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S_N^H methodology and new approaches to condensed heterocyclic systems*

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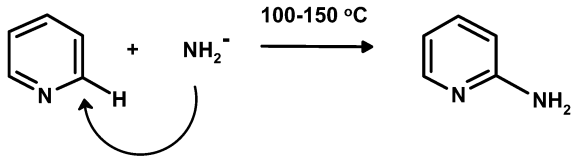
Abstract: The review surveys the reactions of electron-deficient azaaromatic compounds with mono- and bifunctional nucleophilies in which a nucleophilic attack at the unsubstituted CH carbon of an aromatic ring is one of the key steps. Use of the S_N^H methodology for the synthesis of fused heterocyclic systems by means of nucleophilic addition–addition A_N–A_N, addition–substitution of hydrogen A_N–S_N^H, tandem substitution of hydrogen S_N^H–S_N^H, and other strategies will be discussed. Intramolecular S_N^H reactions will also be considered as effective synthetic tools to obtain condensed heterocyclic systems.

INTRODUCTION

Nearly 100 years have passed since Russian professor Alexey Chichibabin first discovered the ability of pyridine to replace hydrogen atom in the α-position by action of sodium amide, and the formation of this type of carbon–nitrogen bond seems to be one of the first examples of the so-called nucleophilic substitution of hydrogen (S_N^H) reactions (Scheme 1) [1].



Scheme 1

For many years, the synthetic potential of the S_N^H reactions, however, had not been estimated properly, and nucleophilic aromatic substitution reactions were associated mainly with the *ipso*-attack resulting in displacement of good leaving groups (S_N^{ipso}) (Scheme 2) [2–7].

During the last two to three decades, it has been established that carbon atoms in electron-deficient aromatics bearing hydrogen are more vulnerable for nucleophilic attack than those bearing good leaving groups [8–25]. For instance, ammonia reacts with 2-methylthio-5-nitropyrimidine at C4 and C6 at low temperatures to give the corresponding 4-amino adduct which is oxidized by potassium permanganate into 4-amino compound [26]. In case the methoxy group is present in position 4, amination of 5-nitropyrimidine takes place at C2 or C6 to give the corresponding 2- and 6-amino compounds [26].

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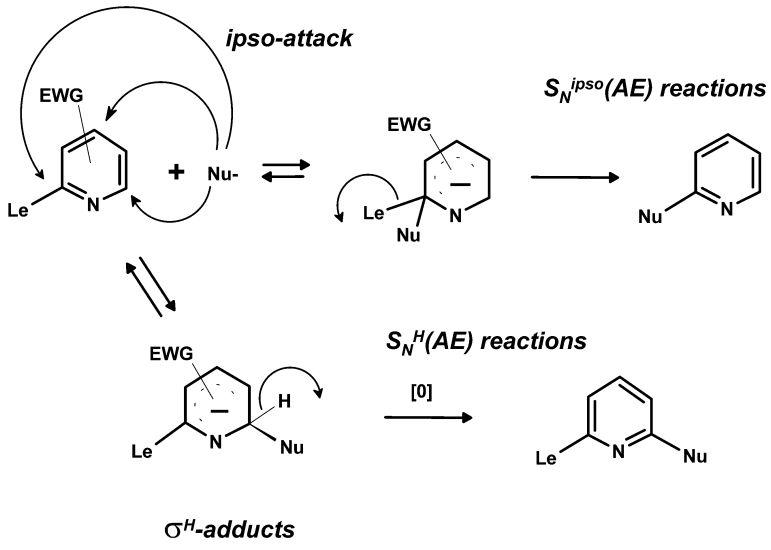
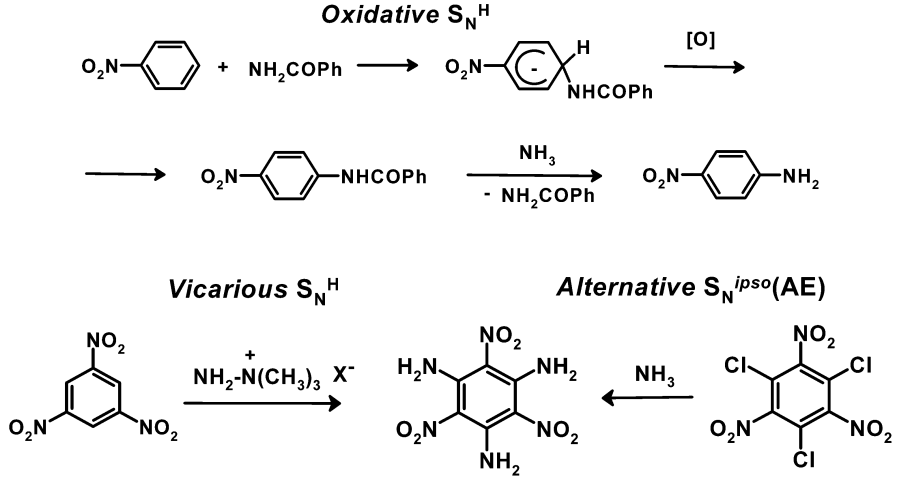


