl and related compounds (oxazoles, thiazoles, imidazoles, pyrazoles, triazoles, tetrazoles, with the exception of 1,2,3-thiadiazoles), as well as to consider earlier useful methodologies not mentioned in other reviews.

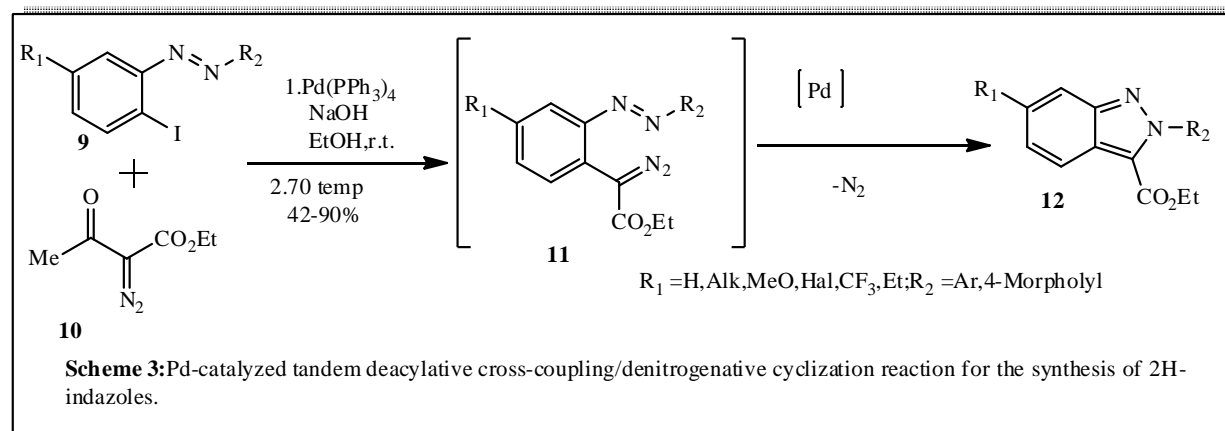
Diazo azoles, namely diazo pyrazoles **1** and diazo imidazoles **4**, were employed for the synthesis of N-azolyl-1,2,4-triazoles **3** and **5** in a study reported by Sadchikova and co-workers [1]. Interestingly, when a primary or secondary amide moiety was present in the diazo heterocyclic substrate, the reaction gave azolotriazinones instead of triazoles.



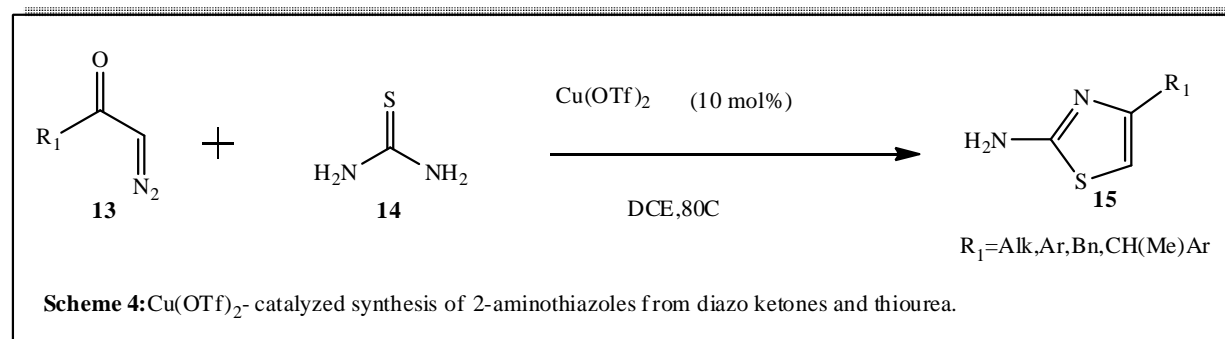
In 2016, Ikawa and co-workers demonstrated that 2,4-bis(trimethylsilyl)-1,3-bis(trifluoromethanesulfonyloxy) benzene **6** could act as a 1,4-benzdiyne equivalent [2]. Moreover, aryne "triple bonds" could be generated in a sequential manner. Using this methodology, an exotic indazole derivative **8** was prepared in a two-step procedure with good overall yield (Scheme 2)



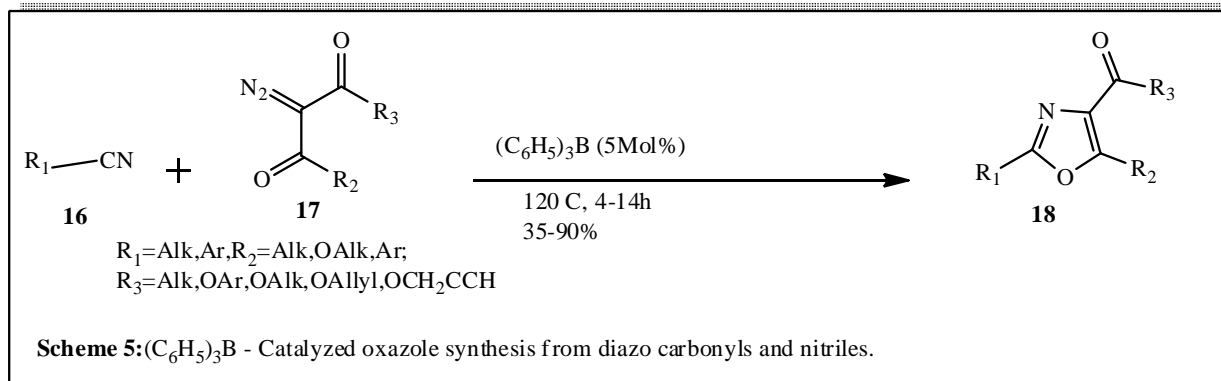
In a study by the Lee group, 2*H*-indazoles were prepared from 2-iodoazoarenes/2-iodoaryltriazenes **9** and α -acetyldiazoacetate **10** by a one-pot tandem palladium-catalyzed deacylative cross-coupling/denitrogenative cyclization process [3]. The method was applicable to a broad range of substituents in the aryl moiety of **9** and afforded 2-substituted 2*H*-indazoles **12** in generally good yields (Scheme 3). Moreover, this protocol provided entry into indazoles with different substituents in the two aryl rings, which is not achievable when using «aryne» methodologies.



