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Iodine-DMSO catalyzed chemoselective oxidative aromatization and deallylation, nondeallylation of aryl allyl ether of tetrahydro- β -carboline

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Abstract

We have developed a simple method for the chemoselective aromatization of tetrahydro- β -carboline with selective nondeallylation *O*-allyl groups in the presence of iodine (100 mol %) in dimethyl sulfoxide/H₂O₂. A convergent approach toward the oxidative aromatization with selective deallylation (deprotection) of *O*-allyl-tetrahydro- β -carboline using iodine in dimethyl sulfoxide/HCl has been described. The present protocol contains cheap catalyst, easy work up, normal reaction conditions, and high selectivity.

1 | INTRODUCTION

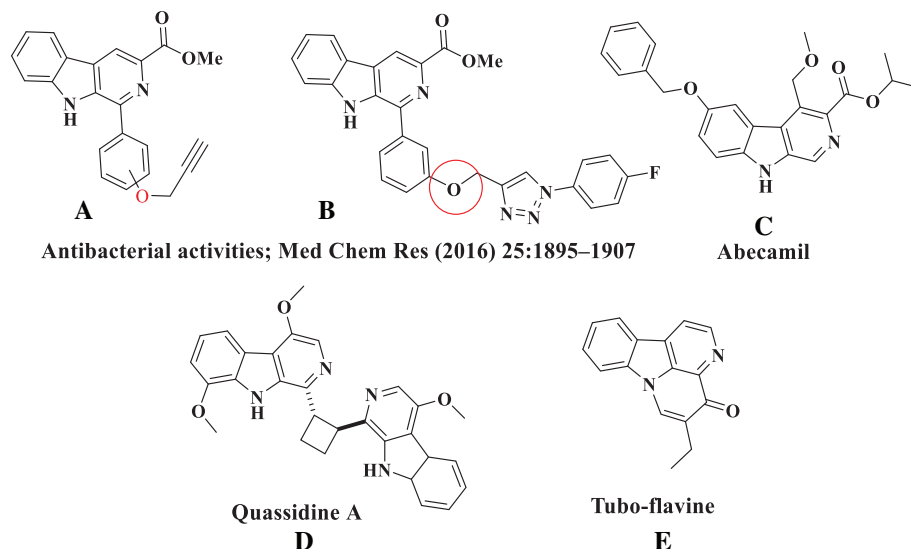
In organic synthesis, functional group transformation, protection, and selective deprotection have their significant importance.^[1] For the protection of phenols, alcohols, acids, and amine functional groups, the allyl group is commonly used as a protecting group. The stability of allyl group in the acidic and basic conditions is an important factor during organic transformation.^[2] As per literature, various methods are found for the selective deallylation and nondeallylation of the *O*-allyl group in the organic transformation.^[3,4] The protection and deprotection strategies must be performed in the vicinity of a variety of other functional groups.^[5] The deprotection of allyl has been achieved by using various reagents such as metal salts (Pd, Rh, Ir, Ru, Cu, Zn) or their expensive complexes^[6] DDQ,^[7] ZnCl₂,^[8] NBS,^[9] OsO₄,^[10] *p*-TSA,^[11] SmI₂,^[12] Pd(PPh₃)₄ (mol %) with K₂CO₃ and molecular iodine.^[13] The copper boryl reagents enable the selective cleavage of aryl allyl ethers and selective nondeprotection of allyl ester, but such transformation needed reaction activator.^[14]

Therefore, it is desirable to develop new methodologies that afford high yielding with nondeallylation and oxidative aromatization/dehydrogenation will occur under mild reaction conditions. β -carbolines are important

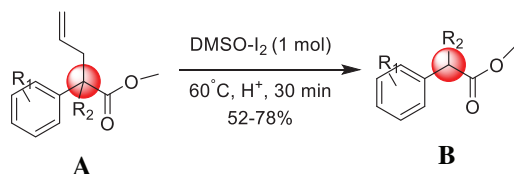
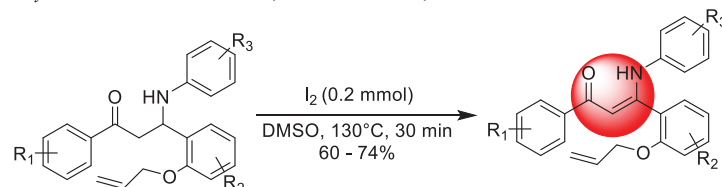
heterocyclic compounds with a wide range of biological activity^[15] (Figure 1).

