Tetrahedron Letters 61 (2020) 152017

Contents lists available at ScienceDirect

Tetrahedron Letters

journal homepage: www.elsevier.com/locate/tetlet

Micellar catalysis enabled synthesis of indolylbenzothiazoles and their functionalization *via* Mn(II)-catalyzed C_2–H amination using pyridones

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ARTICLE INFO

Article history:
Received 30 March 2020
Revised 2 May 2020
Accepted 6 May 2020
Available online 11 May 2020

Keywords:
Micellar catalysis
Indolylbenzothiazoles
Indole C-H amination
Manganese
Green chemistry

ABSTRACT

The sustainable synthesis of indolylbenzothiazloes is reported utilising TPGS-750-M enabled nanomicellar catalysis in water. The reactions do not require additional catalysts and/or oxidants and proceed at room temperature. Subsequently, the indole rings were functionalized to construct novel tris-heterocyclic scaffolds *via* benzothiazole directed Mn(II)-catalyzed C_2–H amination utilizing pyridones as the amine partner.

The introduction of sustainability into chemical processes is an ongoing demand due to the adverse effects of manufacturing processes on the environment [1]. Consequently, the development of new processes for the synthesis and functionalization of carbo/heterocycles is an important area of research. In this regard, the application of water-mediated synthesis [2] and earth-abundant metal catalysis [3] have emerged as viable synthetic tools to fulfil these objectives.

Indoles [4] and benzothiazoles [5] are privileged compounds constituting the building blocks of various pharmaceuticals, agrochemicals, and natural products. This inspired us to merge both of these frameworks in order to exploit their molecular properties *via* a single molecular architecture [6]. Dehydrative cyclocondensation [7] employing appropriate synthons could be a general strategy for the construction of such target molecules; however, performing such reactions in water at room temperature in the absence of additives (catalyst, oxidants, acids/bases) are often challenging, limiting their practical utility (Figure Scheme 1[8]. Similarly, the direct functionalization of indoles *via* C-H amination [9] using earth-abundant catalysis offers significant challenges [10]. Herein, we have described nanomicellar catalysis employing the "designer" surfactant TPGS-750-M for the synthesis of high value indolylbenzothiazoles [11] in water at ambient temperature and their subsequent functionalization to construct novel tris-heterocyclic scaffolds *via* benzothiazole directed indole C_2–H amination under Mn(II)-catalysis.

Pioneering work by the Lipshutz group [12] and others [13] has established that designer surfactants empower catalysis by the aggregation of amphiphiles into nanomicelles leading to facile organic transformations. Keeping this philosophy intact, our study began by evaluating various designer surfactants (PTS, TPGS-750-M, and SPGS-550-M) for the nanomicellar enabled synthesis of 2-(indol-3-yl)benzothiazole **3a** by the treatment of 2-aminothiophenol **1a** with indole-3-carboxaldehyde **2a** in water at room temperature in the absence of an additional oxidant or catalyst. The formation of **3a** was observed in all cases, however, TPGS-750-M gave best result (88% isolated yield) after 12 h (Entry 3, Table 1). Comparative analysis with other commonly utilized surfactants, including anionic surfactants (SDS and *N*-Lauroylsarcosinate Na), non-ionic surfactants (Tritox-X-100 and Transcutol P), and the cationic surfactant (TBAB) showed that TPGS-750-M is superior (Entries 5–9, Table 1). To examine the specific role of water, the model reactions were performed in various organic solvents in the presence of TPGS-750-M. Except for 1,4-dioxane, only trace amounts of **3a** formation were observed in organic solvents, implying the specific role of the water-surfactant system. It further suggests the role of TPGS-750-M as nanomicelles in water [12].