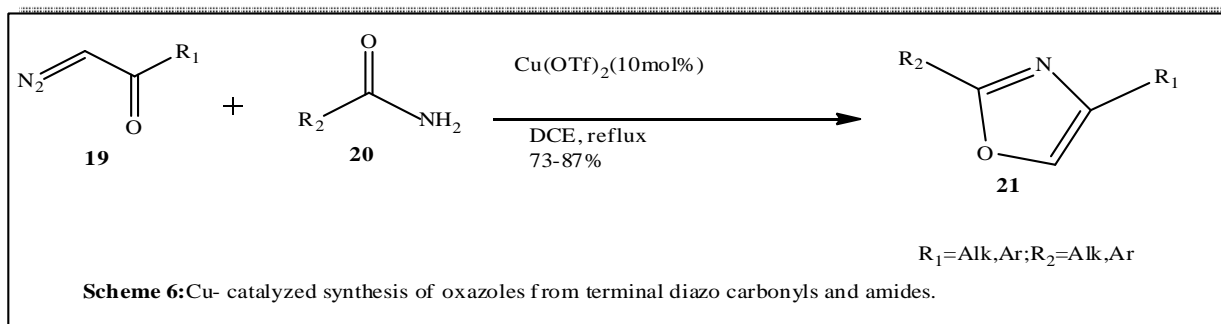
The Narsaiah group reported another example of the coupling of diazo compounds **13** with thiourea **14** under $\text{Cu}(\text{OTf})_2$ catalysis [4]. This protocol worked well for a variety of aliphatic, donor- and acceptor-substituted aromatic, and benzylic diazoketones, affording 2-aminothiazoles **15** in excellent yields. A catalyst-free version of this reaction using PEG-400 as a solvent was also reported (Scheme 4) [5].



Anusual tris (pentafluorophenyl) borane catalyst was employed for the reaction of nitriles **16** and diazo carbonyls **17** by Kumaraswamy and Gangadhar [6]. This metalfree protocol was found compatible with many functional groups including alkyl, aryl,alkyl(aryl)oxy, and allowed using a lower (5-fold) excess of the nitrile partners. A plausible reaction mechanism implies initial activation of diazocarbonyl compound **17** by the catalyst generating alkenediazonium salt, which is attacked by nitrile **16** with extrusion of N₂ molecule to give intermediate . The latter undergoes cyclization to form oxazole product **18** (Scheme 5).

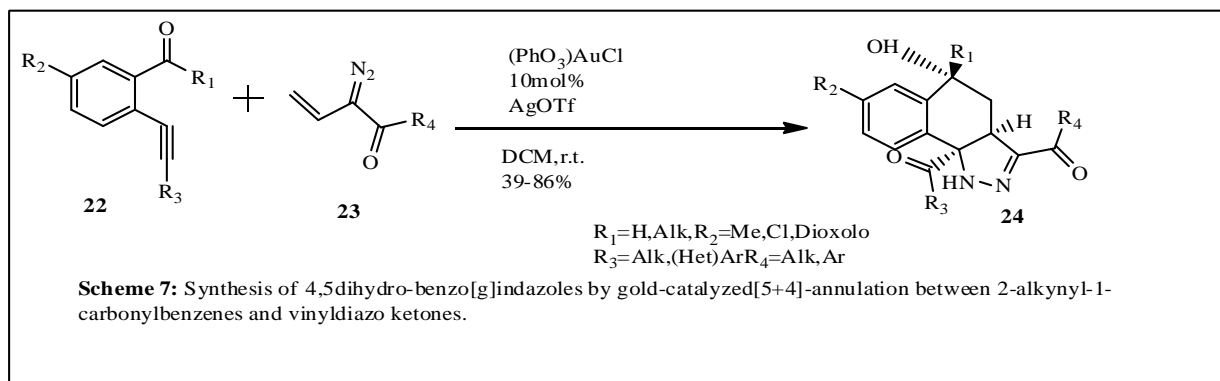


Cu(OTf)₂-catalyzed synthesis of 2,4-substituted oxazoles **21** from terminal diazocarbonyl compounds **19** and amides **20** was developed by the Subba Reddy group [7]. This method works well for both aliphatic and aromatic substrates providing good product yields. The reaction was unsuccessful only when both substrates were aliphatic. The following mechanism was invoked. Initially, copper carbene intermediate is formed on interaction of **19** with the Cu-catalyst. Rather unexpectedly, the subsequent attack of the amide on the Cu-carbene occurs through the carbonyl group, giving corresponding ylide in preference to the attack by the nitrogen atom, which would result in an NH-insertion product. Subsequent protodemetalation and cyclodehydration gives the observed oxazole **21**(Scheme 6).

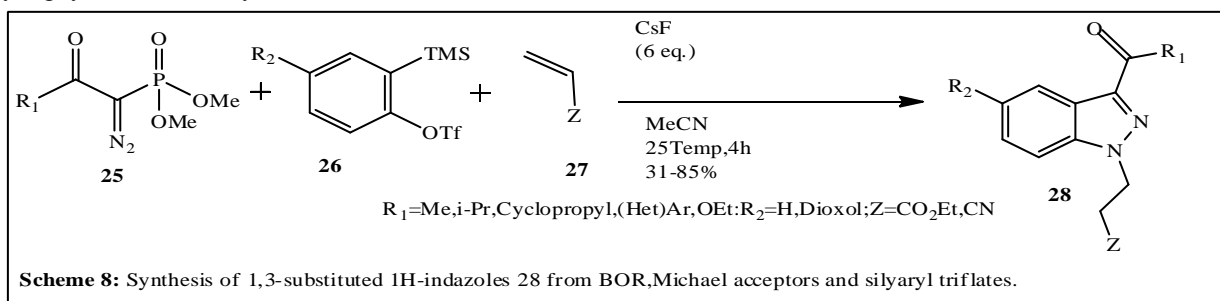


Imidazole derivatives are widely used in medicinal chemistry. Agents based on imidazole moiety possess anticancer, antifungal, antibacterial, antiviral, anti-inflammatory, and other biological activities [8]. Another area of application of imidazoles is in organic light-emitting diodes (OLEDs) and semiconductors [9]. To date, many methods have been developed for the synthesis of imidazoles, including the classical Debus-Radziszewski imidazole synthesis and reaction of *α*-halo carbonyl compounds with amidines [10,11]. However, syntheses based on diazocarbonyl compounds have not yet become widespread. There are two main approaches to the preparation of imidazoles using diazocarbonyl compounds: Metal catalyzed insertion into the NH bond of ureas followed by cyclization with formation of 2-imidazolones (which can be easily converted to substituted imidazoles), and NH-insertion into amides followed by cyclization in the presence of amine or an ammonia source. In addition, there are also some more exotic syntheses making use of isocyanides, imines, and nitriles