Since the last decade, our research group explores the iodine chemistry in the novel organic transformation, always useful to develop a cost-effective, safe, greener new synthesis approach for organic synthesis.^[16] The milder acidic nature of iodine facilitates its usage in organic synthesis. Previously, we have reported the iodine-mediated useful chemoselective transformation, such as oxidative chemoselective dehydrogenation, aromatization, and their synthesis utility for natural product synthesis.^[17–18] *O*-allyl-deallylation for a 2'-allyloxy chalcones is followed by oxidative cyclizations to flavones.^[19] The substoichiometric amount of I₂ in DMSO has been used for the cascade synthesis of flavones.^[20] The iodine in DMSO at 130 °C, it prefers deprotection of allyl group carboxylic ester^[21] and direct C-deallylation of allyl group from the active methylene position^[13] along with chemoselective oxidative dehydrogenation of allyloxy substituted β -anilino-dihydro-chalcones to β -anilino-chalcones^[22] was preferred reaction over deallylation (Figure 2). The Significant biological properties of β -carboline makes it synthetically important Unit.^[23]

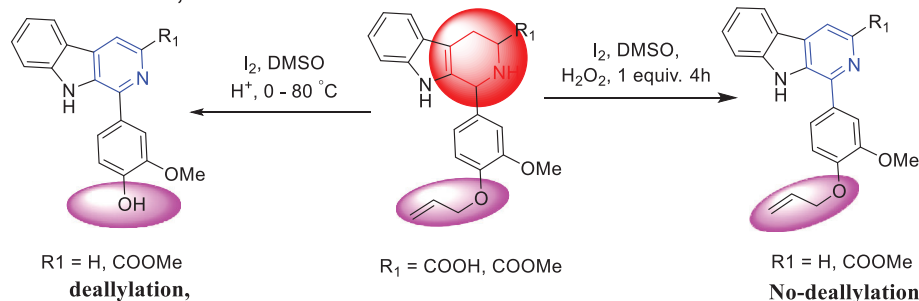
Considering the difficulties in the coinciding deallylation or nondeallylation accompanied by dehydrogenation/aromatization of *O*-allyl TH β C with low

FIGURE 1 Bioactive β -carboline

Previous Literature Report ;

*Synthetic Communications*, 43: 1955–1963, 2013FIGURE 2 Synthesis of O-allyl- β -carboline and β -carboline phenol; (deallylation occurs with the addition of H^+)

Present Work ;



temperature, the development of an efficient and environmentally friendly method is highly desirable.

Herein, we wish to report a chemoselective nondeallylation followed by aromatization of O-allyl TH β C using iodine in DMSO and external oxidant H_2O_2 . Also, we have disclosed the consecutive deallylation followed by aromatization of O-allyl-TH β C using molecular iodine in DMSO/HCl. As per the literature search, to the best of our knowledge, there are no such reports available for

the consecutive aromatization with nondeallylation of O-allyl aryl ether (TH β C).

2 | RESULT AND DISCUSSION

The O-allyl tetrahydro- β -carboline **3a** was conveniently prepared by the treatment of a variety of O-allyl aldehyde **2** with tryptophan/tryptamine/tryptophan methyl ester

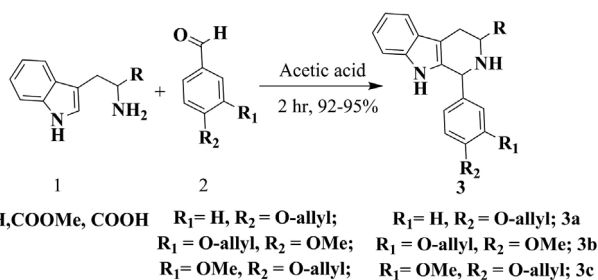
in acetic acid by Pictet Spengler condensation to achieve the required substrate (Scheme 1).

To study the comparative effectiveness and to overcome the limitation of our previously reported protocol, the development of a new methodology for aromatization followed by nondeallylation *O*-allyl is essential. Recently, we have been reported I_2 -DMSO-/H₂O₂-mediated aromatization of TH β C to β -carboline.^[17,18] Herein, we have proposed to apply a similar reaction condition on *O*-allyl-substituted TH β C.