The feasibility of a generalized protocol for the synthesis of different (indol-3-yl)benzothiazoles was examined next. As summarized in Scheme 2, electronically different 2- aminothiolphenols

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https://doi.org/10.1016/j.tetlet.2020.152017
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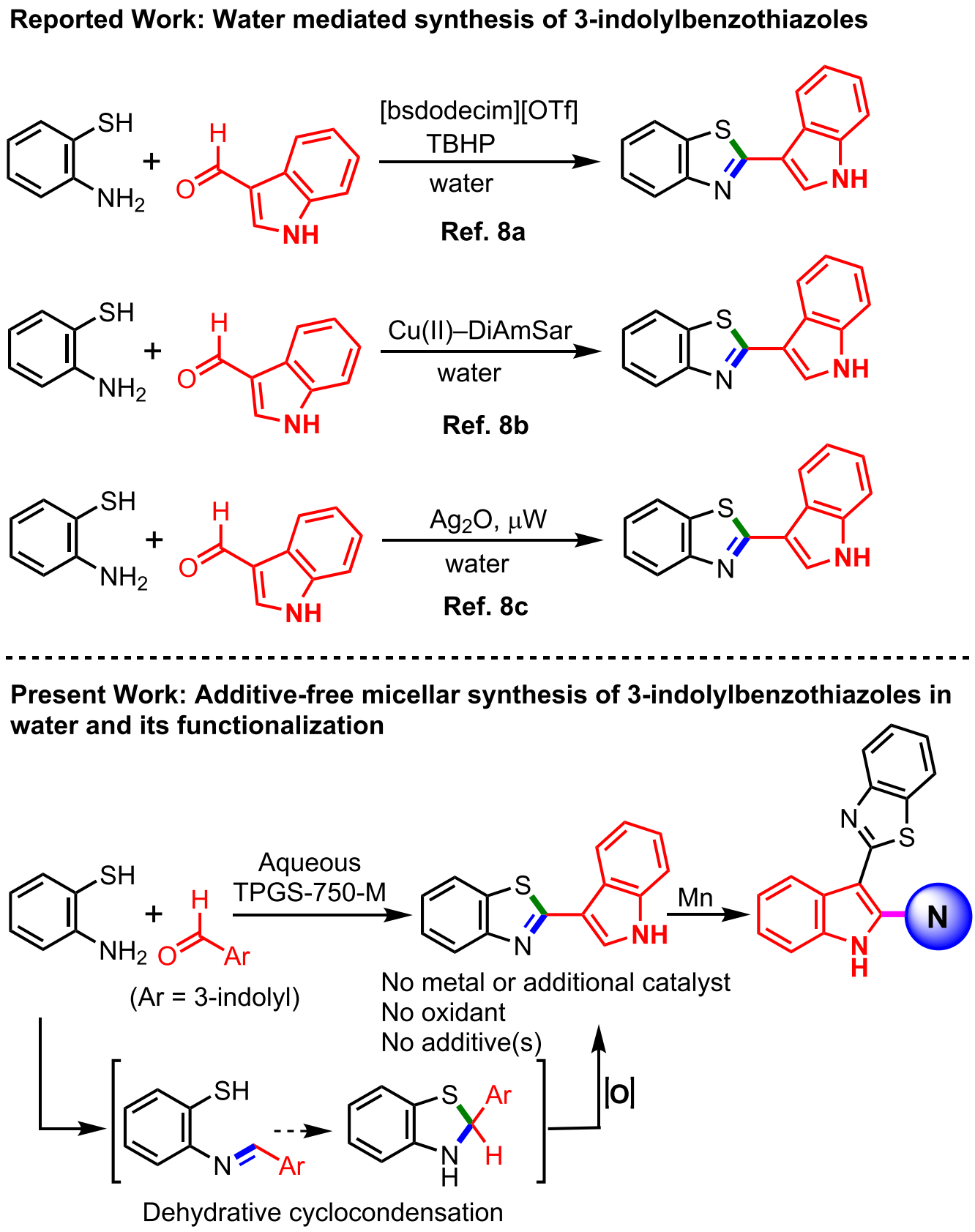
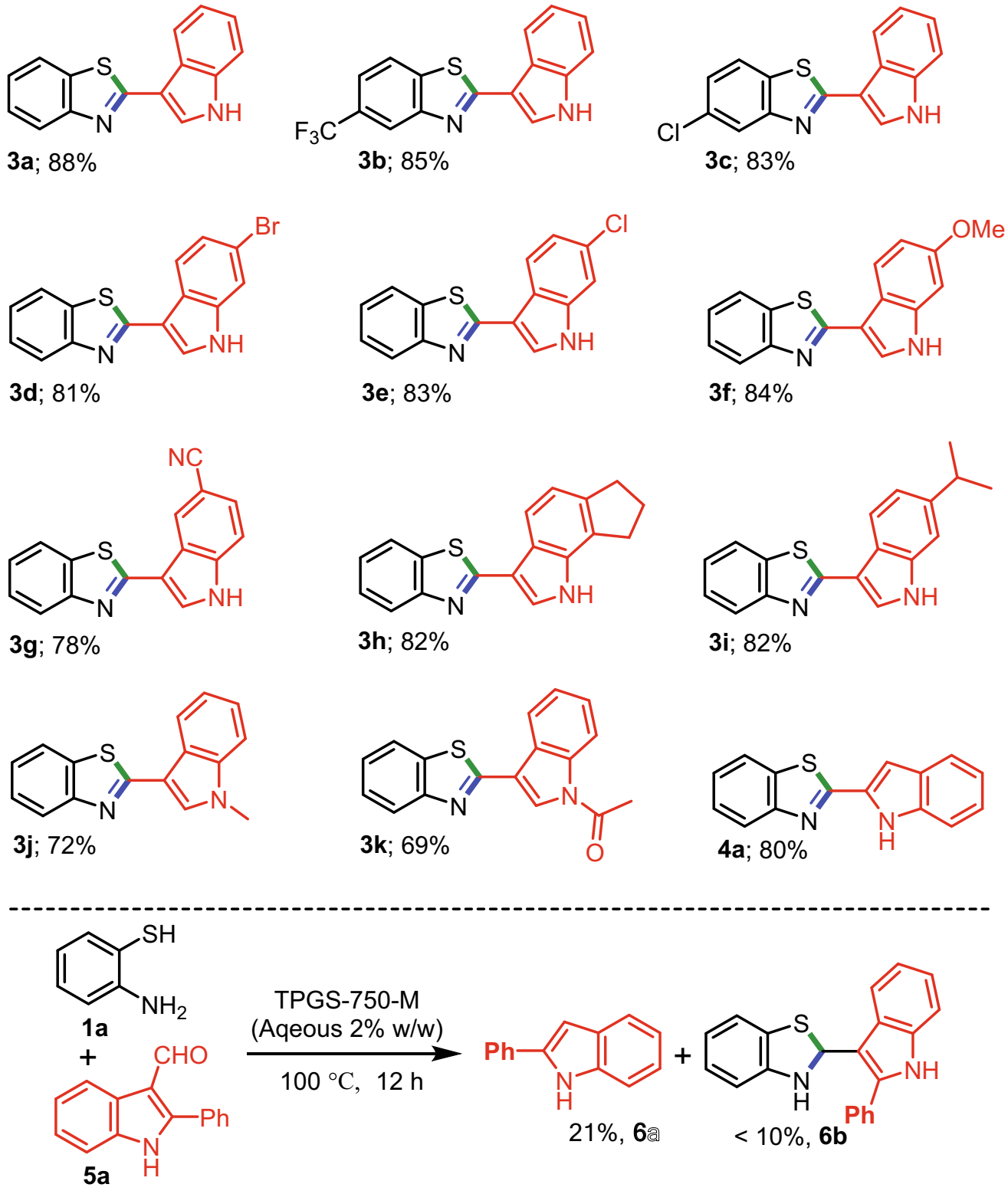


