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Aluminium chloride-potassium iodide-acetonitrile system: A mild reagent system for aromatic claisen rearrangement at ambient temperature

Nayan Kamal Bhattacharyya [a,*], Deepjyoti Dutta [b], Joydeep Biswas [a]

[a] Department of Chemistry, Sikkim Manipal Institute of Technology, Majitar, 7371 36, Sikkim, India
[b] North East Institute of Science and Technology - CSIR, Jorhat, 785006, Assam, India

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ABSTRACT

Claisen rearrangement is used as the standard methods for the generation of complex organic substance. It is one of the well-known methods for the introduction of carbon-carbon bond. We have developed a protocol using allyl aryl ether as a substrate and $AlCl_3$-KI as a mild reagent system and acetonitrile (CH_3CN) is taken as solvent at ambient temperature. The reagent system presented in this current work is found to be appropriate for Claisen rearrangement of several aromatic alcohols with excellent yields.

1. Introduction

Claisen rearrangement is one of the essential and beneficial synthetic procedure for organic chemists [1a]. The innovation of the Claisen rearrangement presented a potentially useful synthetic tool to organic chemists. Claisen rearrangement attracts most synthetic chemists due to its versatile application in synthesizing natural products and medicinal chemistry [1b]. The Claisen rearrangement is a chemical reaction where strong carbon–carbon bond formation occurs [2]. Investigation and research established the chemo-, regio-, diastereo-, and enantioselectivity of this reaction and the reaction can be carried out under gentle conditions and potentially useful for polyfunctionalized molecules [3a].

Ethers are applied as protecting groups for hydroxyl functions in organic synthesis and moreover a large number of ethers known as good protecting groups. Several examples illustrating the application of the Claisen rearrangement in preparing a wide range of synthetically striking building blocks and natural or biologically active compounds are considered. A numerous way can be applied to convert aromatic ethers to corresponding phenols.

Sonnenberg et al. [39] reported aromatic Claisen rearrangement of allyl phenyl ethers in the presence of alkyl aluminium halides under mild conditions. Treatment of allyl phenyl ether in hexane with an excess of diethyl aluminium chloride in hexane produced ortho allyl phenol. Borgulya et al. [40] also reported the Claisen rearrangement of allyl aryl ethers to corresponding phenols using Boron trichloride under thermal conditions to produce a mixture of ortho and para products. Maruyama et al. [41] reported the Claisen rearrangement of aryl-penta dienyl ethers in the presence of BF_3–OEt_2 lewis acid at very low temperature. Dauben et al. [42] used montmorillonite clays as a catalyst for the Claisen rearrangement of substituted allyl aryl ethers to corresponding ortho-allyl phenols under mild conditions. Sharma et al. [43] reported the Ytterbium catalyzed Claisen rearrangement of allyl phenyl ethers to corresponding ortho allyl phenols under reflux condition at a reaction time 48–72 h. Gignare [44] carried out aromatic Claisen rearrangement by microwave irradiation under very high thermal condition. Zi Hui [24b] studied the microwave-accelerated Claisen rearrangement of allyl aryl ethers using phosphomolybdic acid (PMA) as a catalyst. Ollevier et al. [45] used Bismuth triflate as a catalyst in catalytic amount for the Claisen rearrangement of allyl naphthyl ethers to corresponding ortho naphthol. Deodhar et al. [46] reported selective Claisen rearrangement of allyl aryl ethers using zeolite mediated catalyst under microwave irradiation. Claisen rearrangement of allyl aryl ethers catalyzed by zinc to corresponding *ortho*-allylated adducts was reported by Gupta et al. [47]at 55 °C in liquid phase.

In recent years, scientists have given interest in aluminium chemistry and aluminium iodide, which has attained much importance in synthetic organic chemistry [3b,38a-38d,49]. However, the chemistry of aluminium chloride in the presence of potassium iodide is less explored [3c,3d] although it works as an efficient reagent for various reactions. M. Boruah et al. [51] used $AlCl_3{\cdot}6H_2O/KI/H_2O/CH_3CN$ system in hydrated media for dehydration of oximes and amides. M. Boruah et al. [52] also used $AlCl_3{\cdot}6H_2O/KI/CH_3CN/H_2O$ system in hydrated media for deoxygenation of organic N-oxide. Dilip Konwar et al. [38a] used $AlCl_3$–NaI reagent for dehydration of oximes, amides and Beckmann rearrangement

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* Corresponding author.
E-mail address: nayan.b@smit.smu.edu.in (N.K. Bhattacharyya).