Therefore, we have explored the amount of iodine-DMSO/H₂O₂ required to catalyze the reaction with methyl 1-(4-[allyloxy]phenyl)-2,3,4,9-tetrahydro-1H-pyrido[3,4-*b*]indole-3-carboxylate **3g** as a model substrate. Initially, we have applied 10 mol%, 20 mol% and 20 mol% iodine in DMSO and external oxidant H₂O₂ on *O*-allyl tetrahydro- β -carboline methyl ester; no significant changes were observed at room temperature (Table 1,

entry 1, 2, 3). While on increasing the temperature from 60 °C to 80 °C, the aromatic product was isolated with the *O*-allyl group intact with the OH group with a 50% yield (Table 1, entry 4, 5). The aromatization with nondeallylation formation was confirmed by spectral analysis. In the ¹H NMR spectra, the peak at 11.94 (s, 1H) corresponding to N-H proton. The allyl group shows the characteristic peak pattern 6.24–6.00 (m, 1H), 5.47 (d, *J* = 15.8 Hz, 1H), 5.32 (d, *J* = 9.3 Hz, 1H), 4.71 (d, *J* = 5.1 Hz, 2H) and singlet at 3.95 for OMe group. In ¹³CNMR, spectra showed the 166.36 corresponding to the carbonyl of the COOMe group. From the spectral analysis, it conforms to the product methyl 1-(4-[allyloxy]phenyl)-9H-pyrido [3,4-*b*] indole-3-carboxylate **6d** formed with nondeallylation. Next, we moved to increase the yield of the product; therefore, we have used 50 mol% and 100 mol% of the iodine in DMSO/H₂O₂ at 90 °C, with H₂O₂; it was observed that the aromatization of *O*-allyl-tetrahydro- β -carboline methyl ester obtained with very good yield and nondeallylation (Table 1, entry 9).

The time required for completion of the reaction was longer when 25 mol% of iodine was used. However, 50 and 100 mol% iodine were found as adequate quantities for the oxidative aromatization of *O*-allyl-TH β C with nondeallylation with less reaction time (Table 1, entry 7, 9). By this result, we have decided to use 100 mol% iodine in DMSO/H₂O₂ as the best reaction condition for aromatization of *O*-allyl-TH β C. To understand the role of DMSO solvent and H₂O₂ external oxidant, we performed the reaction on the same substrate in DMSO solvent, and



SCHEME 1 Preparation of *O*-allyl tetrahydro- β -carboline

TABLE 1 Optimization of reaction condition

Sr/no	Iodine Mol%	Oxidant	Temperature	Time/h	Yield 6	Yield 5
1	10	-	RT	24	no	-
2	20	H ₂ O ₂	RT	24	no	-
3	20	H ₂ O ₂	60	24	no	-
4	25	H ₂ O ₂	80	12	45	-
5	25	H ₂ O ₂	90	8	65	-
6	30	H ₂ O ₂	90	8	65	-
7	50	H ₂ O ₂	90	8	75	-
8	75	H ₂ O ₂	90	5	77	-
9	100	H ₂ O ₂	90	4	85	-
10	25	HCl	90	3		75 ^[19]
11	50	HCl	100	3		78 ^[19]

Note: Use of DMSO in HCl at higher temperature reaction get decomposed, and dellylation occurs, and without the use of HCl, no good result observed.

TABLE 2 Synthesis of O-allyl- β -carboline

1	84%
2	77%
3	70%
4	80%
5	78%
6	81%
7	70%
8	62%

TABLE 2 (Continued)

9	64%
10	50%
11	78%
12	80%

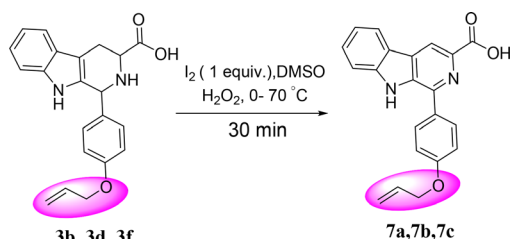
a drop of hydrochloric acid (HCl) gives corresponding β -carboline with deprotection of O-allyl groups (Table 1, entry 10, 11).

