NJC

ROYAL SOCIETY OF CHEMISTRY

PAPER

Cite this: *New J. Chem.*, 2021, **45**, 4632

Metal-free efficient thiolation of C(sp^2) functionalization *via in situ*-generated NHTS for the synthesis of novel sulfenylated 2-aminothiazole and imidazothiazole†

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Received 3rd December 2020, Accepted 4th February 2021

DOI: 10.1039/d0nj05904h

rsc.li/njc

A direct metal-free approach for the synthesis of novel sulfenylated 2-aminothiazole and imidazothiazole derivatives at room temperature is reported *via* an *in situ*-generated electrophilic thiolating agent. The present protocol provides mild and selective access for the insertion of C–S bond functionalization with good yield. The mechanistic path was justified *via* density functional theory (DFT) calculations, which explore the role of the solvent in the reaction mechanism.

Introduction

The prevalent occurrence of organosulfur compounds in vital biological systems, drug architectures and natural products present themselves as versatile scaffolds in organic chemistry, medicinal chemistry and materials chemistry.[1–5] They constitute an active portion of commercially available drugs.[6,7] These consequences have led to an unending quest for a capable catalytic system, comprising a blend of carbon-sulfur bonds to create organosulfur compounds.[8–16] The majority of reported transformations for C–S bond coupling includes the synthesis of diaryl sulfides using imidazoheterocycles,[17–20] indoles[21–25] or aryl halides[26–30] by reaction with thiols or thiones. Several catalytic systems utilized for the cross dehydrogenative coupling reaction (CDC) of the C–S bond include the use of transition metals,[31–36] elemental sulfur,[37–39] and iodine.[40–44] Amongst these protocols, those capable of encountering direct metal-free regioselective C–S bond coupling in bifunctional motifs for the selective synthesis of heterocyclic organosulfur compounds are highly desirable.[45–52] Moreover, among numerous catalytic systems reported for the synthesis of organosulfur compounds, the use of *N*-halosuccinimides was proven to be a highly useful approach;[53–59] however, *N*-halosuccinimides have a general tendency to oxidise secondary alcohols to their corresponding ketones.[60,61] In recent years, the use of *N*-sulfanylsuccinimides for the direct sulfenylation of aromatic and heteroaromatic C–H bonds has become an interesting strategy.[62–73] Very few reports are available for the synthesis of catechol thioethers.[75–77] However, the selective synthesis of organosulfur compounds has not been reported hitherto *via in situ*-genarated *N*-(heteroarylthio)-succinimide (NHTS), by utilizing *N*-halosuccinimide and heterocyclic thiols such as 1H-benzo[*d*]imidazole-2-thiol, benzo[*d*]oxazole-2-thiol and 5-(pyridin-4-yl)-1,3,4-oxadiazole-2-thiol. The use of these heterocyclic thiols may impart advantages in the areas of small molecule syntheses as well as pharmaceuticals as imidazothiazole and thiazoles are considered to possess a broad spectrum of biological activity.[79,80] Consequently, the selective C-5 electrophilic sulfenylation of pseudo aromatic imidazothiazoles with secondary alcohols may provide a beneficial synthetic route for medicinal chemistry research. Jie *et al.* have reported the organocatalytic sulfenylation of β-naphthols using *N*-(arylthio)succinimide as the sulfur source, and they have observed that the dearomatization of β-naphthols takes place with the oxidation of an alcoholic group to a ketone (Scheme 1).[78]

Nevertheless, alcohols also possess the propensity to react with thiols to generate thioethers in the presence of certain catalytic systems.[81–86] These annotations and our previous study regarding the synthesis of bioactive compounds[87–89] have provoked us to focus on the development of a new catalytic system for the selective C(sp^2)–H bond thiolation of 2-aminothiazoles and imidazothiazoles using heterocyclic thiols and *N*-halosuccinimide.