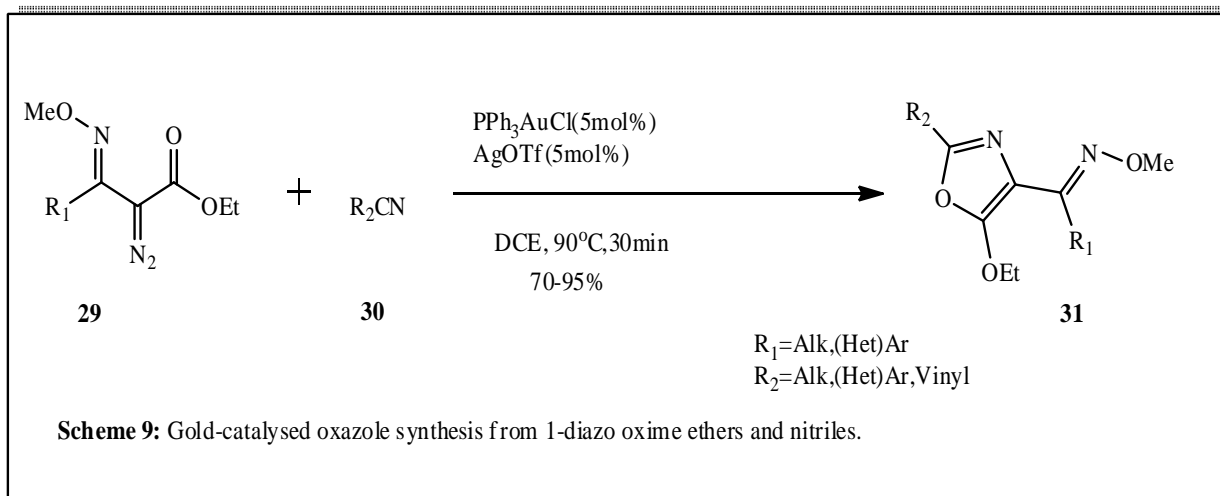
Another example of gold-catalyzed reaction of vinyl diazo compounds for the synthesis of pyrazole derivatives was described in 2019 by Raj and Liu [12]. The procedure involving [5+4]-annulation between 2-alkynyl-1-carbonylbenzenes **22** and vinyl diazo ketones **23** afforded 4,5-dihydro-benzo[g]indazoles **24** in generally good yields and with high diastereoselectivity. These initial products can be further converted to aromatic pyrazoles . The possible reaction mechanism likely involves the goldcatalyzed formation of benzopyrilium cation followed by its [5+4]-cycloaddition with vinyl diazo ketone **23** to yield intermediate. The latter can be hydrolyzed to give intermediate which then undergoes a disrotatory 6_e-electrocyclization with subsequent diastereoselective protonation leading to the observed pyrazoline **24**(Scheme 7).



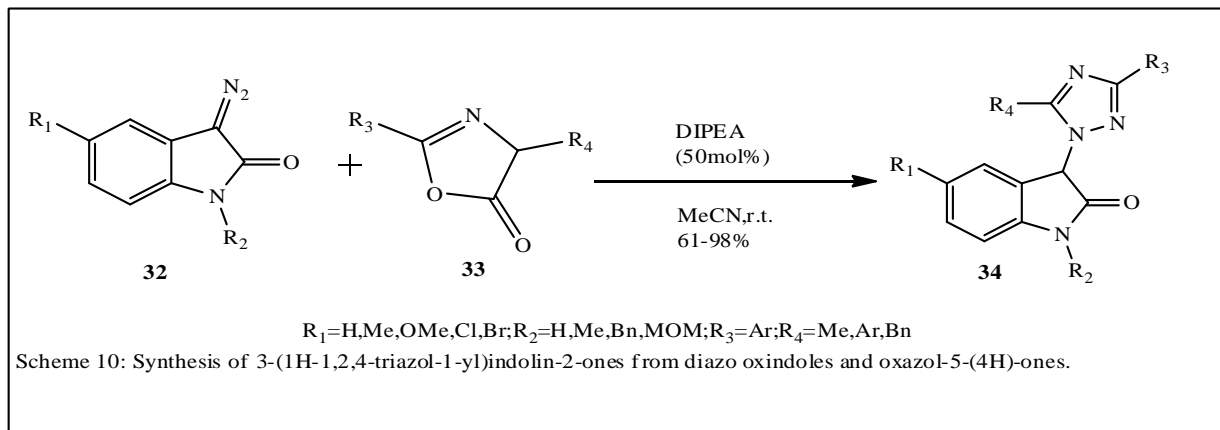
An efficient protocol for the synthesis of *N*-substituted indazoles from the Bestmann–Ohira reagent (BOR) analogs **25**, silylaryl triflates **26**, and Michael acceptors **27** was developed by Phatake and co-workers [13]. A role of fluoride anion in this reaction was not only in the generation of the aryne intermediate, but also in the dephosphonylation of BOR. The cascade [3+2] cycloaddition of the acyl diazomethane anion to aryne following the aza-Michael addition process furnished 1,3-substituted indazoles **28** in generally high yields under very mild reaction conditions (Scheme 8).



In 2016, the Park group developed an efficient method for the synthesis of highly substituted oxazoles **31** by gold-catalyzed cycloaddition reaction of α -diazo oxime ethers **29** and nitriles **30** (Scheme 9) [14]. The reaction tolerated a broad range of substituents (including alkyl, aryl, heteroaryl, and vinyl) and afforded good to excellent product yields. The formation of the oxazole ring is believed to proceed via the formation of zwitterionic intermediate, which subsequently undergoes cyclization to oxazole **31**. Interestingly, the cyclization occurred via the attack of the carbonyl group rather than the attack of the oxime ether group on the nitrilium electrophilic center, presumably due to the *Z*-configuration of the C=N double bond (Scheme 9)

