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Regioselective competitive synthesis of 3,5-bis(het) aryl pyrrole-2-carboxylates/carbonitriles vs. β-enaminones from β-thioxoketones

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ABSTRACT

An easily adaptable protocol for the synthesis of 3,5-bis(het)aryl-2-carboxylate/nitrile pyrroles and the related 2,3,5-tri(het)aryl-pyrroles with complementary regioselectivity from the corresponding 1,3-bis (het)aryl-1,3-monothio diketones or β-enaminone precursors has been reported. Initially, regioselective base catalyzed condensation of glycine ethyl/methyl esters or aminoacetonitrile with 1,3-bis(het)aryl-1,3-monothio diketones give β-enaminones, which then in-situ undergoes cyclization to form 3,5-bis (het)aryl-2-carboxylate/nitrile pyrroles. The synthesis of 2,3,5 tri substituted pyrroles with full control over 3rd & 5th position on pyrrole ring is the noteworthy feature of this protocol. Although the product yields are moderate to good, the method offers a facile regioselective entry to 2,3,5 trisubstituted pyrroles without the aid of transition metal.

Introduction

Pyrrole is an important five-membered heteroaromatic moiety as its nucleus present in alkaloids [1,2], chlorophyll, porphyrins, haematin, rigidins, storniamide A and corrins. They serves as a building blocks of several pharmaceuticals [3,4] and many drugs contain pyrrole core including Atrovastatin (marketed by Pfizer as Lipitor®), which is one of the best selling drug in the pharmaceutical history used as cholesterol-lowering agent [5,6]. Pyrrole motifs are also ubiquitous in functional materials [7,8], agrochemicals [9] and dyes [10]. The prime source to approach pyrroles relies majorly on chemical synthesis. Traditional cyclocondensation methods such as Paal-Knorr [11], Hantzsch [12], Knorr [13] and Fischer [14] reactions are well-known routes for the synthesis of pyrroles. As the pyrrole ring offers versatile properties, many attempts were made to construct pyrroles, majorly photochemical reactions [15–17], multicomponent reactions [18–20], cycloaddition of alkynes with isocyanides [21–22] and with primary amines [23–25], enolizable aldehydes as 2C [26,27] and 4C donor [28,29]. In addition, Pyrrole-2-carboxylates are very good precursors for the total synthesis of Ningalin A, Lamellarin O and Lukinol A and also for many pharmaceuticals, as a result many attempts have been made for the synthesis of these motif [30–35] including Copper catalysed oxidation/cycloaddition of chalcones and iminodiacetates [36], cyclocondensation of alkenyl sulfones with ethyl isocyanoacetate [37], oxidative cyclization of alkenes with enaminone esters [38] and condensation of nitro-olefins with isocyanoacetates [39].

Despite of being good bioactive nucleus, there are very few reports on the synthesis of 3,5-disubstituted-pyrrole-2-carboxylates [40–48] and only few of these protocols are simple and high yielding, most of them are not efficient. Common problems encountered in the generation of 3,5 disubstituted-pyrrole-2-carboxylates/carbonitriles include regioselectivity, poor yields, metal free conditions, harsh reaction conditions and major concern is the substrate scope, none of the literature reported methods have bis-heterocyclic substitution on 3rd and 5th position on the pyrrole ring. Therefore, a general, catalyst free, regioselective method for assembling 3,5-bis(het)arylpyrrole-2-carboxylates/carbonitriles with broad functionality is highly desirable.

β-thioxoketones are surrogates of 1,3-diketone which have considerably attracted synthetic organic chemists due to their