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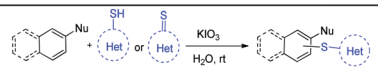
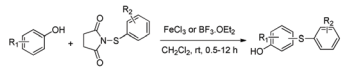
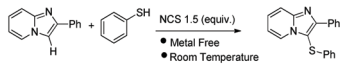
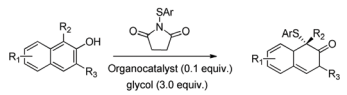
† Electronic supplementary information (ESI) available. See DOI: 10.1039/d0nj05904h

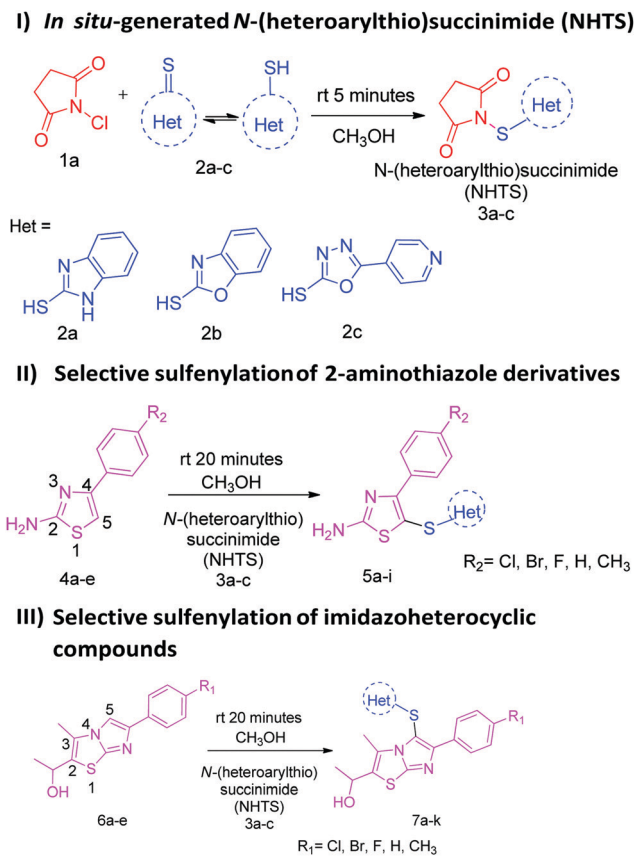
Sr. No.	Scheme	Time	Ref. No.
a)		15 h	72
b)		0.5 h	73
c)		30 min	74
d)		30 min	53
e)		75 h	78

Scheme 1 Earlier approaches for C–S bond coupling.

Results and discussion

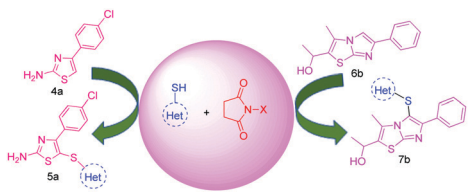
The hypothesized synthetic route commences with the reaction of *N*-chlorosuccinimide with aromatic thiophenols, as predicted by previous literature.[53] We have further demonstrated that further reaction of *N*-chloro-thiols smoothly allows the formation of C–S bonds.[53,74] When this halogenation-thiolation tandem strategy was first implemented, the formation of the product took place with poor yield and prolonged time of 10 h.

When we carried out the same reaction taking 1 equiv. of *N*-chlorosuccinimide with 1 equiv. of 1H-benzo[*d*]imidazole-2(3H)-thione using methanol as the solvent, we came across a curious observation that instead of introducing chlorination in heterocyclic thione, the construction of *in situ*-generated NHTS (Scheme 2I) takes place within 5 min by stirring at room temperature. A plausible reason for this observation may be that the resonance stability of heterocyclic thiols eventually supports the *in situ* generation of NHTS. When the same strategy was employed in the case of aromatic thiols, the generation of *N*-(arylthio)succinimide does not take place to such an extent. We investigated the utilization of NHTS for the sulfenyllation of substituted 4-phenylthiazol-2-amine **4a–e** (Scheme 2II) and 1-(3-methyl-6-phenylimidazo[2,1-*b*]thiazol-2-yl)ethanol **6a–e** (Scheme 2III) in the same reaction pot. The starting reactant **6a–e** was obtained by reducing 1-(5-((1H-benzo[*d*]imidazol-2-yl)thio)-6-(4-chlorophenyl)-3-methylimidazo[2,1-*b*]thiazol-2-yl)ethenone with $NaBH_4$.[90] For exploring the optimal reaction conditions, *N*-halosuccinimide and solvents were examined using 4-(4-chlorophenyl)thiazol-2-amine or 1-(3-methyl-6-phenylimidazo[2,1-*b*]thiazol-2-yl)ethanol as the standard reactant (Table 1). Initially, *N*-chlorosuccinimide (NCS) 1 equiv. was employed under dichloromethane (DCM) as the solvent yield was observed to decrease (Table 1, entry 1). When the amount of NCS was increased, the yield improved (Table 1, entry 2). Further improvement of the yield was seen when 1.5 equiv. of NCS was employed with 2 equiv. of the reactant (Table 1, entry 3). Optimized reaction conditions were found when methanol was used as the solvent with 1.5 equiv. of NCS and 2 equiv. of reactant