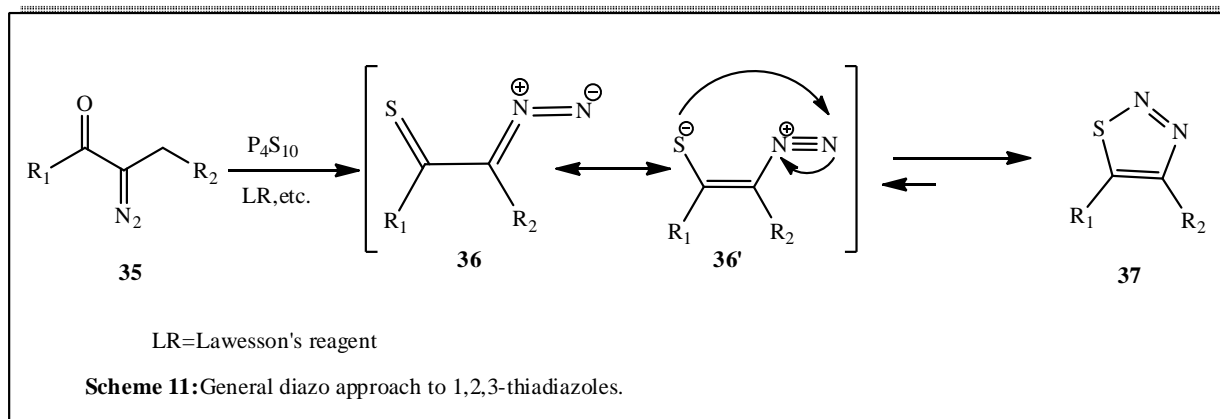


In 2018, the Chen group described a base-mediated reaction of diazo oxindoles **32** with oxazol-5-(4H)-ones **33** affording 3-(1H-1,2,4-triazol-1-yl)indolin-2-ones **34** in reasonable yields (Scheme 10) [15]. The best results were obtained with electron-donating R1 groups on the phenyl ring of **32**. As for the oxazolones **33**, electron-poor aryl rings as the R3 substituents provided the desired product in higher yields in comparison with electron-rich ones. Noteworthy, when 4-unsubstituted oxazolone **33** (R4 = H) was used, the reaction failed to give any triazole product. Attempts to perform the reaction with diazo dicarbonyl compounds and a vinyl diazo compound did not work as well. The mechanism of the reaction is presumably similar to the previous example.

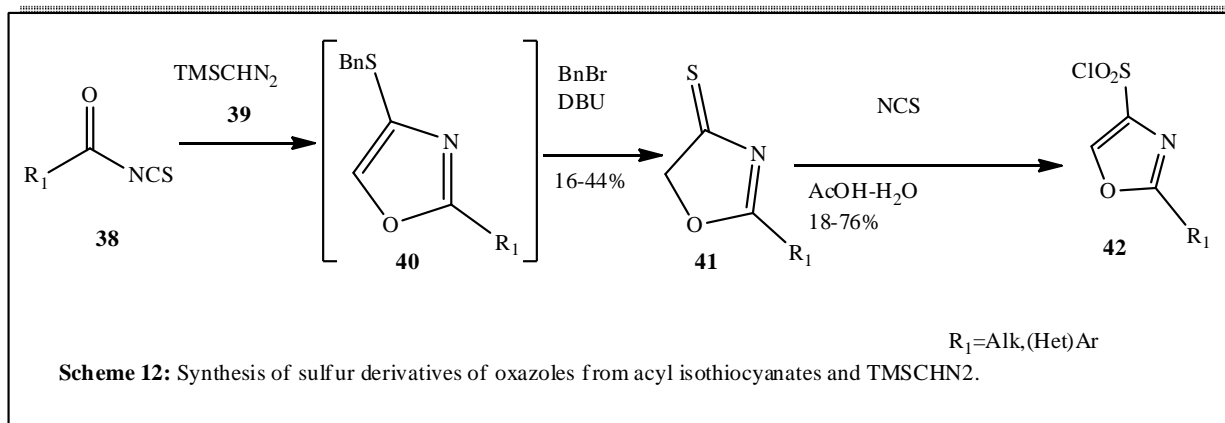


1,2,3-Thiadiazoles

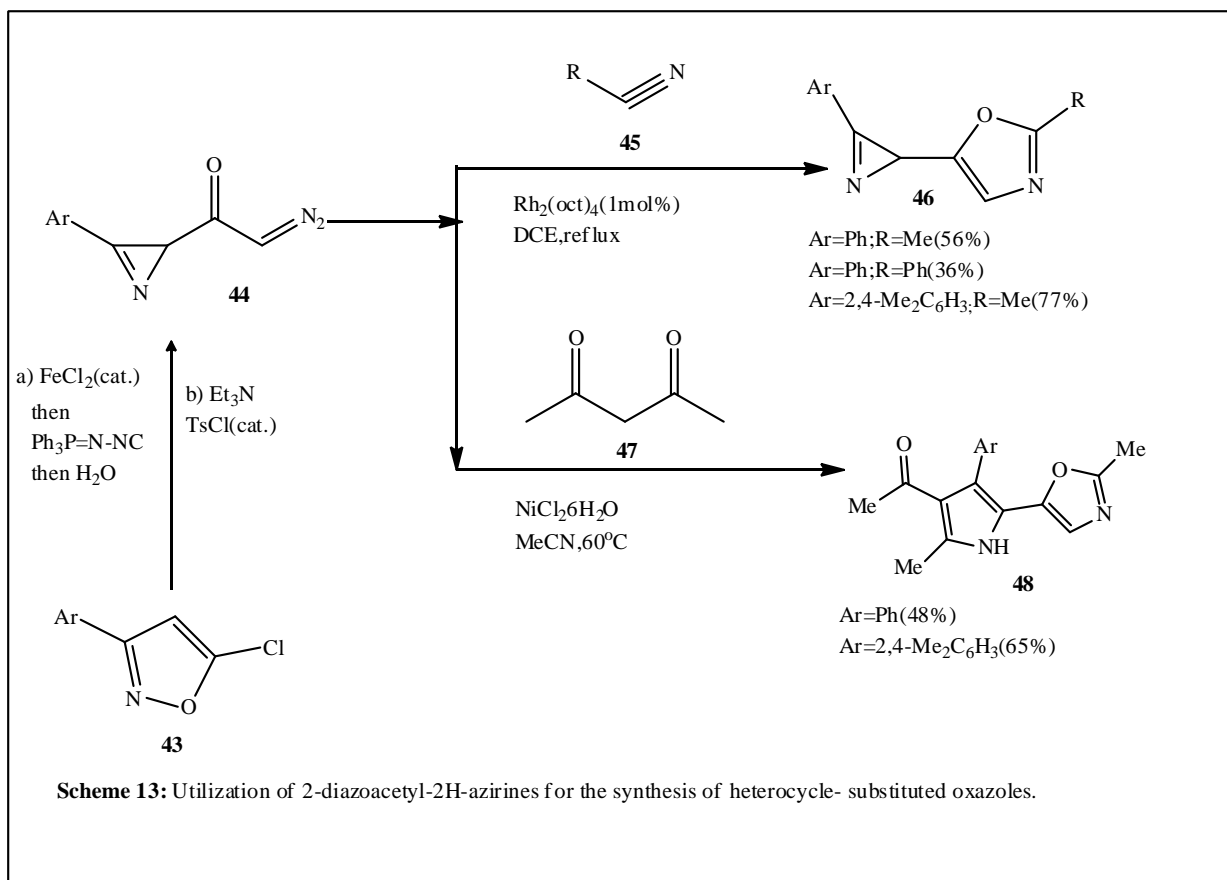
A general approach to the synthesis of 1,2,3-thiadiazoles from diazo compounds is based on the reaction of diazocarbonyl compound **35** with a thionation reagent. The initially formed diazothiocarbonyl compound **36** is usually unstable and undergoes 1,5-electrocyclization to form 1,2,3-thiadiazole **37** (Scheme 11). This topic has been recently reviewed by Shafran and co-authors [16], therefore we chose not cover it in the present review.