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of ketoximes to anilides at room temperature. Dilip Konwar et al. [49] also reported the development of aluminium iodide as an efficient reagent for dehydration of aldoxime to nitriles and Beckmann rearrangement of ketoximes to anilides. We report the utility of $AlCl_3$-KI in CH_3CN as a mild and efficient reagent for the Claisen rearrangement. Aluminium iodide has multifaceted reactivity. Because of oxophilic character of lewis acid it can form complexes with esters, ethers, oxiranes, diols, *N*-oxides, and sulfoxides that decompose spontaneously to give acids, alcohols and olefins via ester and ether cleavage, deoxygenation of oxiranes, and deoxydehydration of diols, respectively [50].

There are various methods developed for modification of this reaction. In general, Claisen rearrangement is carried out in thermal condition [4]. Nevertheless, in the last few decades, there has been developing such methods where the reaction is carried out in the low-temperature condition. In this respect BCl_3 [5], BF_3–OEt_2 [6], Et_2AlCl [7], n-Bu_3SnH [8], CF_3COOH [9], pyridine/reflux [10]etc.; some metal triflate such as $Bi(OTf)_3$ [11], $Yb(OTf)_3$ [12], $Sn(OTf)_2$, $Cu(OTf)_2$, $Zn(OTf)_2$ [13], Me_3SiOTf [14] and other reagents like montmorillonite clays [15], decline [16] being reported for Claisen rearrangement type reaction.

Some metal complexes such as $Rh_2(OAc)_4$ [17], $RuCl_2(PPh_3)_3$ [18], $(dba)_3Pd_2{\cdot}CHCl_3$ [19], $PdCl_2(CH_3CN)_2$ [20], $(Cl)_2(CPy_3)_2Ru=CHPh$ [21], chiral aluminium Bi-Napthol [22], chiral C2-symmetric bisulfonamide derived Boron reagent [23], *etc*. have been identified as more selective and efficient reagents for modification of this reaction. Nowadays, some greener methods such as microwave irradiation [24a,24b], ultraviolet irradiation [25] etc. are also used in this purpose.

Besides these other reagents such as MgI_2-bis(oxazolinyl)aryl ligands [26], KDMSO-LiCl ligands [27], Ag-KI/HOAc system [28], *etc*. and some of the metals such as Zn dust [29], Zn in THF [30], domino Cu [31], Al-MCM-41 [32], zeolite catalyst [33] being also reported.

Introduction of various solvents such as tetradecane, carbitol, EtOH–H_2O, *etc*., affecting the Claisen rearrangement reaction [34]. Some polar solvents in mild condition give rearrangement products, but it cannot be obtained via classical conditions due to thermal decomposition [35].

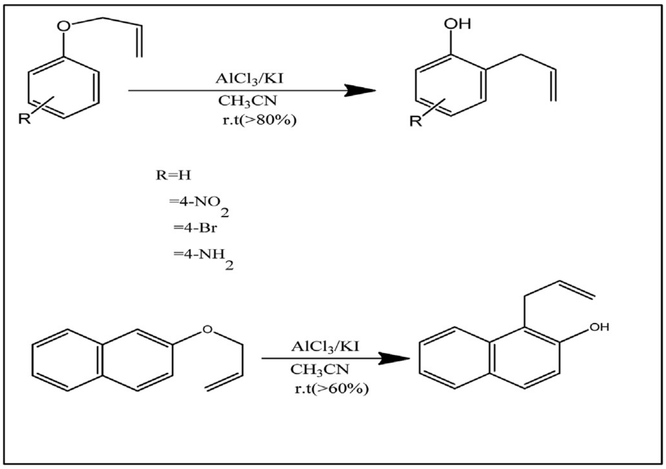
However, the majority of these methods have some limitations like harsh conditions, monotonous work up procedure, uncomfortable product isolation and bi-products formation. Besides a large number of catalysts reported in the literature are expensive. Therefore, looking of new procedure is still going on to carry out the Claisen type rearrangement reaction at ambient temperature condition using a cheap, commercially available reagent.