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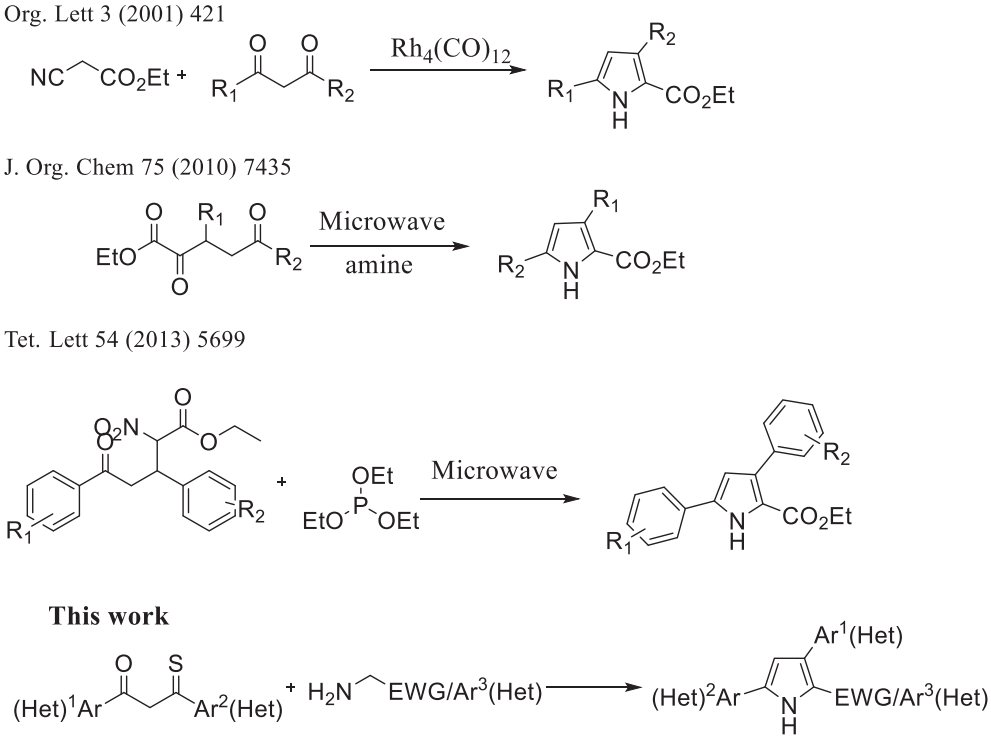
selective reactivity towards electrophilic and nucleophilic reagents, and they are most valuable 3-carbon 1,3-bielectrophilic synthons for five- and six-membered heterocyclic construction [49–51]. However, the synthetic potential of β-thioxoketones as a precursor for regioselective synthesis of functionalized pyrroles is unexplored. We herein exploring the selective reactivity of β-thioxoketones towards nucleophilic amines for the highly regioselective route for the generation of 3,5-bis(het)aryl-pyrrole-2-carboxylates/carbonitriles via cyclocondensation of ethyl glycine esters and aminoacetonitrile in one-pot two-component reaction which overcomes most of the shortcomings of the literature reported methods (Scheme 1).

Results and discussion

The monothio-diketones **1a-m** were integrated in excellent yields via slight modification of the literature methods [52–57] by sodium hydride catalyzed thioacylation of different (het)aryl methyl ketones with dithioesters in DMF. Initially, one-pot protocol was investigated for the regioselective synthesis of 3,5-disubstituted-pyrrole-2-carboxylates directly from monothio-1,3-diketones under varying reaction parameters and solvents. The 1,3-diphenyl-3-thioxopropan-1-one **1a** was opted as a model substrate for optimizing the reaction conditions to construct pyrrole **4a** with the aid of several bases and solvents at different temperature and time intervals (Table 1, Scheme 2). Thus, treatment of **1a** with glycine ethyl ester hydrochloride **2a** and cesium carbonate in DMF at 120 °C (Table 1, Entry 10) was found to be better reaction condition for the synthesis of pyrrole with 75% yield and sodium acetate in ethanol at room temperature is promoted the formation of β-enaminone in excellent yield (Table 1, Entry 12). Unfortunately, we could not able to improve the desired product yield in terms of conversion as judged by TLC with the different bases like triethyl amine, sodium ethoxide, sodium hydride, potassium ter-