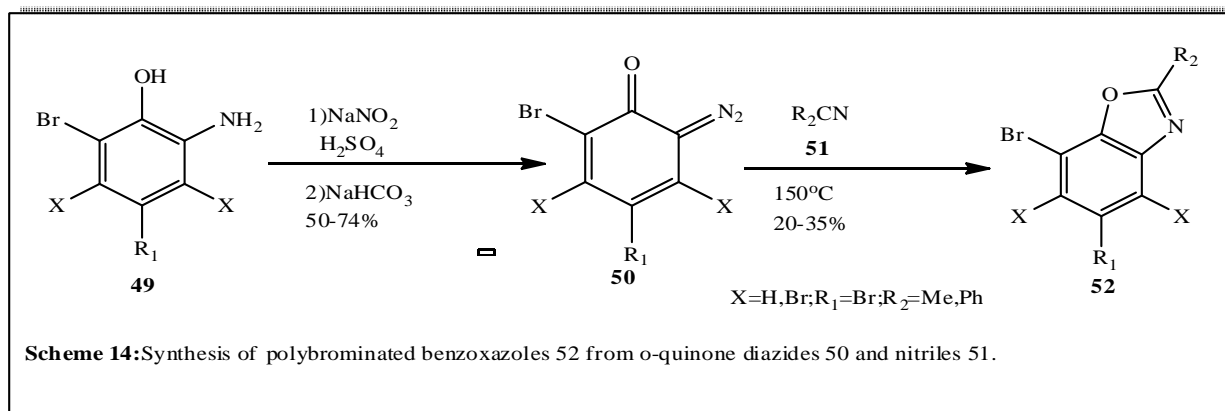
In 2015, Trujillo and co-workers synthesized a series of novel 4-(benzylthio)oxazoles **41** by reacting acyl isothiocyanates **38** with trimethylsilyl diazomethane (TMSCHN₂) **39** with subsequent treatment of the oxazole thiones **40** with BnBr-DBU [17]. Isolated in moderate yields, compounds **41** were then converted to oxazole sulfonyl chlorides **42** in treatment with N-chlorosuccinimide (NCS). BnS-derivatives **41** were formed along with thiadiazoles, which is explained by two possible reaction pathways. In path A, initially formed intermediate underwent cyclization via the attack of the carbonyl group on the carbon atom adjacent to N₂⁺, while in path B, intermediate undergoes cyclization via the attack of the thiocarbonyl group on the N₂⁺ moiety (Scheme 12).



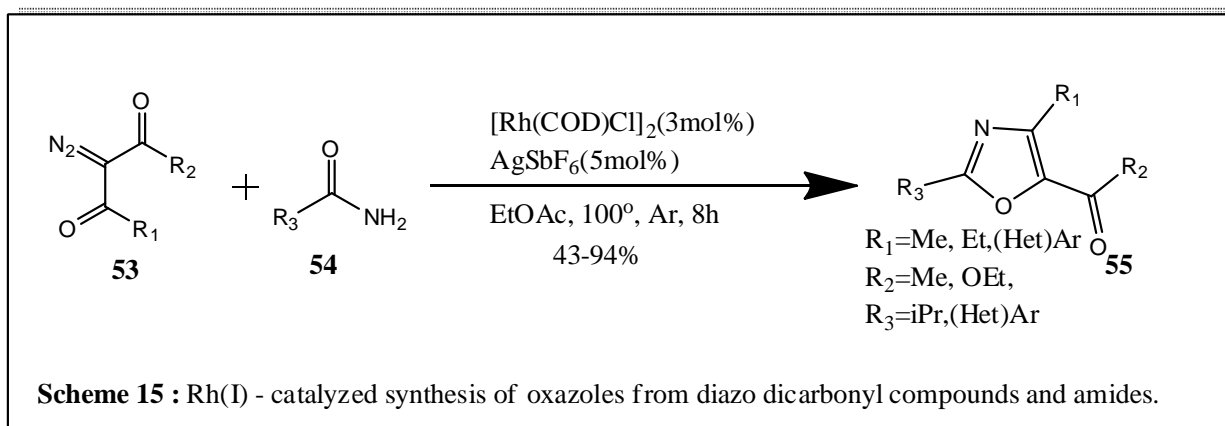
In the synthetic exploration of 2-diazoacetyl-2H-azirines **5**, which are available from 5-chlorisoxazoles **43**, Khlebnikov and co-workers demonstrated the utility of **44** for the preparation of oxazoles [24]. Rhodium-catalyzed reaction with nitriles **45** afforded oxazoles **46**. In the synthetic exploration of 2-diazoacetyl-2H-azirines **5**, which are available from 5-chlorisoxazoles **43**, Khlebnikov and co-workers demonstrated the utility of **5** for the preparation of oxazoles [18]. Rhodium-catalyzed reaction with nitriles **45** afforded oxazoles **46** with preservation of azirine functionality. On the other hand, NiCl₂-catalyzed reaction with acetylacetone **47** in acetonitrile provided pyrrolyl-substituted oxazoles **48** (Scheme 13).



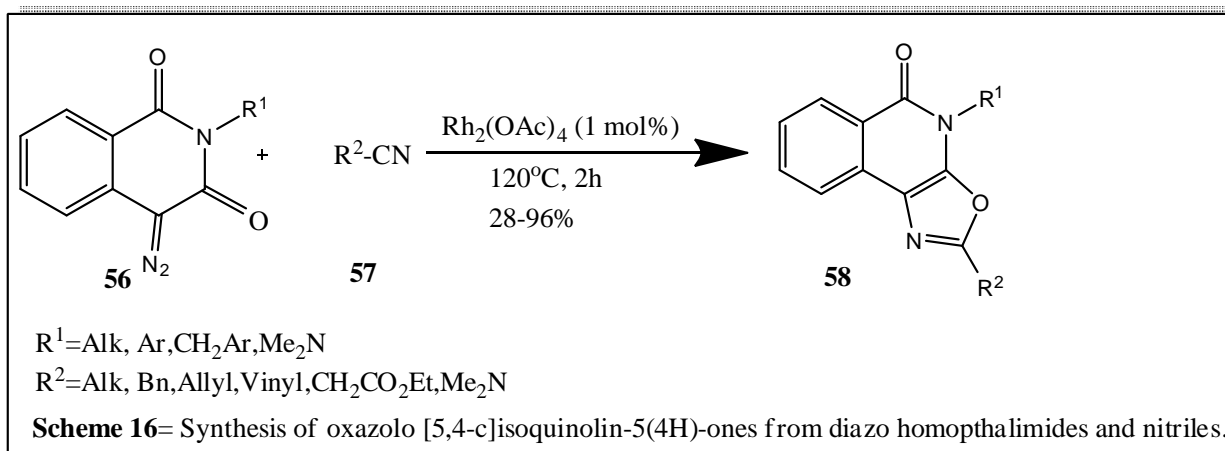
The reaction between polybrominated o-quinone diazides **50** (available via diazotization of aminophenols **49**) and nitriles **51** leading to benzoxazoles **52** was described in 2017 by Vasin and co-workers [19]. The reaction proceeded on conventional heating, albeit in modest yields (Scheme 14).