Anhydrous $AlCl_3$ has recently been applied as a lewis acid catalyst for varieties of conversions as a low-cost and readily available reagent. Gogoi et al., 2006 used the $AlCl_3$-KI-CH_3CN system for chemoselective C–O bond cleavage of esters, acetals, ethers and oxathiolanes [38b]. Node et al., 1992 explained a method for selective dealkylation aliphatic methyl ether under mild conditions using $AlCl_3$–NaI–CH_3CN system [36]. Sang et al., 2018 proposed a method for cleavage of aryl methyl ether under mild conditions using $AlCl_3$-KI-CH_3CN system [38c]. Keeping this in mind, we have used the same reagent system for the Claisen rearrangement of aromatic alcohols from its ether moiety (Scheme 1).

2. Results and discussion

Most of the early preparative methods for the aromatic Claisen rearrangement were in thermal condition as well as catalyzed by transition metals. Furthermore, most of the methods suffer from the limitations like – (a) use of a stoichiometric amount of catalyst, (b) high-temperature condition, (c) use of expensive catalyst, (d) a low percentage of yield, (e) cumbersome reaction condition as mentioned in Table 1.

Our present investigation, therefore, directed to address the limitation mentioned above in aromatic Claisen rearrangement. Here we describe an alternate procedure of aromatic Claisen rearrangement at ambient temperature applicable to both phenyl and naphthalene system to afford the corresponding product in good yield. The noteworthy significances of our present system are – (i) comparatively short reaction time, (ii) ambient temperature condition, (iii) cost-effectiveness and (iv) simple work-up procedure.



Scheme 1. Synthesis of *o*-allylated adducts from corresponding aryl allyl ether using $AlCl_3$-KI-CH_3CN.

Initially, we carried out the synthesis of allyl aryl ether by adding a solution of NaOH to the phenol solution using phase transfer catalyst like cetyltrimethylammonium bromide (CTAB). In this process, the drop-wise addition of allyl bromide was gradually completed and dichloromethane was used as a solvent. After this, the reaction mixture's stirring was continued at ambient temperature and the reaction's progress was monitored using TLC. We observed that reaction was being completed at 3 h and corresponding phenyl allyl ether was being isolated [37].

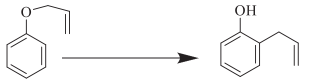
After the success of this reaction, the process was extended to a variety of phenol derivatives. It can be seen from Table 2 that various phenols containing functional groups reacted successfully to give corresponding ether in sufficiently high yields. In general, the phenyl system derivative takes 3–8 h to complete the reaction while the naphthalene ring containing derivatives reacts at faster reaction rates that take 1.5–2 h to complete the reaction. Naphthalene is more reactive than benzene, because the resonance energy of naphthalene (61 kcal/mol) is less (11 kcal/mol) than that of two benzene rings (2*36).

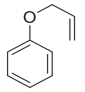
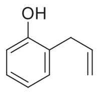
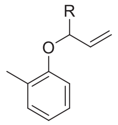
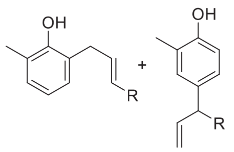
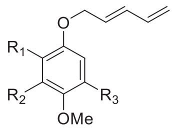
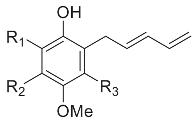
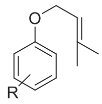
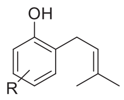
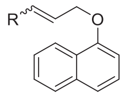
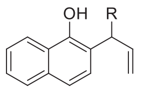
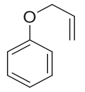
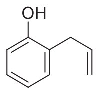
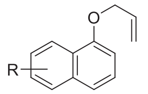
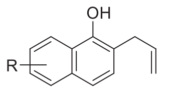
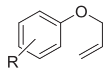
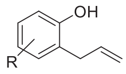
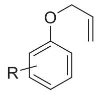
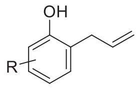
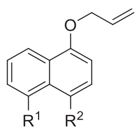
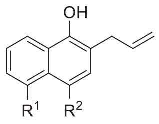
The main part is to carry out Claisen rearrangement at ambient temperature using $AlCl_3$-KI- CH_3CN system [38] as a mild reagent. At first, $AlCl_3$ and KI (1:3) were taken in dry acetonitrile and the stirring of the mixture was continued magnetically at ambient temperature for 40 min. After that phenyl allyl ether was added and stirring was continued at ambient temperature. Thin-layer chromatography was used to monitor the progress of the reaction. It was observed that reaction was taken place at ambient temperature and correspondingly *o*-allylated phenol was being isolated.