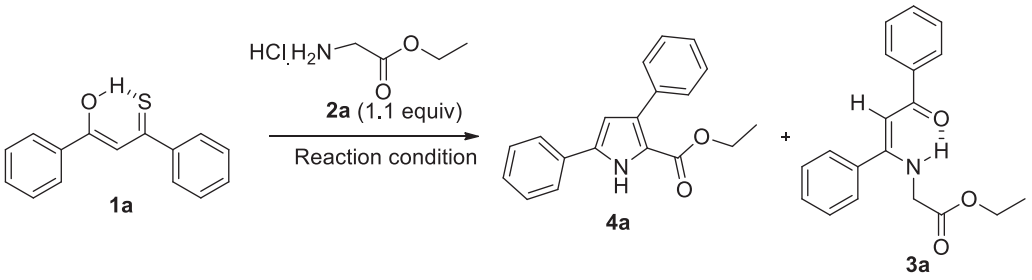


Scheme 1. Different routes to 3,5-disubstituted-pyrrole-2-carboxylates.

Table 1
Optimization condition for the generation of Pyrrole **4a** in one pot condition.

Entry	Base (equiv)	Solvent	T(°C)	t(h)	Yield (%)[a] 3a/4a
1	TEA (2.0)	DMF	80	8	10/0
2	NaOEt (2.0)	Ethanol	90	12	60/0
3	NaH (2.0)	DMF	100	8	30/45
4	NaH (2.5)	DMF	100	8	28/42
5	*t*-BuOK(2.0)	*t*-butanol	80	10	65/trace
6	DBU(2.0)	DMF	120	8	80/trace
7	K_2CO_3(2.0)	Ethanol	90	8	30/30
8	K_2CO_3 (2.0)	DMF	120	6	30/35
9	CS_2CO_3 (2.0)	DMF	120	6	15/45
10	**CS_2CO_3 (2.5)**	**DMF**	**120**	**8**	**8/75**
11	CS_2CO_3 (3.0)	DMF	120	8	7/58
12	NaOAc (1.5)	Ethanol	RT	10	92/0

[a] Isolated Yield



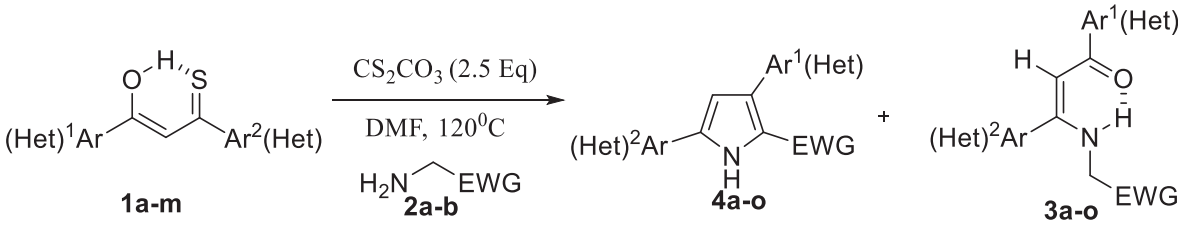
Scheme 2. Synthesis of pyrrole derivative **4a.**

tiarybutoxide, DBU and potassium carbonate under different reaction conditions (Table 1 Entry 1–9). In all the cases we found β-enaminone as a major byproduct.

The generality of the protocol was explored using suitable reaction condition for the preparation of other structurally diversified pyrroles **4a-o** (Scheme 3) from the corresponding monothio-1,3-diketones **1a-m** and ethyl glycine ester/amino acetonitrile **2a-b** as shown in the Table 2. The ethyl glycine ester and amino acetonitrile underwent cyclocondensation smoothly in presence of CS_2CO_3 in DMF at reflux condition. Thus, the monothio-1,3-diketones bearing various electron donating substituents on the different position of aryl ring such as methoxy, chloro, bromo and trimethoxy groups gave the desired product in good yield (Table 2, Entry 2, 6, 8 & 11). Similarly, the monothio-1,3-diketones bearing electron withdrawing substituents on the different position of aryl ring such as nitro, cyano and trifluoromethyl groups have also gave the considerable product yield (Table 2, Entry 4, 5 & 9). On the other hand, the pyrroles bearing 2-furyl/2-thienyl/4-pyridyl groups at 3rd and 5th positions were obtained in appreciable yields from the corresponding monothio-1,3-diketones (Table 2, Entry 3, 7 and 10). Expectedly cyclocondenstaion of amino acetonitrile with monothio-1,3-diketones gave considerably high yields compare to ethyl glycine ester because of more electron withdrawing ability of cyano group, which makes the methylene protons more acidic (Table 2, Entry 12, 13, 14 and 15). It is noteworthy to mention that, it was possible to regioselectively install two different heterocyclic rings at 3rd and 5th position of pyrrole which may display NLO properties because of the push–pull chromophores i.e., electron donating substituents at 3rd & 5th position and electron withdrawing carboxylate/carbonitrile group at 2nd position on pyrrole moiety, which was evident while TLC monitoring showing fluorescent spots. Therefore, three different heterocycles with extended π-conjugation could become useful fluorophores in optoelectronic devices [58–59].

