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An efficient approach to isoquinoline *via* $AgNO_3$-promoted 6-*endo*-dig cyclization followed by oxidative elimination of *o*-alkynylarylaldimines and its application in fluoride recognition

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ABSTRACT

A novel 6-*endo*-dig cyclization followed by oxidation/elimination of *o*-alkynylarylaldimines with 4-hydroxybenzylamine was developed for preparation of isoquinolines. The intermediates of this tandem reaction were monitored by mass spectroscopy (MS) to confirm the reaction pathway. This methodology was further applied to the design and synthesis of a novel ratiometric chemosensor for determination of fluoride.

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Isoquinoline and its derivatives have gained widespread attention due to their extensive range of therapeutic and biological activities, such as antimicrobial, anti-HIV-1, antifungal and antitumour properties [1]. Isoquinolinium salts, formed by *N*-alkylation of isoquinolines, are also important alkaloids with numerous valuable biological activities and other applications in dyes and paints [2]. In addition to the excellent bioactivities of these compounds, they also are versatile intermediates for the synthesis of therapeutic agents [3].

A number of methods for the preparation of isoquinoline and derivative compounds have been reported because of their remarkable physiological properties and significant role in organic synthesis. Among the wide range of strategies employed, catalysis of 6-*endo*-dig ring closures of 2-(1-alkynyl) arene carboxaldehyde imines by metal ions or other cations have emerged as some of the most efficient, owing to their high selectivity and mild conditions (Scheme 1a) [4]. These reactions are mainly restricted to transition metals such as Cu, Ag, Pd and Au, with Ag-catalysed ring closure reactions giving the best results [5]. For an example, Liang developed an efficient method for the synthesis of substituted isoquinoline *via* silver catalyzed cyclization of 2-alkynyl benzyl azides (Scheme 1a). But the synthesis of substrates 2-alkynyl benzyl azides was complicated and needed dangerous azides as raw meterials (Scheme 1a) [6]. Another method that has been employed is transition metal-catalysed direct C—H bond functionalisation of arenes bearing nitrogen-containing directing groups, followed by cyclisation with the resulting internal alkynes. This approach has enabled the highly efficient preparation of diverse isoquinolines containing a wide range of functional groups [7]. However, noble-metal rhodium catalysts were usually required to achieve C—H functionalization in some reactions (Scheme 1b). Other nitrogen-containing compounds, such as 2-(cyanomethyl)-benzonitriles, 3-aminopyrazine-2-carbohydrazide and *tert*-butylamine/benzamidine were also used to prepare isoquinolines [8]. Ammonium salts, ammonia were reported as efficient nitrogen sources to synthesize isoquinolines catalyzed by transition metals [9]. In our previous studies, methods for the synthesis of isoquinoline derivatives by Ag-mediated annulation/N—N bond cleavage were reported [10].

Methylene benzoquinone is an excellent synthetic intermediate that is often used as the Michael receptor in the synthesis of various compounds [11]. This unit is a very good leaving molecule *via* oxidation/elimination because of its stable electronic structure [12]. Therefore, this motif or a similar unit has been incorporated

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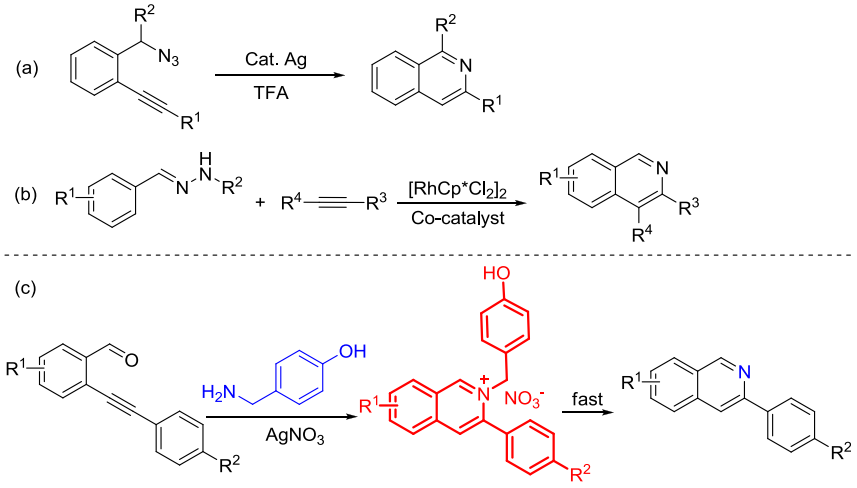


Scheme 1. Synthetic routes for isoquinolines.

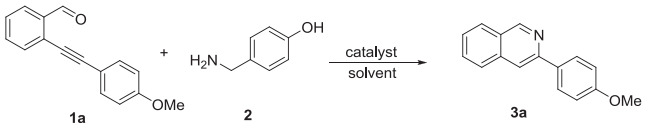
into many fluorescent molecules for recognition of reactive oxygen species [13]. When these chemosensors are subjected to an oxidant they release methylene benzoquinone (or similar unit) as a result of the change of probe structure. Accordingly, the fluorescence wavelength and intensity will change as a result of the change in conjugation in the structure.