Scheme 2 Present work.

Table 1 Optimal reaction condition[a]



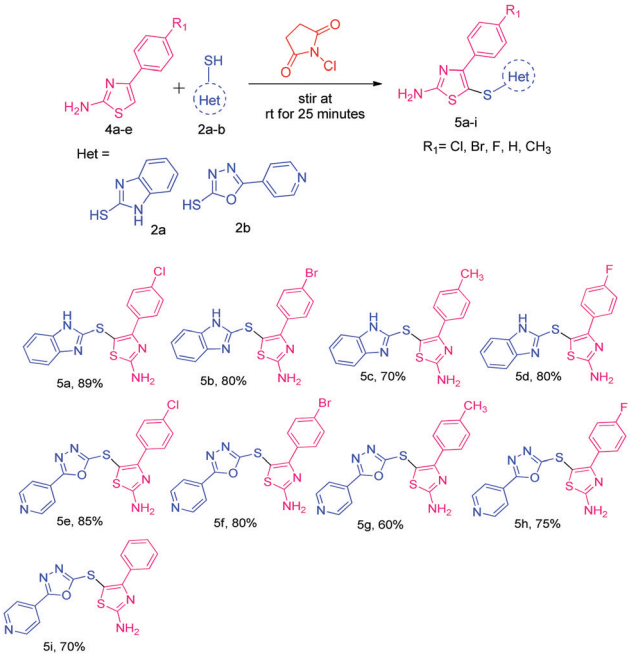
Entry	NXS equiv.	Heterocyclic thiols equiv.	Solvent (3 ml)	Yield[b] (%)
1	NCS (1.0)	1	DCM	43
2	NCS (1.5)	1	DCM	47
3	NCS (1.5)	2	DCM	52
4	NCS (1.5)	1	CH_3OH	71
5	NCS (2.0)	1	CH_3OH	64
6	**NCS (1.5)**	**2**	**CH_3OH**	**89**
7	NCS (1.5)	2	CH_3COOH	34
8	NCS (1.5)	2	Toluene	41
9	NCS (1.5)	2	CH_3CN	44
10	NCS (1.5)	2	DMF	31
11	NCS (1.5)	2	DMSO	51
12	NIS (1.5)	2	CH_3OH	58
13	NBS (1.5)	2	CH_3OH	78
14	—	2	CH_3OH	NR[c]
15	NCS (1.5)	2	No solvent	Trace

[a] Reaction conditions: *N*-halosuccinimide, heterocyclic thiols are stirred for 5 min at room temperature first and then reactant **4a** or **6a** was added and reaction mass was further stirred for next 20 min at room temperature. [b] Isolated yield. [c] NR = No reaction.