We have synthesized a good number of pyrrole derivatives with most of the possible (Scheme 4) substituents on 2nd, 3rd and 5th positions in one pot operation. The present method showed wide functional group tolerance with product yield ranging from 60 to 80% and also, we found the expected β-enaminone byproduct in lower yields. Having established the regioselective route for 3,5-bis(het)arylpyrrole-2-carboxylates/carbonitriles generation from monothio-1,3-diketones with the moderate product yield, we became interested in isolating β-enaminones intermediate and to perform a base catalysed cyclocondensation reaction stepwise to compare efficiency in terms of product yield. Based on the optimization study for one pot synthesis of pyrroles, it is clear that sodium acetate in ethanol at room temperature is the better reaction condition to prepare β-enaminones (Table 1, Entry 12). Both, ethyl glycine ester and aminoacetonitrile **2a-b** undergo facile condensation reaction with 1,3-monothio-β-diketones **1a-m** to afford corresponding enaminones **3a-o** in good yields (See supplementary file Scheme S1 and Table S1). The chemical shift value of >11.0 corresponds to NH confirmed that the enaminones exists in more stable intra-molecularly hydrogen bonded Z configuration form. In further, to optimize the cyclocondenstaion reaction of β-enaminones to generate pyrroles, enaminone **3b** was selected as a model reactant for the synthesis of pyrrole **4b** in the presence of various bases and solvents in different reaction conditions (Scheme S2 and Table S2). Thus, the condensation of **3b** with different bases like K_2CO_3, NaH and sodium ethoxide doesn't gave the desired product **4b** in appreciable yield (Table S2, Entry 1, 4 & 5). When the reaction was performed in presence of potassium tertiary butoxide, only trace amount of desired product was observed (Table S2, Entry 3). When the condensation reaction was performed with the triethyl amine as a base in DMF solvent, the reaction doesn't yield the product **4b** (Table S2, Entry 6). However, dramatic increase in the yield (70% & 72%) of pyrrole **4b** was observed when cesium carbonate and DBU was used as a base respectively in DMF solvent at 120 °C (Table S2, Entry 2 & 7), probably due to enhanced base strength of cesium carbonate and DBU. Similarly, pyrrole **4b** was obtained in an improved yield, when the reaction was conducted in toluene as solvent and DBU as a base (Table S2, Entry 8). Attempt to further improvement of the product yield of **4b** by varying the reaction time was not successful (Table S2, Entry 9). Interestingly, there was an increase in yields via step-wise procedure but not an appreciable improvement in the formation of pyrrole derivatives **4a-o** on comparison with one-pot protocol (Scheme S3 and Table S3).

Next, we have diverged our interest in substituting aryl/heteroaryl groups on second position of pyrrole ring by using different aryl/heteroaryl methylene amines. Condensation of 2-furfury-



Scheme 3. Synthesis of 3,5-bis(het)arylpyrrole-2-carboxylates/carbonitriles (**4a-o**) from monothio-1,3-diketones.

Table 2
One pot synthesis of 2,3,5-tri substituted pyrroles (**4a-o**) from monothio-1,3-diketones.