Therefore, 100 mol% iodine, DMSO, and external oxidant H_2O_2 (1 equiv.) give the best result. With the optimized conditions in hand, with this result, we turned our attention toward testing the scope of the protocol with a variety of tetrahydro- β -carboline. The series of O-allyl substituted tetrahydro- β -carboline having H, COOH, and COOMe groups at C-3 position was synthesized and treated with iodine in DMSO/ H_2O_2 at 100°C for up to 3h reaction time; the O-allyl substituted- β -carboline was isolated with 50–84% yield. The TH β C with H at C-3 position **3a**, **3b**, and **3c** was treated with iodine in DMSO/ H_2O_2 at 100°C , obtaining compound **6a**, **6b**, and **6c** with 84, 77, and 70% yield (Table 2, entry 1, 2, 3). We tested the generality of the protocol by subjecting other substrates **3b**, **3d**, and **3f** (COOH at C-3) in the reaction and found that, in each case, the transformation was successful, affording the corresponding products **6a**, **6b**, and **6c**. Therefore, in the first set of reactions, several substituted

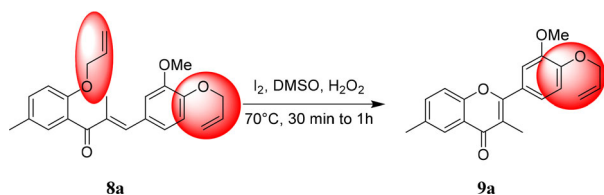
TH β C (**3a–3f**) were evaluated, and it was found that the substrates which hold *O*-allyl group at meta position of phenyl ring afforded **6c** in 65–70% yields. Next, we investigated the substrates **3g**, **3h**, **3i**, and **3j** (Table 2, entry 4, 5, 6, 7) bearing ester moiety at C-3 position of *O*-allyl TH β C methyl ester; we treated with iodine in DMSO/H₂O₂ under the standardized conditions leading to formation of corresponding *O*-allyl- β -carboline methyl ester (Table 2, **6d**, **6e**, **6f**, **6g**) with 72–80% yield. Similarly, the *meta* *O*-allyl substituted β -carboline-methyl ester gave 70% yield.

Further, we extended our investigation toward the selective deallylation followed by dehydrogenation of *O*-allyl TH β C; As per our previous report^[19,20], we have found that deallylation proceed by using molecular iodine in DMSO+H⁺ (20 mol %) at 60 °C and optimized condition in our hand (Table 1, entry 11). Thereafter, we have applied our previously developed protocol molecular iodine in DMSO/H⁺ with 50 and 100 mol% at 90 °C; it was observed that the aromatization of *O*-allyl-tetrahydro- β -carboline **3a**, **3b**, and **3g** (Table 2, entry 11, 12) gave **4a**, **5a** with 75–80% yield with deallylation (Table 2, entry 11, 12).

Recently, we have reported a novel methodology for chemoselective aromatization of TH β C with intact of the acid group at C-3 position (Scheme 2)^[18]; *O*-allyl tetrahydro- β -carboline-3-carboxylic acid **3b** was treated with I₂/DMSO/H₂O₂ at 70 °C for 30 min to 1.5 h afforded β -carboline-3-carboxylic acid **7a**, 62% yield with retention of acid group (Scheme 2). The *O*-allyl-substituted tetrahydro- β -carboline-3-carboxylic acid **3c**, **3d** (Table 2, entry 9, 10) treated with I₂ (1equiv.) DMSO H₂O₂ (1 equiv.) at 70 °C for 1 h afforded *O*-allyl-substituted



SCHEME 2 Synthesis of 1-(4-[allyloxy] phenyl)-9H-pyrido [3,4-b]indole-3-carboxylic acid



SCHEME 3 Synthesis of flavanone

β -carboline-3-carboxylic acid **7b**, **7c** with 64, 50% yield (Table 2, entry 9, 10).

In order to evaluate the possibility of applying this methodology on the variety of substrate and to avoid previous limitations, and to check the selectivity of the present protocol, we attempted the synthesis of flavanone (Scheme 3).^[19]

When the (E)-3-(4-[allyloxy]-3-methoxyphenyl)-1-(2-[allyloxy]-5-methylphenyl)prop-2-en-1-one **8a** was treated with iodine in DMSO/H₂O₂ at 100 °C, it gives 2-(4-[allyloxy]-3-methoxyphenyl)-3,6-dimethyl-4H-chromen-4-one **9a** with 85% yield (Scheme 3).

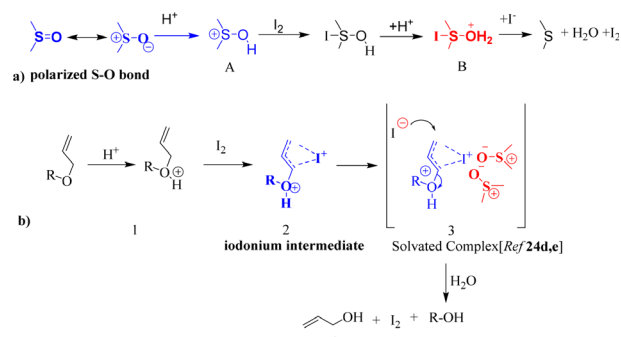
The methods show the new approaches for the non-*O*-deallylation followed by aromatization of *O*-allyl-TH β C. Compared to the previous studies, this method is more superior in terms of the selectivity, temperature, reaction time, cost, and efficiency.