Scheme 1. Micellar catalysis enabled construction of indolyl benzothiazoles and their functionalization *via* C_2–H amination.

and indole-3-carboxaldehydes gave the desired products (**3a**–**3k**) in moderate to high yields (69–88%) at rt. Various functional groups were tolerated, including –Cl, –Br, –OMe, $-CF_3$, –CN, and alkyl. Protected indole-3-caraboxaldehydes (*N*-Me; **3j** and *N*-acetyl; **3 k**) were also compatible with yields of 72% and 79%, respectively. The protocol was compatible with

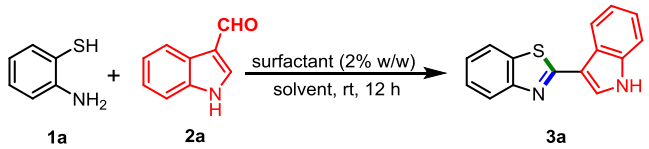


Scheme 2. Optimized reaction conditions for the synthesis of indolylbenzothiazoles: 2-aminothiphenol (0.2 mmol), aldehyde (1.2 equiv.), aq. 2% w/w TPGS-750-M, room temperature. Yields are for isolated, chromatographically purified materials.

indole-2-caraboxaldehyde and gave **4a** in 82% yield. It should be noted that, in the case of 2-phenyl indole-3-carboxaldehdye **5a**, no product formation was observed at rt, and the starting material was recovered. However, under heating (100 °C), an unexpected decarbonylation occurred, resulting in the formation of 2-phenyl indole **6a** (21%) along with the desired product **6b** (8%).

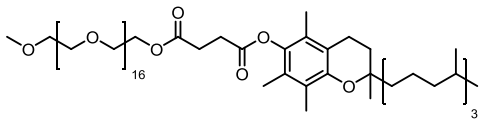
The scalability of the protocol was demonstrated by performing the model reaction on a gram scale (Scheme 3 and ESI). The spent

Table 1
Evaluation of various surfactants for the dehydrative-cyclocondensation-oxidation protocol.



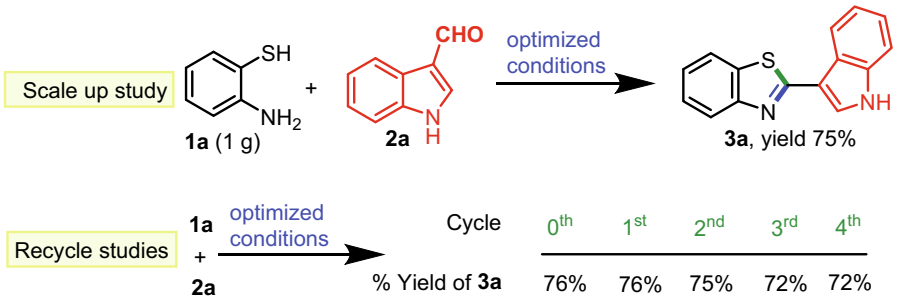
1a 2a 3a

Entry	Surfactant (2% w/w)	Solvent	Yield 3a (%)[b]
1	None	Water	Trace[c]
2	PTS	Water	56
3	TPGS-750-M	Water	88
4	SPGS-550-M	Water	51
5	SDS (SLS)	Water	40
6	*N*-Lauroylsarcosinate Na	Water	37
7	Tritox-X-100	Water	38
8	Transcutol P	Water	36
9	TBAB	Water	37
10	TPGS-750-M	Toluene	Trace[c]
11	TPGS-750-M	1,4-Dioxane	19
12	TPGS-750-M	THF	Trace[c]
13	TPGS-750-M	DMF	Trace[c]
14	TPGS-750-M	DCE	Trace[c]



TPGS-750-M

[a] Reagents and conditions: **1a** (0.2 mmol) was treated with **2a** (0.24 mmol, 1.2 equiv.) in a micellar media (2% w/w) at room temperature for 12 h. [b]Isolated yield of **3a**. [c]Starting **1a** was recovered.



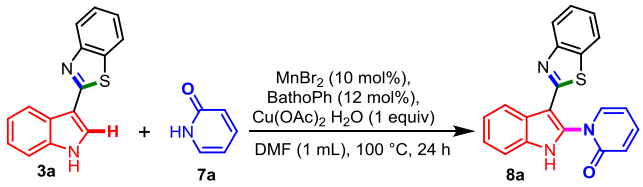
Scheme 3. Gram scale reaction and recycling studies (1 g scale).

water containing TPGS-750-M was reused for four successive reactions without any significant decrease in the yield (Scheme 3 and ESI).