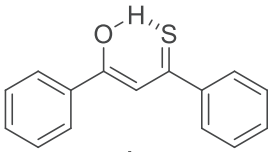
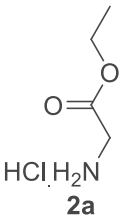
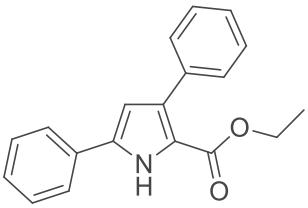
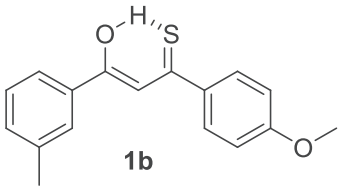
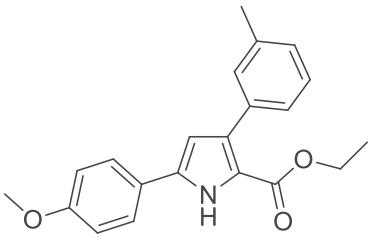
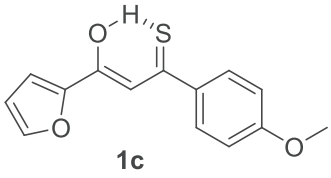
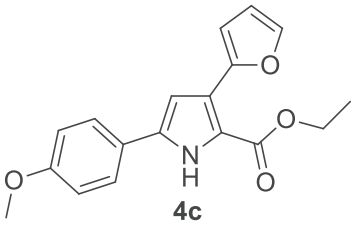
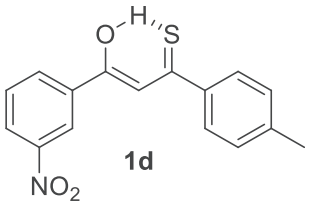
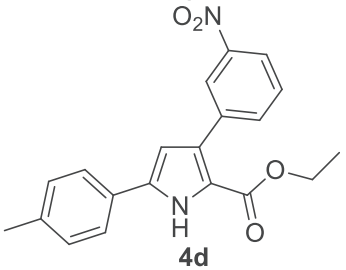
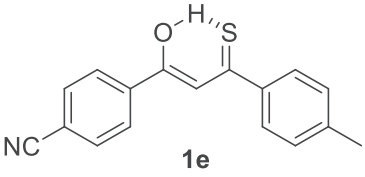
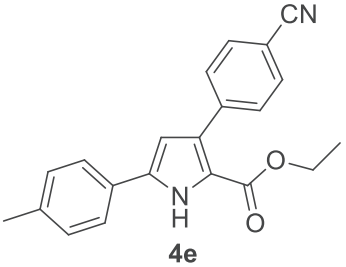
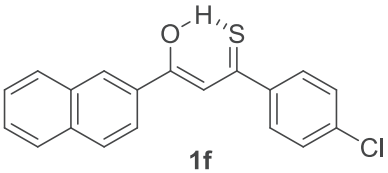
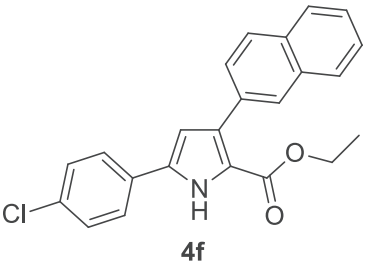
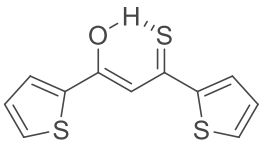
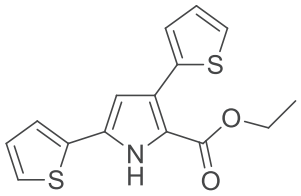
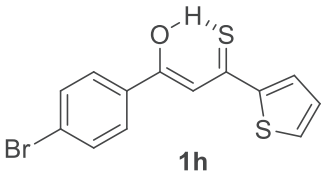
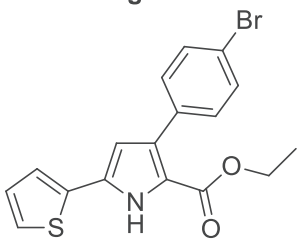
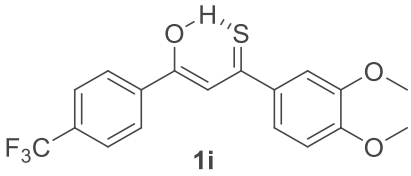