EWG – Electron-withdrawing group; Le – Leaving group

Scheme 2

The Chichibabin amination reaction was applied to a variety of heterocycles [1,8], and many research groups contributed to this field [1,8–10,26–29]. In particular, Prof. H. C. van der Plas and coworkers modified the reaction considerably and suggested the use of a rather effective system "liquid ammonia-potassium permanganate" for amination of azaaromatics (for a review, see [27]).

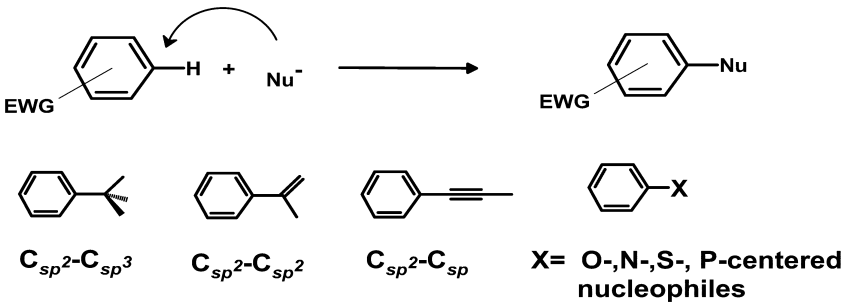
A considerable body of the data on nucleophilic amination and other types of the S_N^H reactions accumulated in the literature [1,8–10,24–29] show that these reactions are of fundamental value for the chemistry of electron-deficient aromatics. Two principal versions of the S_N^H reactions, the oxidative one and vicarious substitution of hydrogen, appear to be complimentary to each other. Indeed, the oxidative procedure for preparation of *p*-nitroaniline from nitrobenzene and benzamide [28] and vicarious amination of 1,3,5-trinitrobenzene into the corresponding 1,3,5-triamino-2,4,6-trinitrobenzene (Scheme 3) [29] are examples of the advanced organic syntheses which have found applications as large-scale industrial processes.



Scheme 3

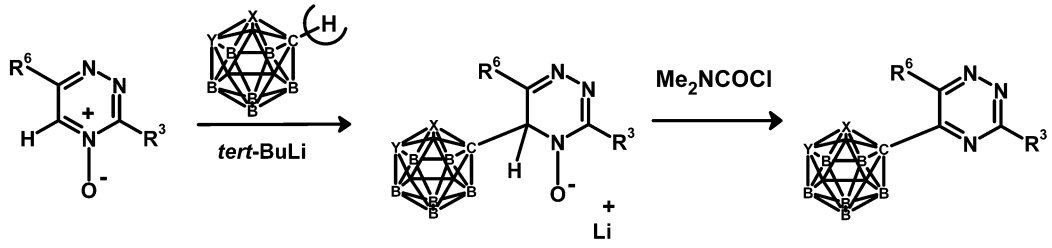
S_N^H METHODOLOGY

Introductory notes and the examples presented above provide some details concerning the progress in the S_N^H amination of aromatic compounds as an efficient way to form C–N bond with an aromatic ring, but they cannot pretend to describe even a small part of this promising field of organic synthesis. It is well recognized now that the S_N^H methodology is a powerful synthetic tool to build a variety of new carbon–carbon or carbon–heteroatom chemical bonds, as generalized in Scheme 4 [1,10–29].