The direct functionalization of indoles represents a step- and atom-economical approach over classical protocols [14]. Although notable progress has been made in the transition metal catalyzed directed C—H functionalization of indoles, [15] the direct functionalization of heteroaryl-substituted indoles with *N*-heteroarenes to construct tris-heterocyclic system is still in its infancy [16]. Taking this as an opportunity, we explored the benzothiazole directed indole C_2–H amination under earth-abundant metal catalysis using pyridones, an important heteroaromatic scaffold found in natural products and pharmaceuticals, [17] as an amine partner. This represents a cooperative functionalization of the *N*-heterocycles to construct tris-heterocyclic systems, hybrid molecules containing indolylbenzothiazole and pyridone motifs [18]. Moreover, 2-pyridones are also useful for the construction of other nitrogen-containing heterocycles such as piperidines, β-lactams, indolizidines, and quinolizidines [19]. Using the model reaction involving **3a** and pyridone **7a**, we explored the Mn-catalysed formation of tris-heterocyclic system **8a**. Optimization of the reaction parameters revealed that the use of $MnBr_2$ (10 mol%), bathophenanthroline (12 mol%), and $Cu(OAc)_2{\cdot}H_2O$ (1 equiv.), in DMF at 100 °C for 24 h gave **8a** (NMR, HRMS, and single XRD; CCDC number: 1963300) in 66% yield (Table 2). The use of other Mn-salts resulted in inferior yields. The utilization of phosphine, bisphosphines, and NHC ligands instead of bathophenanthroline also decreased the yield. Other solvents including toluene, 1,4-dioxane, THF, DMSO, DCE, MeCN, and EtOH (Table 2) were inferior. The addition of either an acid or base inhibited the reaction or gave lower yields.

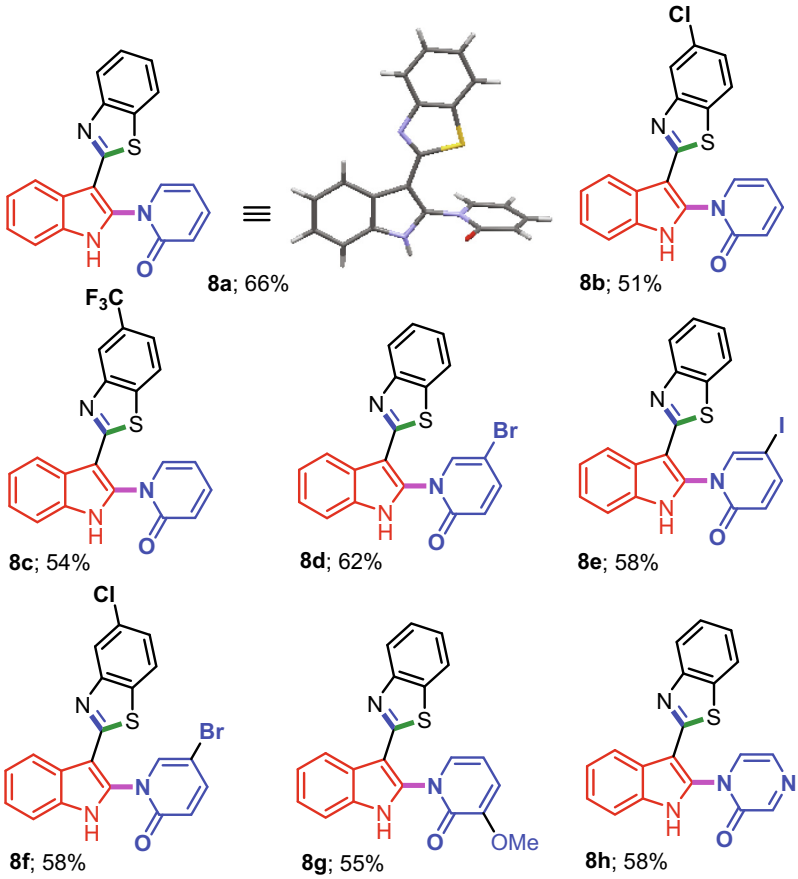
We next examined the scope of this reaction (Scheme 4). The reaction conditions were compatible with substitution on the benzothiazole part (–CF_3, –Cl), however it failed to produce the desired products with substrates with substitution on the indole