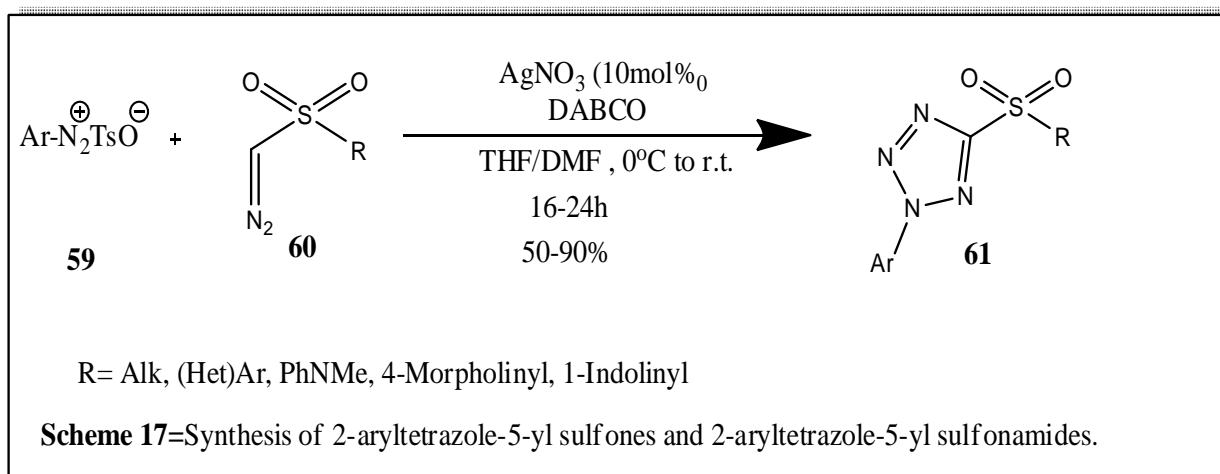
In 2018, Chen and co-workers reported the involvement of diazo dicarbonyl compounds **53** in a similar reaction under Rh(I) catalysis, which afforded fully substituted oxazoles **55** [20]. In this case, however, the yields were significantly lower for aliphatic substrates in comparison to the previous example. From the mechanistic perspective, rhodium(I) carbene complex likely reacted with the carbonyl oxygen atom of amide to form intermediate, which underwent metal protonation (with regeneration of the catalyst) to afford *b*-ketoester. The latter entered the cyclization/dehydration sequence, thus producing trisubstituted oxazole. Preliminary mechanistic studies revealed that the attack on Rh(I) carbenes was likely the rate-determining step (Scheme 15).



Diazo homophthalimides **56** were the first heterocycles containing an *α*-diazocarbonyl moiety, which were involved in a [3+2] cycloaddition with nitriles **57** to deliver oxazole-fused adducts **58** as reported by the Krasavin group [21]. The reaction displayed a generally high functional group tolerance giving low yields only in case of olefinic, benzylic, and sterically hindered nitriles. Diazo homophthalimides **56** are easily available via diazo transfer to the corresponding homophthalimides (Scheme 16).

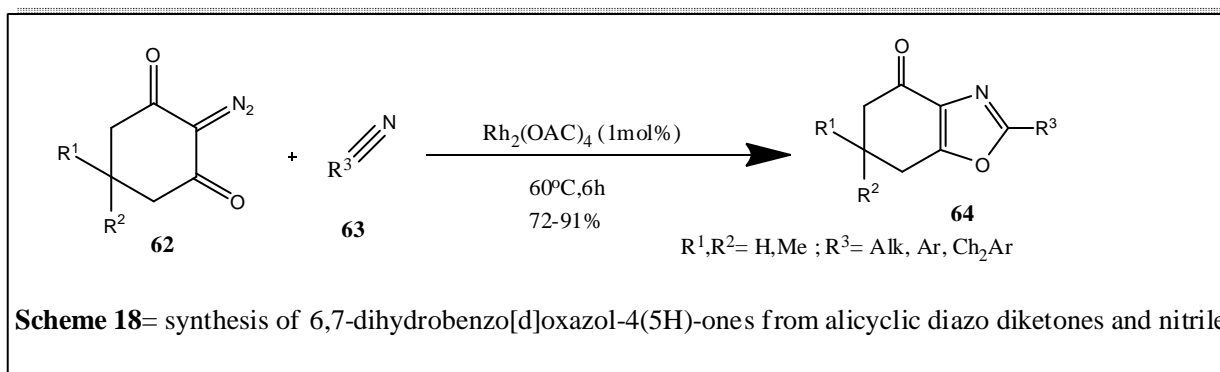


In a 2020 work by the Krasavin group, it was demonstrated that (hetero)aryl-, alkylsulfonyl, and aminosulfonyl diazomethanes **60** are also applicable for this reaction [22]. Thus, the rare 2-aryltetrazol-5-yl sulfones and previously not described in the literature 2-aryltetrazol-5-yl sulfonamides **61** were prepared in generally good yields. However, diminished yields were observed with aryl diazonium tosylates **59** bearing electron donating or ortho-substituents. The following plausible mechanism was suggested (which can be presumably extended also to the examples discussed above). Diazo substrate **60** was deprotonated by the base and bound by the Ag cation to give intermediate . The latter coordinated with diazonium salt in a way that unambiguously determines the regiochemistry of the final product, i.e., providing intermediate . Subsequent silverassisted cycloaddition with the liberation of the catalyst delivers the desired tetrazole **61** (Scheme 17).