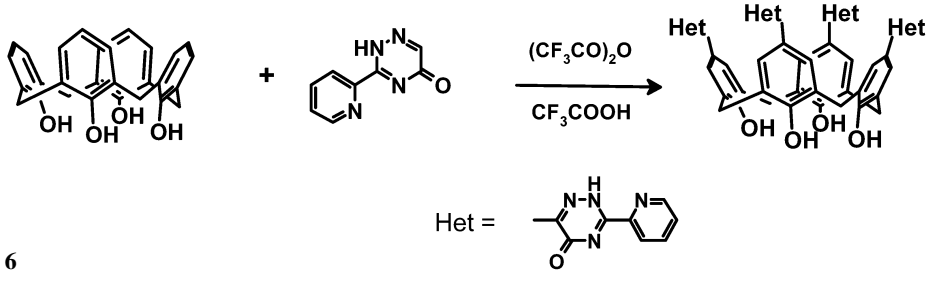
Scheme 4

We would like to illustrate the scope of the S_N^H reactions by several examples. Nucleophilic substitution at a sp^2-carbon in electron-deficient aromatic compounds enables one to modify their structure by introducing fragments of all kind of carbanions. For instance, it has recently been shown that carbanions generated from carboranes on treatment with lithium *tert*-butyl are able to substitute hydrogen in pyrazines, triazine-*N*-oxides, and other π-deficient azines (Scheme 5) [30].



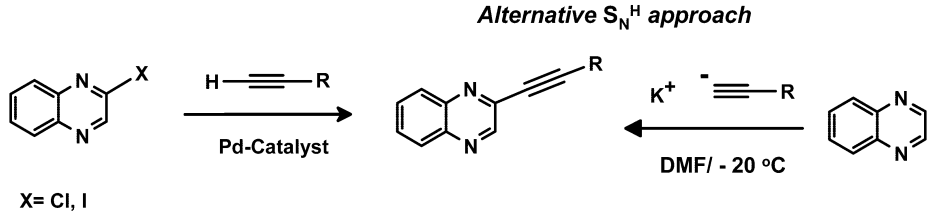
Scheme 5

Another example concerns calixarenes, a very important class of macrocyclic compounds which are widely used as ligands in supramolecular chemistry. Modification of the upper rim of calix[4]phenols by means of the S_N^H coupling with electron-deficient triazinones is a new approach to change molecular cavities of these compounds, which opens new possibilities for construction of highly selective ligands (Scheme 6) [31].



Scheme 6

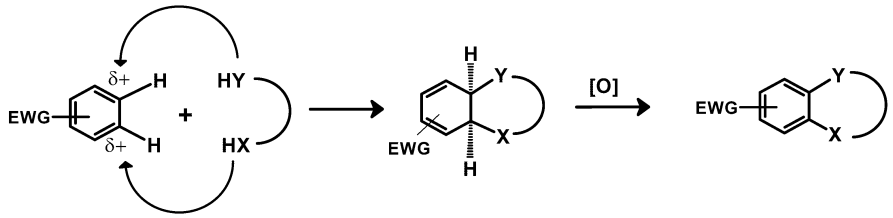
The S_N^H methodology has recently been used to introduce a triple carbon–carbon bond into an aromatic ring and this approach can be regarded as an alternative to the well-known palladium-catalyzed cross-coupling Sonogashira reaction (Scheme 7) [32].



Scheme 7

TANDEM REACTIONS

In this paper, we wish to show how the S_N^H methodology can be used for the synthesis of condensed heterocycles. In addition to well-known approaches for the synthesis of fused carbo- and heterocyclic systems which are based on displacement of two good-leaving groups we will outline new methods which exploit tandem nucleophilic addition or substitution reactions on two neighboring carbons in an aromatic ring (Scheme 8) [1,12].