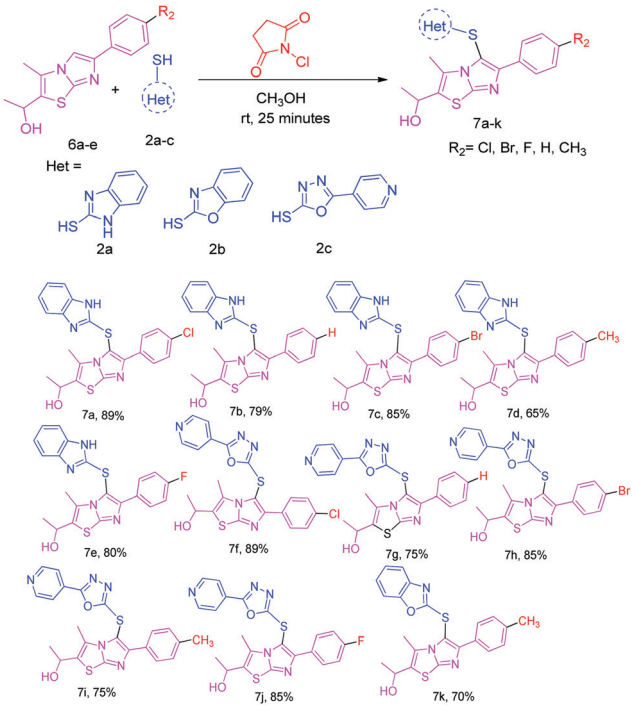
(Table 1, entry 6). The as-synthesized product was washed with cold ethanol. Column chromatography was not needed for purification. Further variation in the amount of the reactant and/or NCS using methanol as the solvent was shown to decrease the yield (Table 1, entries 7–11). The implementation of *N*-iodosuccinimide or *N*-bromosuccinimide was observed to diminish the yield (Table 1, entries 12 and 13). In absence of *N*-halosuccinimide the reaction did not proceed (Table 1, entry 14). Furthermore, the reaction was not observed under solvent-free conditions (Table 1, entry 15).

With this optimal set of reaction conditions (Table 1, entry 6), we proceeded to investigate the sulfenylation of 2-aminothiazole derivatives. While exploring the effects of the substrate scope of 2-aminothiazole derivatives, the unsubstituted 2-aminothiazole ring at the C-4 position was unable to furnish the product. Amongst the substituted 2-aminothiazole derivatives, those derivatives possessing electron withdrawing substituents at the para position of the aromatic ring successfully furnished the product with satisfactory yield (Scheme 3). Electron-donating groups were found to retard the yield (Scheme 3), while substituents at the meta position were unable to furnish the product.

Interestingly, the method achieves the selective C-5 sulfenylation of 2-aminothiazole derivatives, and no reactions were observed to give a nuclear sulfenylation product. We further proceeded to investigate numerous 1-(3-methyl-6-phenylimidazo[2,1-*b*]thiazol-2-yl)ethanol derivatives for their sulfenylation (Scheme 4). Subsequently, adding 1 equiv. of reactant **6a** in the same pot and stirring for 20 min at ambient temperature affords the synthesis of final products. Results obtained



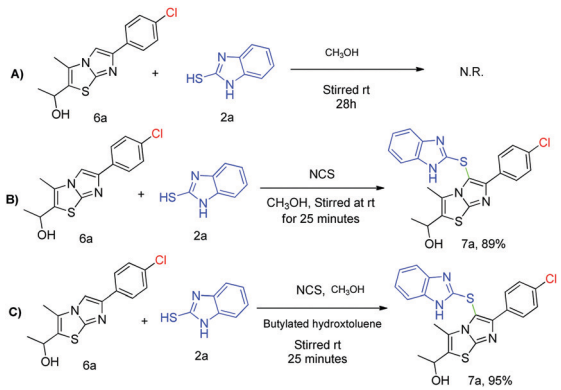
Scheme 3 Scope of substrate: variation of substituent on 2 aminothiazole[a,b]. [a]Reaction conditions: NCS (2.0 mmol) heterocyclic thiol (1.0 mmol), substituted 2-aminothiazole **4a–e** (1 mmol), CH_3OH (3 mL), reaction time (24–25 min). [b]Isolated yield.