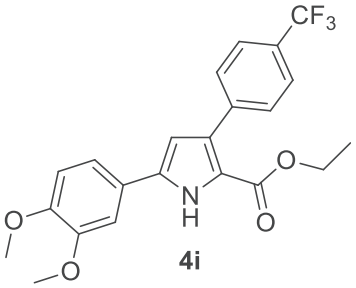
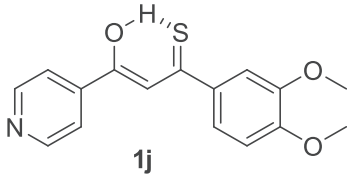
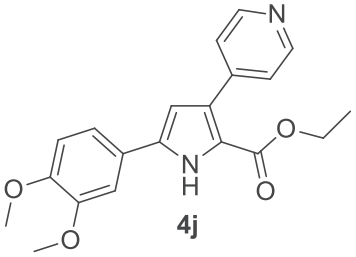
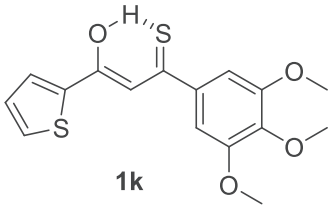
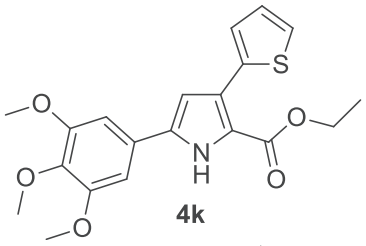
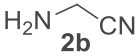
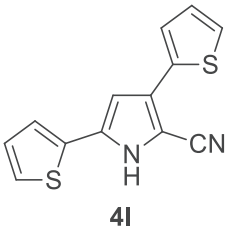
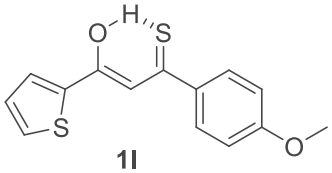
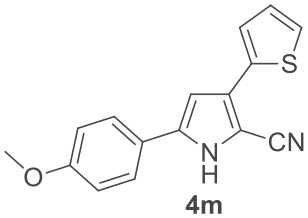
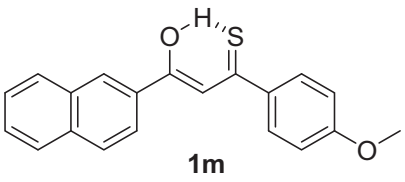
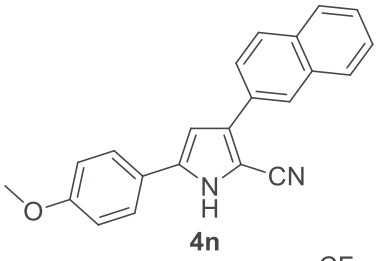
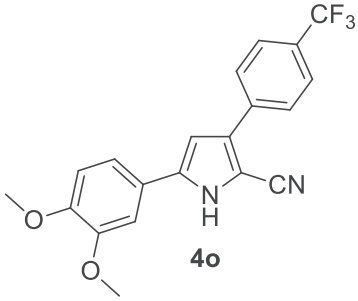
Entry	1a-m	2a-b	4a-o	Yield[a] % 4/3
1	 **1a**	 **2a**	 **4a**	75[40]/8
2	 **1b**	**2a**	 **4b**	74/10
3	 **1c**	**2a**	 **4c**	77/12
4	 **1d**	**2a**	 **4d**	64/15
5	 **1e**	**2a**	 **4e**	68/12
6	 **1f**	**2a**	 **4f**	75/trace