After the reaction's success, the process was also extended to various allyl aryl ether derivatives. It was being seen from Table 3 that allyl aryl ethers containing functional groups reacted successfully to give corresponding *o*-allylated alcohol products in suitably high yields. In general, allyl aryl ether derivatives require 16–20 h for the completion of the reaction. It was also observed that the naphthalene ring containing ether derivative took 6–10 h for the completion of the reaction and also the percentage of yield was moderately high.

The yields were found very good in all cases with comparatively lesser time. The products were analyzed by spectral analysis.

Table 1
The following table suggests the various procedure used for Claisen rearrangement.



Substrate	Reaction condition	Product	References
	R_2AlCl, 65 °C/Hexane (R = Et, i-Bu)		Sonnenberg et al. [39]
 R = H, CH_3	BCl_3/Heat		Borgulya et al. [40]
 R₁ R₂ R₃ Me Me H Me H Me MeO H H H t-Bu H	BF_3–OEt_2/−25 °C		Maruyama et al. [41]
 R = H, 4-OCH_3, 4-OC_2H_5, 3,5-CH_3, 4-$COCH_3$	Clay, Benzene/50 °C		Dauben et al. [42]
 R = H, CH_3	$Yb(OTf)_3$, CH_3CN/reflux		Sharma et al. [43]
	MW/180 °C		Gignare et al. [44]
 R = H, 4-OCH_3, 4-Cl, 5-OCH_3, 7-OCH_3, 5-OCH_2-CH=CH_2	$Bi(OTf)_3.xH_2O$ (cat.) 1–40 h		Ollevier et al. [45]
 R = H, 6-CH_3, 4- OCH_3, 5-CH_3, 6-OCH_3, 6-CHO, α-Naphthol, β- Naphthol	Zeolite, MW/80 °C		Deodhar et al. [46]
 R = H, 4-OCH_3, 4-Cl, 4-Br, 4-NO_2, 4-$COCH_3$ 4-Me, 4-OEt	Zn/THF, Stirring, 55 °C		Gupta et al. [47]
	20 mol%, $Bi(OTf)_3.xH_2O$		Thierry Ollevier et al. [48]

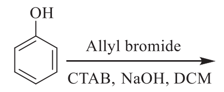
A proposed mechanism for the $AlCl_3$-KI-CH_3CN mediated Claisen rearrangement is depicted in Fig. 1. Solvation of $AlCl_3$ in CH_3–C≡N led to the formation of complex CH_3CN–$AlCl_3$ which reacts with allyl ether to give CH_3CN and an oxonium ion $RO^{(+)}CH_2CHCH_2AlCl_3^{(-)}$. The oxonium ion reacts with potassium iodide to form allyl iodide. The lewis acid activated the phenoxide ion undergoes electrophilic substitution reaction. This conversion might involve a transition state where $AlCl_3$ would activate the phenoxide ion as well as the allyl group from allyl iodide.

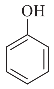
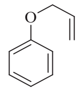
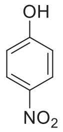
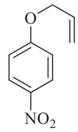
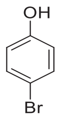
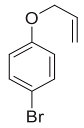
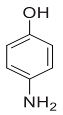
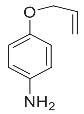
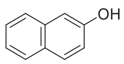
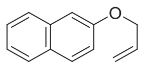
3. Experimental section

3.1. Materials and methods

Analytical reagent grade chemicals were used for the synthesis, which are commercially available, and used without any further purification. The crude product obtained was purified using column chromatography where silica gel was used as stationery phase and petroleum ether and

Table 2
Preparation of allyl aryl ether using allyl bromide.