Table 2
Variation from the optimized conditions.[a]

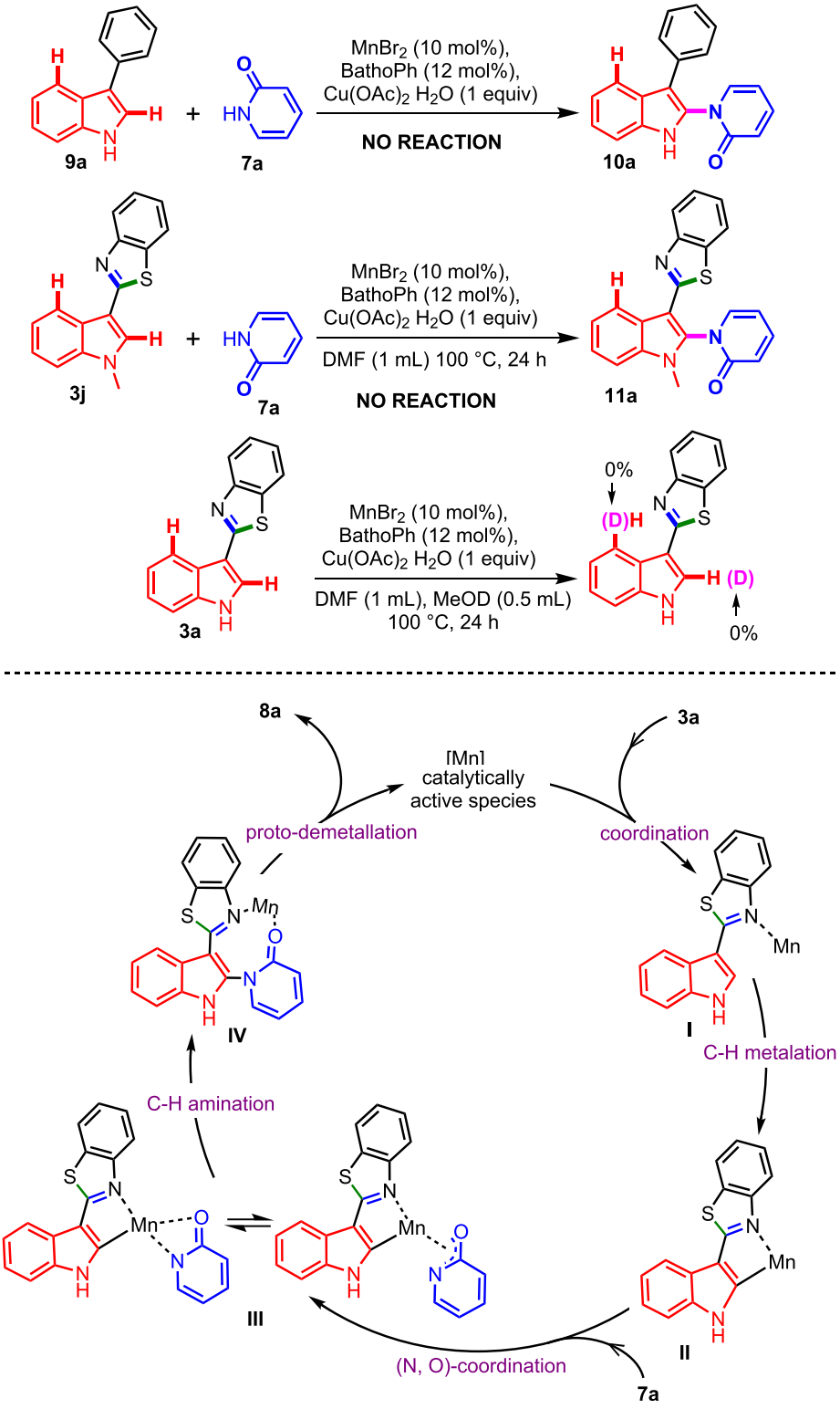


Entry	Variations from the optimized conditions	Yield **8a** (%)[b]
1	None	66
2	MnF_2 instead of $MnBr_2$	12
3	$MnCl_2$ instead of $MnBr_2$	28
4	$MnSO_4$ instead of $MnBr_2$	29
5	$Mn(ClO_4)_2$ instead of $MnBr_2$	42
6	$Mn_2Br(CO)_5$ instead of $MnBr_2$	39
7	$Mn(OAc)_3$ instead of $MnBr_2$	38
8	AgOAc instead of $Cu(OAc)_2{\cdot}H_2O$	0[c]
9	*p*-Benzoquinone instead of $Cu(OAc)_2{\cdot}H_2O$	0[c]
10	$K_2S_2O_8$ instead of $Cu(OAc)_2{\cdot}H_2O$	21
11	PPh_3 instead of Bathophen	41
12	DPPP instead of Bathophen	45
13	RuPhos instead of Bathophen	49
14	XantPhos instead of Bathophen	41
15	Bipy instead of Bathophen	52
16	Ipr.HCl instead of Bathophen	46
17	Toluene instead of DMF	12
18	1,4-Dioxane instead of DMF	15
19	THF instead of DMF	0[c]
20	DMSO instead of DMF	32
21	DCE instead of DMF	52
22	MeCN instead of DMF	24
23	EtOH instead of DMF	28

[a] Reagents and conditions: **3a** (0.2 mmol) was treated with **7a** (0.3 mmol, 1.5 equiv.) under different reaction conditions. [b]Isolated yield of **8a**. [c]Starting **3a** was recovered.



Scheme 4. Synthesis of tris-heterocyclic systems *via* indole C_2-H amination. Yields are for isolated, chromatographically purified materials.



Scheme 5. Preliminary mechanistic investigation for the heteroatom-directed indole C_2-H amination.

component. Using representative examples, we showed the compatibility of electronically different pyridones (–Br, –I, and –OMe), however the reaction failed with –OH and $–NO_2$ containing pyridones. A variety of tautomerizable *N*-heterocycles were examined; however promising results were only obtained in the case of pyrazinones. Other amine partners, including quinazolone, quinolone, benzoxazolone, and morpholine failed to produce the desired products (see ESI for further details).