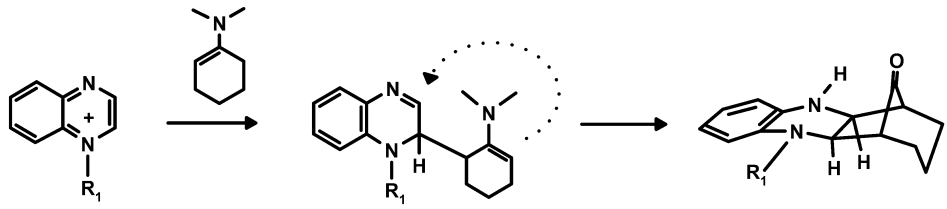


Scheme 8

Taking into account the plausible oxidation step for intermediate σ^H-adducts, there might be a variety of sequences in these tandem reactions: «Addition–Addition» (A_N–A_N), «Addition–Addition–Elimination of Hydrogen» (A_N–S_N^H), «Addition–Addition–Elimination of Two Hydrogen Atoms» (S_N^H–S_N^H), «Addition–Substitution of a Good Leaving Group (A_N–S_N^{ipso}), «Substitution of Hydrogen–Substitution of a Good Leaving Group (S_N^H–S_N^{ipso}), etc. It is clear that in the frames of this short review article we are not able to provide an overwhelming report on all these sequences, therefore we shall consider only main features of the tandem reactions.

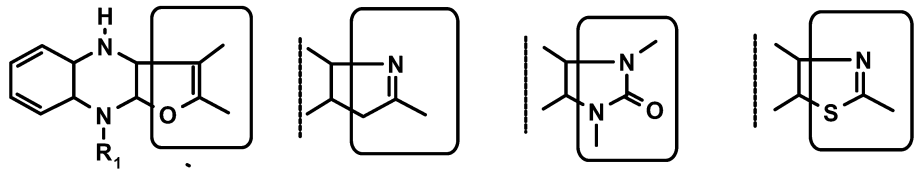
A_N–A_N tandem reactions

The idea of exploiting double addition of bifunctional reagents on two neighboring carbons of an azine ring for the synthesis of fused heterocycles proved to be a constructive one. In the series of azinium cations, it has first been demonstrated by the reaction of *N*-alkylquinoxalinium salts with enamines [33]. In this cyclization, enamines act as bis-carbanionic reagents: the second carbanionic center develops in the course of the reaction due to deprotonation of the intermediate iminium salts (Scheme 9) [33].



Scheme 9

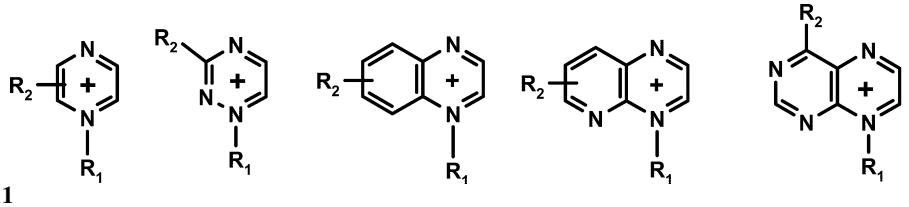
The reaction was applied to diketones [34–36], ureas and thioureas [37], thiamides [38–40], dithiocarbamates [41], iminoesters, and amidines and other 1,3-bifunctional nucleophiles [42–43]. A variety of tetrahydroquinoxalines condensed with five-membered heterocycles have been obtained using this methodology (Scheme 10) [11,12,33–44].