Derivatives of 4-hydroxybenzylamine on *N* atom have the potential to release methylene benzoquinone and to provide nitrogen source under certain oxidation conditions. Therefore, this study reports a novel methodology for the synthesis of isoquinolines *via* 6-*endo*-dig cyclisation followed by oxidation/elimination based on 4-hydroxybenzylamine. 2-(1-Alkynyl) arene carboxaldehyde imines were generated *in situ* from *o*-alkynyl benzaldehydes and amines and then catalyzed by Ag to afford the benzyl isoquinolinium salts, which were oxidised immediately under oxidation conditions to eliminate methylene benzoquinone and provide isoquinolines (Scheme 1c).

To verify the practicability of this route, a set of experiments was performed using 2-((4-methoxyphenyl)ethynyl)benzaldehyde **1a** and 4-hydroxybenzylamine **2** as model substrates. Optimisation of the reaction conditions was carried out with respect to different Lewis acids, solvents and temperatures (Table 1). Silver is an efficient catalyst and has been known to activate alkynes, which prompted the exploration of different methodologies for the synthesis of heterocycles. Initially, 30 mol% $AgNO_3$ in 1,2-dichloroethane (DCE) was selected to catalyze this reaction, resulting in a 42% yield of designed product **3** (Entry 1, Table 1). Other Lewis acids (AgOTf, AgTFA, CuI, $CoCl_2$, $Ni(OAc)_2$, $Zn(NO_3)_2$, and $PdCl_2$) were somewhat inferior (Entries 2–8, Table 1). The efficiency of $AgNO_3$ was greatly improved in EtOH resulted in a yield of 66% (Entry 9, Table 1). Next, the catalyst loading was found to have a significant effect on the conversion: 100 mol% $AgNO_3$ was found to be optimum for the highest conversion of **1a** and **2** in EtOH at 80 °C (Entry 13, Table 1). Increasing the amount of 4-hydroxybenzylamine had negative effect on the yield of the desired product (Entry 15, Table 1). Decreasing or increasing the temperature had a negative effect on the yield of target product **3** (not shown).

The scope of the reaction was explored next using the optimised reaction conditions (Scheme 2). Generally, the domino reactions provided the desired isoquinolines in moderate to good yields. Various R^1 and R^2 substituents on the phenyl ring, including chloro, methyl, fluoro and methoxy groups, were well tolerated. As seen from Scheme 2, the electronic effects of R^1 and R^3 had significant influence on the reaction efficiency: the yields of desired products increased from 62% to 78% and then 88% when the substituents at R^1 changed from methoxyl (**3c**) to methyl (**3b**) to chloro (**3d**), respectively (Scheme 2). The results indicated that the electron withdrawing groups at R^1 provided better product yields than electron donating groups. Indeed, a similar trend was observed for the compound series **3f**, **3k** and **3g**. An opposite phenomenon was observed for substituents at R^3. The yields of targeted products decreased from 86% to 69% and 62% when the substituents at R^3 changed from methoxy (**3a**) to methyl (**3 h**) and chloro (**3 k**), respectively, indicating that electron donating groups at R^3 resulted in greater reaction efficiency than electron withdrawing groups. This result may be explained by the fact that the higher the density of the electron cloud on the triple bond, the stronger the 6-*endo*-dig cyclisation ability of substrate **1** with the amine **2** [14].

Table 1
Optimisation of reaction conditions for synthesis of isoquinolines.[a]



Entry	Catalyst	Solvent	Proportion (**1a:2**)	Yield %[b]
1	$AgNO_3$ (30%)	DCE	1:1	42
2	AgOTf (30%)	DCE	1:1	35
3	AgTFA (30%)	DCE	1:1	24
4	CuI (30%)	DCE	1:1	15
5	$CoCl_2$ (30%)	DCE	1:1	8
6	$Ni(AcO)_2$ (30%)	DCE	1:1	11
7	$Zn(NO_3)_2$ (30%)	DCE	1:1	Trace
8	$PdCl_2$ (30%)	DCE	1:1	20%
9	$AgNO_3$(30%)	EtOH	1:1	66
10	$AgNO_3$(50%)	EtOH	1:1	74
11	$AgNO_3$(80%)	EtOH	1:1	80
12	$AgNO_3$(90%)	EtOH	1:1	82
13	**$AgNO_3$(100%)**	**EtOH**	**1:1**	**86**
14	$AgNO_3$(120%)	EtOH	1:1	81
15	$AgNO_3$(100%)	EtOH	1:1.5	77

Bold display is to highlight the best reaction condition.
[a] The reactions were performed using 0.3 mmol of 2-(phenylethynyl)benzaldehyde **1a** and **2** in 2.0 mL of solvent at 80 °C for 12 h.
[b] Isolated yield.