Entry	Substrate	Product	Yield	Time (h)
1			82%	3
2			76%	8
3			80%	6
4			72%	5
[a]5			65%	1.5

- Reaction condition: Substrate 0.01 mol, allyl bromide 0.01 mol, phenol 0.02 mol.
- [a]Reaction condition: Substrate 0.01 mol, allyl bromide 0.01 mol, phenol 0.015 mol.

ethyl acetate mixture were used as eluent. The percentage of yield was calculated using the following relation.

$$\text{Percentage Yield} = \frac{\text{Actual Yield}}{\text{Theoretical Yield}} \times 100\%$$

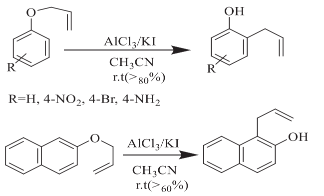
3.2. *General procedure for the preparation of aryl allyl ether*

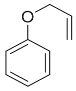
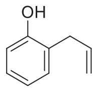
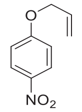
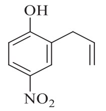
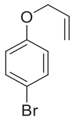
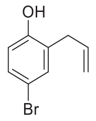
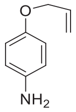
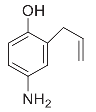
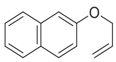
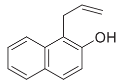
The procedure for the preparation of aryl allyl ether was taken from literature [37,53]. Phenol (0.01 mol) and NaOH (0.02 mol) was taken in dichloromethane (5 mL) and water (5 mL) mixture and then cetyl-trimethyl ammonium bromide (CTAB) (0.0001 mol) was mixed in a 100 mL round-bottomed flask. Allyl bromide is being added dropwise (0.02 mol) and stirring of the reaction mixture at ambient temperature was continued for 3 h. Organic and aqueous layer were separated from each other and 10 mL of dichloromethane (twice) was used to extract the aqueous layer. The organic layers were combined and then dried with anhydrous Na_2SO_4 and then extracted in Buchi Rotavapor under reduced pressure. The product was separated by column-chromatographic technique using 5% of ethyl acetate in petroleum ether as eluent.

3.3. *General procedure for the synthesis of o-allyl phenol in $AlCl_3$–KI–CH_3CN system*

Anhydrous aluminium chloride (1 mmol) and potassium iodide (3 mmol) were added to dry acetonitrile (20 mL) and the mixture was stirred magnetically at ambient temperature for half an hour. Then phenyl allyl ether (1 mmol) was added, stirred for 6–20 h and thin-layer chromatographic technique was used to monitor the reaction's progress. The reaction mixture was added into a cold ammonical water solution and extracted with diethyl ether. The organic layer was washed with water and solvent was removed in Buchi Rotavapor so that crude product was obtained and purification is done using column chromatography where silica gel was used as stationary phase and petroleum ether and ethyl acetate mixture were used as eluent.

Table 3
$AlCl_3$+KI mixture catalyzed Claisen rearrangement of allyl aryl ethers to *o*-allylated products in acetonitrile.



Entry	Substrate	Product	Yield (%)	Time (h)
1			84	20
2			87	16
3			80	18
4			76	12
[a]5			72	6

- Reaction condition: Substrate 1 mmol, aluminium chloride 1 mmol, KI 3 mmol.
- [a]Reaction condition: Substrate 1.5 mmol, aluminium chloride 1 mmol, KI 3 mmol.