Scheme 10

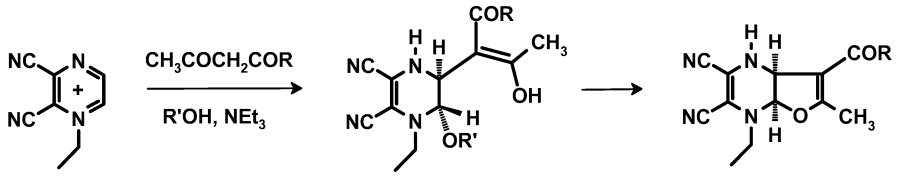
In a similar manner, tetrahydropyrazines condensed with six-membered heterocylces were obtained by cyclizations of 1,4-diazinium salts with 1,4-bifunctional nucleophiles [11,12,44–53]. *Cis*-configuration of the ring junction hydrogen atoms, regio-orientation of asymmetric fragments fused with the pyrazine ring, as well as stereochemical features of cyclizations have been established by NMR and X-ray crystallography analysis [11].

The general character of cyclizations with bifunctional nucleophiles has been demonstrated by using quaternary pyrazinium and quinoxalinium salts [33–53], pyrido[2,3-*b*]pyrazinium [36], 1,2,4-triazinium [54–59], and pteridinium [60] cations, which have in common the 1,4-diazadiene fragment (Scheme 11).



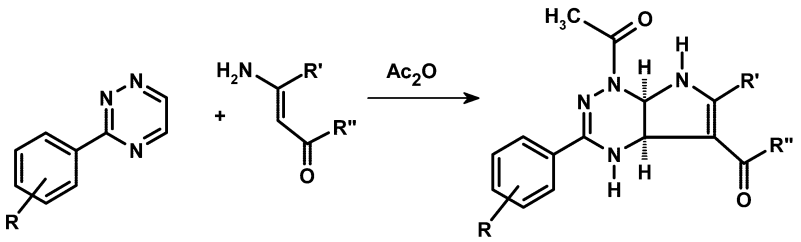
Scheme 11

One of the recent examples of the A_N–A_N tandem reactions in the series of 1,4-diazinium cations is annelation of the furan rings to 2,3-dicyanopyrazines [61]. An interesting feature of this reaction proceeding in alcohols R′OH is that the intermediate diadduct bearing the alkoxy group OR′ in the *trans*-position relative to β-ketoester substituent, as established by X-ray analysis, is transformed into tetrahydrofuro[2,3-*b*]pyrazine with *cis*-configuration of the ring-junction hydrogen atoms (Scheme 12) [61].



Scheme 12

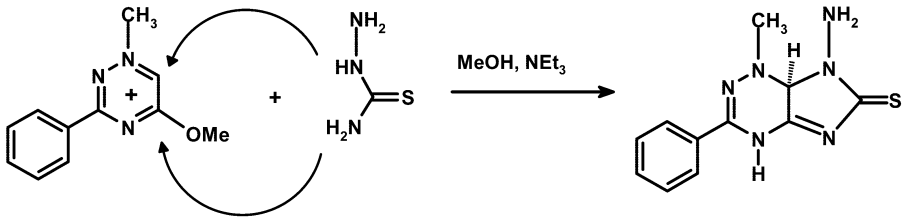
Not only 1,4-diazinium cations are able to undergo the tandem A_N–A_N reactions. 1,2,4-Triazines are similar to 1,4-diazines in reactivity. Being activated by means of *N*-protonation [13,54,56], *N*-alkylation [13,18,54,55], or *N*-acylation [56–59], 1,2,4-triazines are prone to form cyclo adducts with bifunctional nucleophiles (Scheme 13).



Scheme 13

A_N–S_N^{ipso} tandem reactions

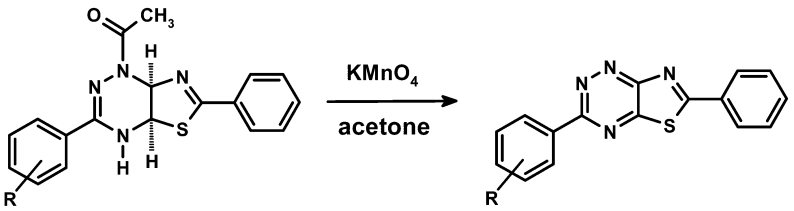
This sequence of tandem reactions has been successfully applied for the synthesis of condensed triazines by reacting 1-methyl-3-phenyl-5-methoxy-1,2,4-triazinium cation with thiosemicarbazide. It is worth noting that in this reaction the A_N addition step occurs faster than the S_N^{ipso} displacement of the methoxy group (Scheme 14) [56].