3 | POSSIBLE MECHANISM

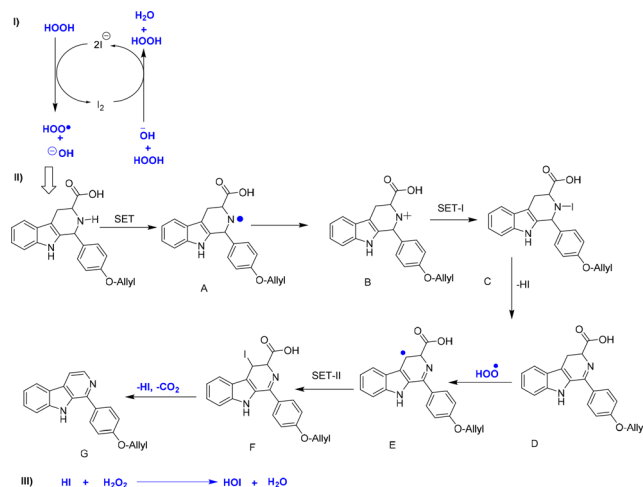
On the basis of the above experiments, a tentative reaction mechanism is delineated in (Scheme 4). This pathway involves the generation of iodonium ion in acidic medium from the reaction of DMSO and I₂, subsequently regenerating the molecular iodine (I₂) by further removal of H₂O and DMS (Scheme 4)^[24]

In the acidic medium, the *O*-allyl oxygen protonated molecular iodine, which activates the C=C bond and forms a three-member iodonium intermediate stabilized with protic DMSO solvent forming solvated complex,^[24c] and DMSO solvent might be helping in deallylation process. (Scheme 4b). The iodide ion is essential, which initiates the reaction and forms deallylated product **4** with molecular iodine and formation of allyl alcohol in the reaction could not be traced.

In diversely, the use of eternal oxidant H₂O₂ in the reaction medium facilitated the oxidation of iodide to iodine,^[24a–27] and best yield was obtained when H₂O₂



SCHEME 4 Proposed mechanism for aromatization and deallylation of allyl ether using iodine in DMSO



SCHEME 5 Proposed mechanism for aromatization of O-allyl THβC

(35 wt% in H₂O) was employed as an external oxidant. The oxidant H₂O₂ also enables the secondary product DMS to DMSO.^[24b] The aromatization of O-allyl THβC with nondeallylation indicated that a radical pathway is involved in this transformation.

Based on the above observations as well as from the previous reports, a plausible mechanism for this oxidative transformation is illustrated in Scheme 5. Initially, H₂O₂ is converted into radical catalytically with the addition of iodide anion, and this radical traps the hydrogen from the tetrahydro-β-carboline and generates radical THβC cation **A**. Subsequently, the hypothetical radical cation **A** is converted into carbocation intermediate **B** via oxidative single-electron transformation (SET).^[25–26] After that, the carbocation **B** can also be attacked with iodide anion to form N-iodo THβC. Subsequently dehydrohalogenation to form intermediates dihydro-β-carboline **D**. Finally, generation of intermediates **F** via SET-II mechanism and hydrogen iodide is departed to yield the final products **G**. The hydrogen iodide, generated during the reaction, is reoxidized to HOI by hydrogen peroxide.^[27]

4 | CONCLUSION

In conclusion, we have developed a novel method for the selective oxidative aromatization with no-O-deallylation of O-allyl THβC by using iodine in DMSO/H₂O₂. The perfection of this method is its tolerance of O-allyl group of THβC. The operational simplicity and economic viability of this method justify further study of this reaction. Besides, we have demonstrated a simple protocol sequential procedure for selective O-deallylation and aromatization of substituted O-allyl

THβC using iodine in DMSO, H⁺. The metal-free milder condition, operational simplicity, and low temperature reaction are some of the salient features that render this protocol to be a better alternative to the regular organic synthesis.

ACKNOWLEDGMENTS

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DATA AVAILABILITY STATEMENT

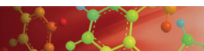
Additional supporting information found online in the Supporting Information section at the end of this article.

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SUPPORTING INFORMATION

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