The reaction of 3-phenyl indole **9a** with **7a** under the optimized conditions did not give the desired product **10a** indicating the hetero-atom directed C_2-H amination process. Similarly, no reaction occurred with *N*-methyl **3j** indicating the possible role of the free

NH during the reaction. Deuterium-scrambling studies indicated the non-reversible nature of the C—H amination process (Scheme 5). Although at this stage we cannot provide a clear mechanistic explanation, based on the limited preliminary studies, the following catalytic cycle was proposed. Following initial coordination of the catalytically active Mn-species with the *N*-heteroatom of **3a**, a five-membered manganacycle(II) is formed. It further reacts with **7a** to afford complex **III**. Then C-H amination takes place followed by the proto-demetallation of **IV** to give the desired product **8a** with concomitant regeneration of the catalytically active Mn-species.

In conclusion, we report a nano-micellar catalytic approach enabled by the designer surfactant TPGS-750-M for the synthesis of 2-(indol-3-yl)benzothiazoles in water at room temperature in the absence of additives (catalyst/oxidant). Using 2-(indol-3-yl) benzothiazole as a starting point, we developed a chemo- and regioselective C—N bond forming reaction for the construction of tris-heterocycles under earth-abundant Mn(II)-catalysis *via* heteroatom directed indole C_2-H bond amination.

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.

Acknowledgements

The authors thank the Department of Pharmaceuticals (DoP), Ministry of Chemical and Fertilizers and NIPER-Ahmedabad for financial support. DK acknowledges Prof. Kiran Kalia, Director NIPER-A for her constant support, encouragement, and motivation. DK also acknowledges the Science and Engineering Research Board, Department of Science and Technology (DST-SERB), Government of India for the award of Ramanujan Fellowship (File No. SB/S2/RJN-135/2017).

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.tetlet.2020.152017.

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