Scheme 14

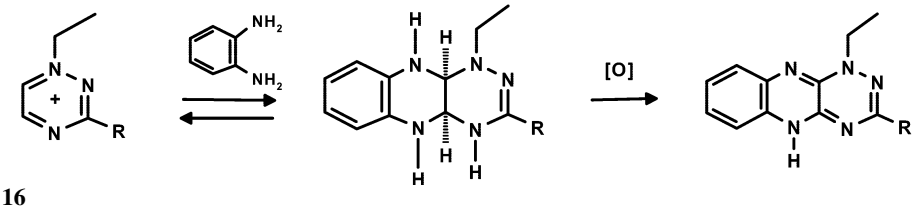
Tandem S_N^H–S_N^H reactions

Condensed tetrahydropyrazines or their aza-analogs derived from the A_N–A_N tandem reactions can be oxidized into the corresponding aromatic systems. These oxidation products can be regarded as those resulting from the tandem S_N^H–S_N^H reactions [1]. For instance, aromatization of tetrahydro derivatives of thiazolo[4,5-*e*]-1,2,4-triazines with potassium permanganate in acetone proceeds smoothly at room temperature, although it is accompanied by elimination of the *N*-acetyl group (Scheme 15) [62]. Other examples of the S_N^H–S_N^H tandem reactions are given in the book [1].



Scheme 15

There are many other examples where oxidation of cyclo adducts resulting from the A_N–A_N tandem reactions is the only constructive way to avoid degradation of their cyclic structure. For instance, the adducts of 1,2,4-triazinium salts with 1,2-diamines are quite unstable and can be registered by NMR only at low temperatures (from –40 to –20 °C), however, these adducts are easily transformed into 1,2,4-triazino[5,6-*b*]quinoxalines under oxidative conditions (Scheme 16) [1].

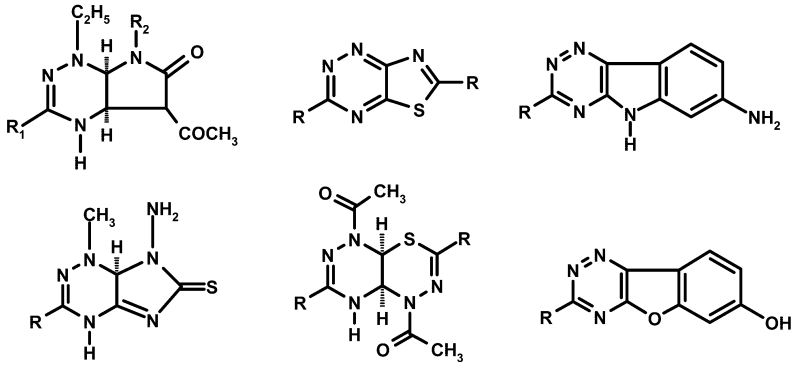


Scheme 16

In the absence of an oxidant, the intermediate hexahydro-1,2,4-triazino[5,6-*b*]-quinoxalines are transformed into quinoxaline by eliminating the corresponding amidrazones [1]. A similar degenerate ring transformation reaction has recently been reported to occur in the reaction of 2,3-dimethyl-5,6-dihydropyrazine with ethylenediamine. It is based on the ability of both C=N bonds of the 1,4-diaza-1,3-diene fragment for a reversible addition and elimination of bifunctional nucleophiles [63].

One more example showing that appropriate oxidative conditions are of great importance for the S_N^H–S_N^H tandem reactions. The cyclo adducts derived from the A_N–A_N reaction of 2-methyl-cinnolinium cation with iminoethers are so unstable that they cannot be registered by NMR, however, in the presence of air oxygen these adducts are converted simultaneously into pyrrolo[2,3-*c*]annelated cinnolines [64]. A very similar cyclization of 6,8-dimethylpyrimido-[4,5-*c*]pyridazine-5,7(6H,8H)-dione with 1,2-diamines proceeding in the presence of silver permanganate (in the complex with pyridine) as oxidant has recently been reported [21,65].