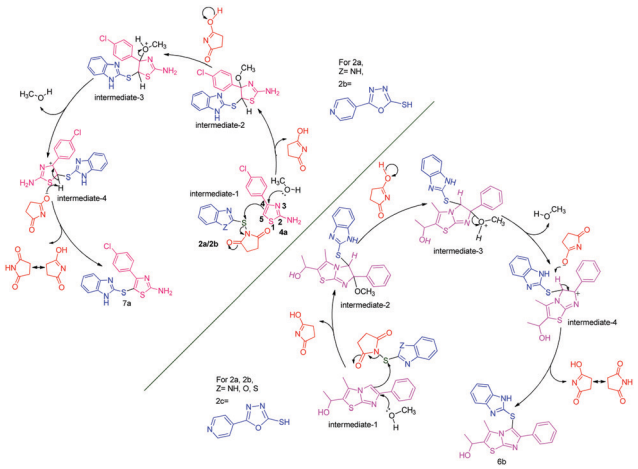
Scheme 4 Variation of substituent on imidazothiazole[a,b]. [a]Reaction conditions: NCS (2.0 mmol) heterocyclic thiol (1.0 mmol), substituted imidazothiazole **6a–e** (1 mmol), CH_3OH (3 mL), reaction time (24–25 min). [b]Isolated yield.

demonstrate that derivatives with electron-withdrawing groups on the aromatic ring at the para position provide the product with a satisfactory yield (Scheme 4), whereas with electron-donating groups such as the methyl group at the same position tend to decrease the yield of the product (Scheme 4). Electron-releasing substituents failed as exemplified by the reactant possessing the methoxy group at the same position. Variation in the heterocyclic thiol was not seen to alter the yield of the product. Interestingly, in each derivative selective sulfenylation was seen to take place at the C-5 position of imidazothiazole derivatives regardless of the pendant alcohol. Also, no derivatives were seen to give sulfenylation on an aromatic ring. The probable mechanistic path was further studied by taking other mechanistic pathways into account (Scheme 5). It came to our attention that the reaction does not proceed in absence of any catalyst or co-reagent after 28 h (Scheme 5A). The implementation of *in situ*-generated (NHTS) results decrease reaction time to 25 min, yielding 89% (Scheme 5B). When same protocol was carried out in presence of the radical quenching agent butylated hydroxtoluene BHT (2 equiv.), we were still able to get 95% yield (Scheme 5c), ruling out the possibility of free radical mechanistic path. Considering the observations of controlled experiments, optimized reaction conditions and (DFT) studies, the tentative mechanism of the reaction is given in (Scheme 6). The 4-(4-chlorophenyl)thiazol-2-amine (**4a**) derivative has been taken as the representative of all the derivatives.

The mechanistic path was studied by DFT calculations, as illustrated in (Scheme 6). The observations in Table 1 and



Scheme 5 Screening experiments.



Scheme 6 Plausible mechanistic path for the thiolation of substituted imidazothiazole and 2-aminothiazole.

Scheme 5 indicating the role of CH_3OH in the reaction is not only limited to the solvent effect but it also takes part in the mechanism as a reagent.[91–94] Initially, the reaction between (**4a**) and (NHTS) in presence of CH_3OH leads to an intermediate-1 with bond formation between C-5 of (**4a**) and -S of (NHTS). Here, methanol is found to be bonded at C-4 that induces the electronegativity of C-5 to facilitate C–S bonding. The release of succinimide is found during the formation of intermediate-2 with -H transfer from methanol. Subsequently, methanolate abstracts -H from succinimide (intermediate-3) and leaves to form intermediate-4. The disappearance of profound IR stretching frequency around 3210 cm^{-1} (Table S1, ESI†) indicates the formation of product (**5a**).

Conclusions

In summary, we have developed a metal-free, mild and selective protocol for the synthesis of novel sulfenylated 2-aminothiazoles and imidazothiazoles *via in situ*-generated (NHTS). Our study exemplifies that NHTS is acting as a co-reagent in the current procedure. The method adopted ensures chemoselectivity towards the C–H bond functionalisation in the presence of secondary alcohol in imidazothiazole, setting up the application of NHTS for the pharmaceutical and agrochemical arenas.

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

Authors S. N. Kadam (F. No. 38/07/14) and A. N. Ambhore (F. No. 36/33/14) are grateful to the UGC for providing teacher fellowship. R. D. K. acknowledge S. P. P. U. Pune for aspire research grant.

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