4. Supportive information

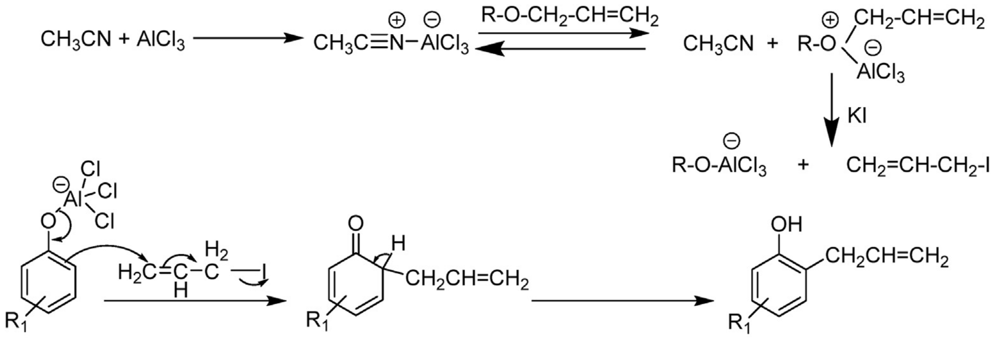
IR spectra were recorded on Elmer FT-IR 2000 spectrometer (KBr discs) in the range from 4000 to 400 cm^{-1}. NMR of the compounds were performed in a 500 MHz NMR spectrometer with tetramethylsilane (TMS) as internal reference and $CDCl_3$ as solvent. All chemical shifts (δ) are reported in ppm and coupling constant (*J*) in Hz.

Phenyl allyl ether: IR (KBr, cm^{-1}) ν: 3086.2 (Aromatic C–H, Alkene = C–H), 1592.4, 1509.2 (Aromatic C=C), 1174.4, 1112.5 cm^{-1} (Ether C–O). ^1H NMR ($CDCl_3$, δ ppm): 7.17 (2H), 6.91 (2H), 6.82 (1H), 6.06 (m, 1H, H), 5.43 (1H, dd, H), 5.31 (1H, dd, H), 4.67 (2H, H), 4.67 (2H, H).

Ortho allyl phenol: IR (KBr, cm^{-1}) ν: 3418.1 (O–H), 1643.9 (Alkene C=C), 1485.7, 1460.1 cm^{-1}(Aromatic C=C). ^1H NMR ($CDCl_3$, δ ppm): 9.68 (s, OH), 7.11 (2H), 6.80 (1H), 6.74 (1H), 5.92 (m, 1H, H), 5.12 (1H, dd, H), 4.87 (1H, dd, H), 3.33 (2H, H), 3.33 (2H, H).

Para nitro allyl phenyl ether: IR (KBr, cm^{-1}) ν: 3076.7, 2980.2 (Aromatic C–H, Alkene = C–H), 1659.1, 1621.6 (Alkene C=C), 1565.6 (N–O), 1236.4 cm^{-1} (Ether C–O). ^1H NMR ($CDCl_3$, δ ppm): 8.03 (2H), 7.15 (2H), 6.06 (m, 1H, H), 5.43 (1H, dd, H), 5.31 (1H, dd, H), 4.67 (2H, H), 4.67 (2H, H).

Para nitro ortho allyl phenol: IR (KBr, cm^{-1}) ν: 3438.7 (O–H), 3045.0 (aromatic C–H), 1594.9 (N–O), 1493.2 cm^{-1}(Aromatic C=C). ^1H NMR ($CDCl_3$, δ ppm): 11.88 (s, OH), 8.21 (1H), 8.12 (1H), 7.07 (1H), 5.92 (m, 1H, H), 5.12 (1H, dd, H), 4.87 (1H, dd, H), 3.33 (2H, H), 3.33 (2H, H).



R_1 = H, NO_2 , NH_2, Br, beta-Napthol

Fig. 1. Proposed reaction mechanism.

5. Conclusion

In summary, an efficient procedure has been developed for the synthesis of *o*-allylated adducts from the corresponding aryl allyl ether using $AlCl_3$-KI-CH_3CN mild reagent system. Our present reagent system's noteworthy significances are comparatively short reaction time, room temperature condition, cost-effectiveness, and simple work-up procedure. The procedure is easy to perform at ambient temperature and applicable to different aryl allyl ether (both phenyl and naphthalene system) to give corresponding *o*-allylated phenols. The reagent system presented in this current work is found to be appropriate for Claisen rearrangement of several aromatic alcohols with excellent yields. Moreover, this procedure is also applicable to the naphthalene ring system to give corresponding *o*-allylated adducts in suitably high yields. Utilizing this kind of efficient reagent system in organic rearrangements would be more influential and immensely encouraging to academic and industrial researches.

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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