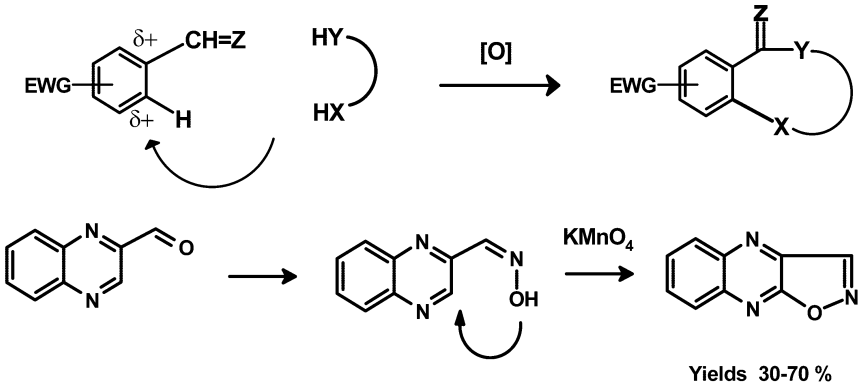
Combination of the A_N and S_N^H strategies with *ipso*-substitution is, undoubtedly, a powerful tool for the construction of various fused heterocyclic systems. In particular, a number of 1,2,4-triazines condensed with five- and six-membered heterocycles have been been obtained using various sequences of the tandem reactions (Scheme 17) [56,62].



Scheme 17

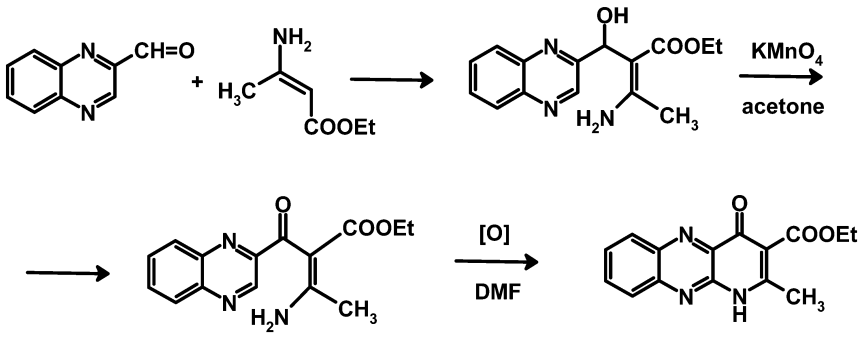
Intramolecular S_N^H reactions

Another approach to condensed azaheterocycles is based on use of intramolecular nucleophilic substitution of hydrogen in combination with a nucleophilic attack at the *exo*-cyclic electrophilic center C=Z (Scheme 18). For instance, this methodology was applied to obtain quinoxalines condensed with five and seven-membered rings from quinoxaline-2-carbaldehyde (Scheme 18) [66–68].



Scheme 18

Condensation of 2-aminoquinoxalines with ketones followed by the intramolecular displacement of hydrogen at C3 in (quinoxalinyl-2)aminovinyl derivatives provides a new approach to pyrrolo[2,3-*b*]- and indolo[2,3-*b*]quinoxalines [69]. Finally, quinoxalinyl-substituted aminovinyl ketone derived from the C-addition of aminovinyl esters to quinoxaline-2-carbaldehyde and oxidation of the adduct with potassium permanganate undergoes the intramolecular S_N^H reaction on reflux in DMF in the presence of air oxygen. This reaction can be regarded as a new synthetic approach for annelation of the pyridine fragment (Scheme 19) [70].



Scheme 19

CONCLUSION

The data surveyed in this review provide evidence that use of the S_N^H methodology in both intramolecular and intermolecular cyclizations is a convenient and powerful tool for the synthesis of condensed heterocyclic systems.

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