Table 2 (*continued*)

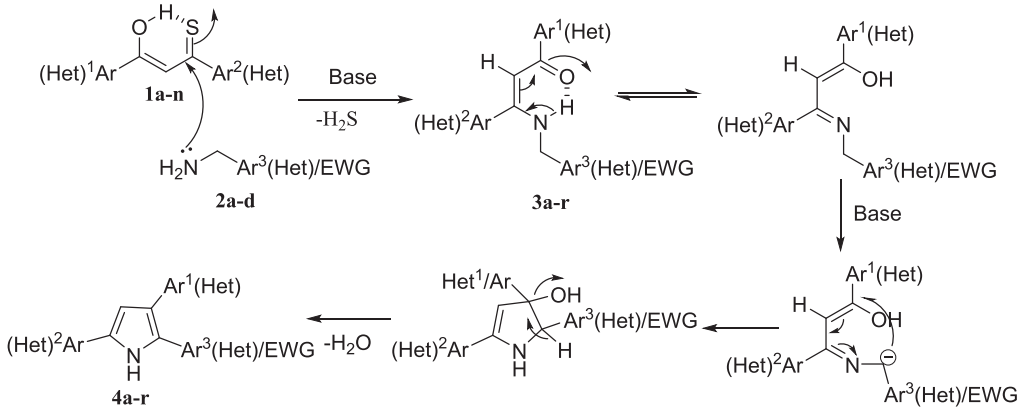
Entry	1a-m	2a-b	4a-o	Yield[a] % 4/3
7	 **1g**	**2a**	 **4g**	65/trace
8	 **1h**	**2a**	 **4h**	73/trace
9	 **1i**	**2a**	 **4i**	69/14
10	 **1j**	**2a**	 **4j**	60/10
11	 **1k**	**2a**	 **4k**	68/trace
12	**1g**	 **2b**	 **4l**	69/trace
13	 **1l**	**2b**	 **4m**	72/10

(*continued on next page*)

Table 2 (*continued*)

Entry	1a-m	2a-b	4a-o	Yield[a] % 4/3
14	 1m	**2b**	 4n	77/trace
15	**1i**	**2b**	 4o	80/trace

[a] Isolated yield.



Scheme 4. Plausible mechanism for the construction of pyrroles.

lamine **2c** and benzylamine **2d** with 1,3-monothio-β-diketones **1a**, **1e** & **1n** underwent smoothly with the aid of sodium acetate in alcohol at room temperature to produce corresponding β-enaminones **3p-r** (Scheme S4). DBU didn't promote the cyclocondensation of these enaminones to respective pyrroles, we have checked these reactions with different bases and sodium hydride was found to be better base in refluxing DMF. Although we were able to get 2,3,5-tri(het)aryl-pyrrole derivatives **4p-r** (Scheme S4, Table S4), but their yields were not much satisfactory.

The plausible mechanism for the construction of pyrroles **4** appears to be simple as depicted in Scheme 4. Ethyl glycine ester, aminoacetonitrile and het/aralkyl amines **2a-d** undergo regiospecific facile condensation reaction with 1,3-monothio-β-diketones **1a-n** to afford corresponding enaminones **3a-r**. Base abstract the methylene proton of the enaminone **3** results in the generation of anion intermediate, which undergoes 1,2-addition to carbonyl group followed by the elimination of water molecule affords pyrroles **4a-r**.

Conclusion

In summary, we have developed a simple, convenient and base catalyzed route to synthesis 2,3,5-tri substituted pyrroles **4a-r** from readily available 1,3-bis(het)aryl-1,3-monothio diketones **1a-n** via one-pot/stepwise regioselective processes. The flexibility and generality of the present protocol allowed us to synthesis regiocontrolled novel 2,3,5 tri(het/aryl/EWG) substituted pyrroles with full control over 2nd, 3rd & 5th position on pyrrole ring. Although the product yields are moderate to good, the method offers a facile regioselective entry to 2,3,5 trisubstituted pyrroles **4a-o** in two component, one-pot operation without isolation of β-enaminone precursors **3a-o** (Scheme 3, Table 2), thus overcoming the limitations of literature reported methods. Easily accessible substrates with approachable reaction conditions allowing quick construction of a structurally diversified pyrrole core in a regiocontrolled manner should demand this protocol as a prime choice for library synthesis in drug discovery research.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.tetlet.2021.153373.

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