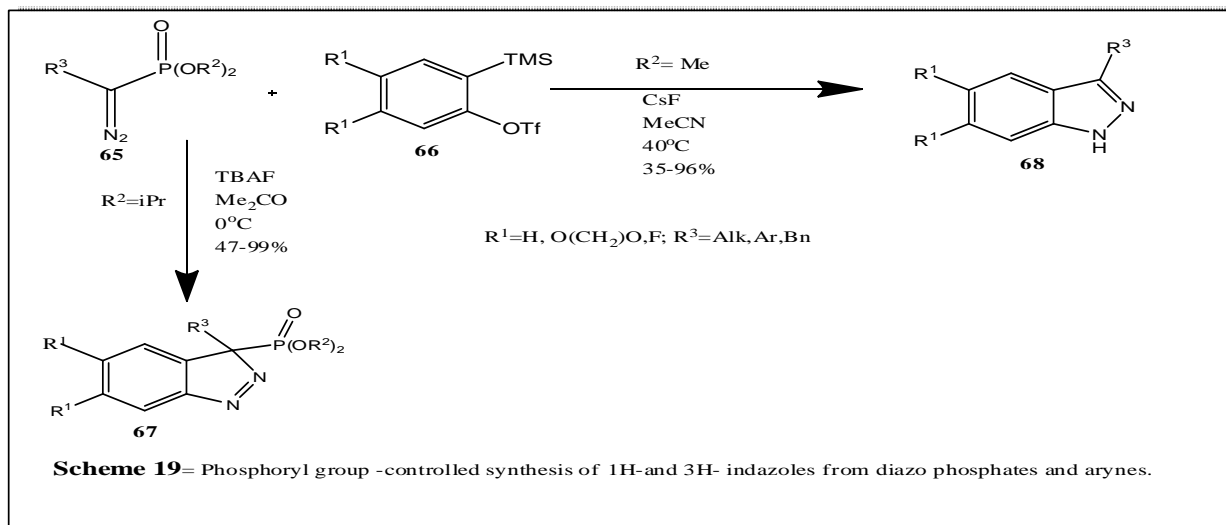
Alicyclic diazocarbonyl compounds have also been employed in oxazole synthesis.

Thus, in 2018, Fan and co-workers described the synthesis of 6,7-dihydrobenzo[d]oxazol-4(5H)-ones **64** by reacting cyclic diazo diketones **62** with nitriles **63** [23]. The best yields were obtained with non-substituted diazo substrates. Analogously to the example discussed above, diazo Meldrum's acid did not provide the desired oxazole. From the mechanistic standpoint, the reaction likely proceeds via the attack of the nitrile on the rhodium carbene, generated from diazo species, following steps similar to the above-mentioned examples (Scheme 18)

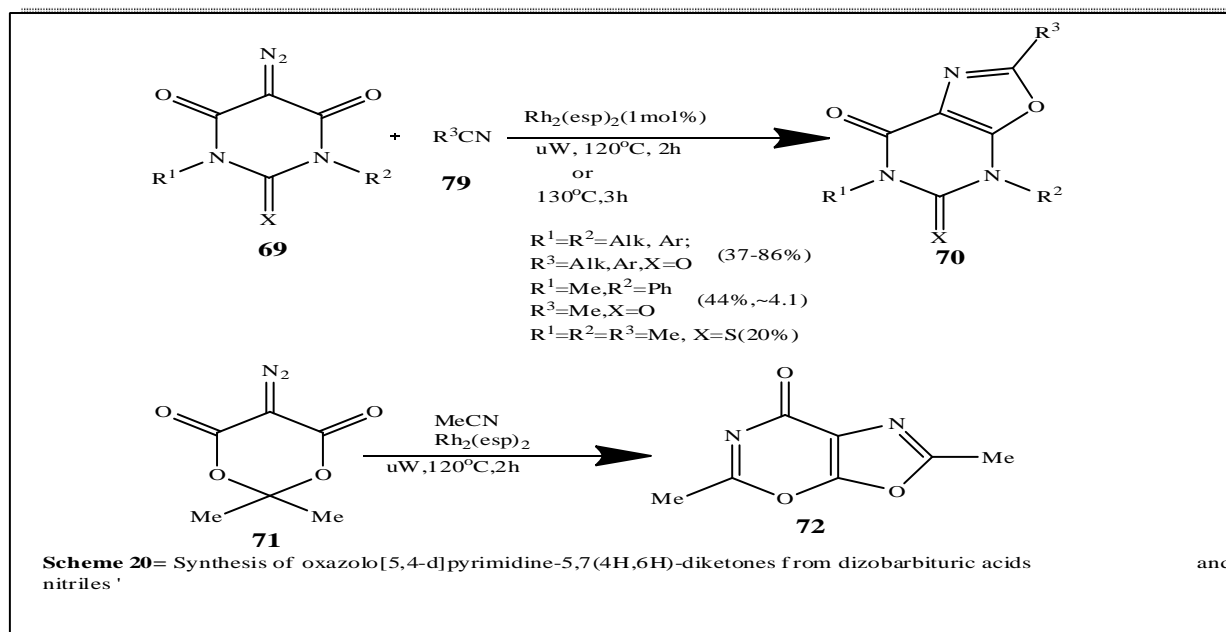


1,3-Thiazole-based materials are now extensively studied for application in organic electronics due to their suitable electronic, optical, and spatial properties. 1,3-Thiazoles have been employed for the preparation of organic field-effect transistors (OFETs), organic photovoltaic cells (OPVs), and organic light-emitting diodes (OLEDs) [24]. Moreover, the thiazole ring has been utilized in the design of medicinal agents for the treatment of viral and bacterial infections, diabetes, Alzheimer's disease, neglected protozoan diseases, and more [25]. Standard approaches to the synthesis of 1,3-thiazoles, such as thionation of α -acylaminoketones (the Gabriel synthesis) and condensation of α -halocarbonyl compounds with thioamides (the Hantzsch synthesis) are frequently employed [26,27].

The diazo approaches to thiazoles discussed in this subsection of the review are somewhat similar to these classic methods: (i) reaction of *a*-diazocarbonyl compounds (acting as the *a*-halocarbonyl compounds equivalent) with thioamides or thioureas; (ii) *a*-carbonyl carbene NH insertion into thioamides or thiourea with subsequent thionation; and (iii) less conventional reactions. A tunable approach for the synthesis of 1*H*- and 3*H*-indazoles was reported by Chen and co-authors [136]. The product distribution depended on the structure of the phosphoryl group in *α*-substituted-*α*-diazophosphonates **65**. Bulky diisopropyl phosphonates provided stable 3*H*-indazoles **67**, whereas less sterically hindered dimethyl phosphonates gave 1*H*-indazoles **68** (Scheme 19)

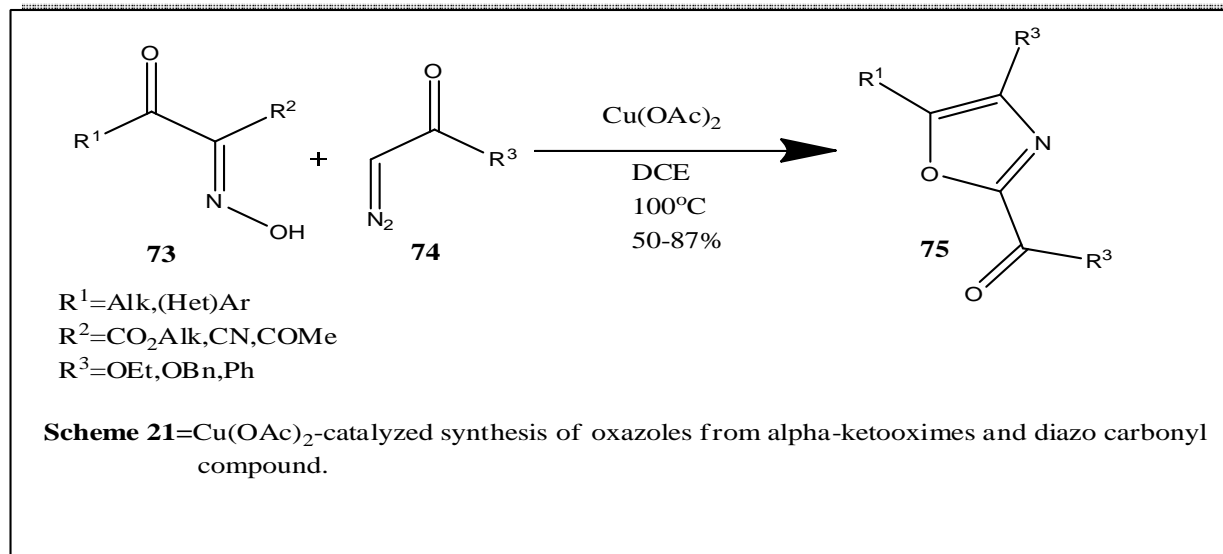


Further exploring the synthesis of fused oxazoles from diazo containing heterocycles, the same group reported a [3+2] cycloaddition reaction of diazobarbituric acid derivatives **69** (available from barbituric acid via a novel sulfonyl-azide-free (SAFE) diazo transfer protocol [28]) with nitriles **79** [28]. Diversely substituted oxazolo[5,4-*d*]pyrimidine protocol [28]) with nitriles **79** [29]. Diversely substituted oxazolo[5,4-*d*]pyrimidine-5,7(4*H*,6*H*)-diones **70** were obtained in generally moderate to good yields. However, mono-*N*-substituted diazobarbituric acid failed to give the desired product. Non-symmetric diazobarbituric acid gave mixture of regioisomers. Notably, the attempt of performing this reaction with diazo Meldrum's acid led to the formation of oxazolo[4,5-*e*][1,3]oxazinone via acyl ketene intermediate, which was involved in [4+2] cycloaddition with another molecule of acetonitrile (Scheme 20).

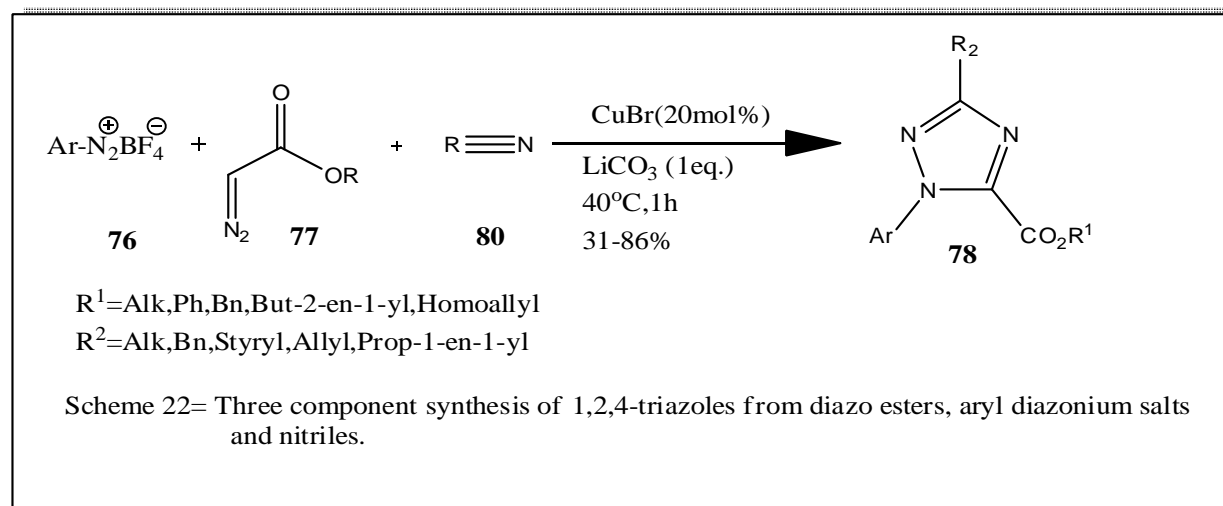


N

The reaction of α -keto oximes **73** with terminal α -diazo carbonyl compounds **74** afforded polysubstituted oxazoles **75** in moderate to good yields as reported in 2018 by Swamy and co-workers [30]. The reaction tolerates many substituents, including alkyl, (hetero)aryl, as well as ester, ketone, and nitrile functional groups. The following reaction mechanism was proposed by the authors. The reaction is believed to be initiated by the interaction between copper carbene and keto oxime, which leads to aziridine. The latter undergoes an intramolecular rearrangement to form intermediate. Subsequent dehydration leads to oxazole **75** (Scheme 21).



A three-component protocol for the preparation of 1,2,4-triazoles **78** from aryl diazonium salts **76**, diazo esters **77**, and nitriles **80** was developed by the Wan group [31]. EDG-substituted aryl diazonium salts performed better than EWG-substituted ones. Based on preliminary mechanistic studies and DFT-calculations, the authors suggest the following mechanism for the reaction. Nitrile **80** most likely attacks copper carbene to form intermediate, which then undergoes a highly regioselective [3+2] cycloaddition to diazonium salt **76** to afford cyclic intermediate. The latter suffers base-mediated isomerization to deliver the desired 1,2,4-triazole **78** (Scheme 22).



II. CONCLUSION

Most recently, great progress has been achieved in the synthesis of tetrazoles from various diazo compounds. However, it is obvious that the synthetic potential of diazo compounds in this area is far from its full disclosure. In addition, despite the extensive research, some mechanistic details are still not clear and need further investigation. Given the easy availability of diazo substrates, the described methodologies represent a valuable alternative to more conventional methods. As we have demonstrated in this review, diazocarbonyl and related compounds provide rapid and efficient access to a wide range of various azoles.

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