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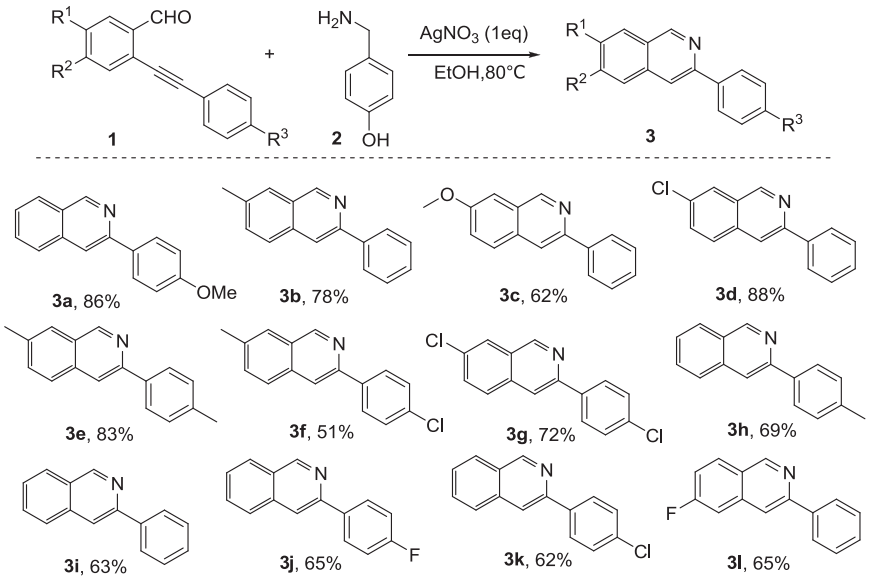
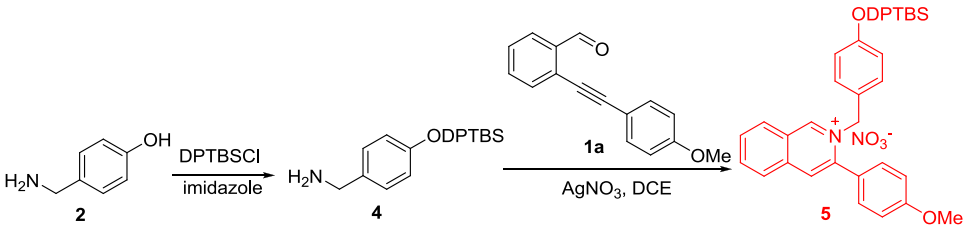


Scheme 2. Substrate scope of different 2-alkynylbenzaldehydes with 4-hydroxybenzylamine in the presence of $AgNO_3$.



Scheme 3. Synthetic route of isoquinolinium salt **5**.

It was discovered that protection of the benzylamine hydroxyl group resulted in halting of the reaction at the isoquinolinium salt. Enlightened by this, a novel ratiometric fluorescent probe based on an isoquinolinium salt was designed and synthesized for determination of fluoride. 4-Hydroxybenzylamine was protected as the diphenyl-*t*-butylsilyl (DPTBS) ether, then reacted with 2-((4-methoxyphenyl)ethynyl)benzaldehyde **1a** to provide probe **5** under the optimized reaction conditions (Scheme 3).

With fluorescent probe **5** in hand, it was expected that deprotection with fluoride would produce the 4-hydroxybenzyl isoquinolinium salt intermediate, which was then applied to detect reactive oxygen species. Unexpectedly, the colour of the probe system changed from yellow-green to blue immediately after the addition of fluoride. This phenomenon meant that there had been significant changes to the conjugated structure, not simply arising from DPTBS removal. It was proposed that the probe had been oxidised, eliminating methylene benzoquinone to form the isoquinoline unit; this was confirmed by the fluorescent emission spectra of compounds **5** and **3a** (Fig. 1). Therefore, probe **5** was successfully applied to detect fluoride [15].

Fluorescence recognition experiments were performed *via* the addition of fluoride to **5** in MeCN/H_2O (9:1 v/v). As shown in Fig. 1, the fluorescence emission peak for **5** was at 522 nm with a large Stokes shift of 172 nm (λ_{ex} = 350 nm, Fig. S1). When F^- was added into the solution, the peak at 522 nm weakened dramatically and a new peak at 394 nm began to appear with a blue shift of 128 nm. The large shift would decrease the interference of background noise and improve the sensitivity of detection, which is highly advantageous compared to general ratiometric or single signal probes [16]. With an increased amount of F^- (from 0 to 4, 6, 9 μmol L^{-1}), the peak at 394 nm increased accordingly, and the peak at 522 nm disappeared gradually. Consequently, chemosensor **5** could be used in quantitative determination of fluoride. Further quantitative determinations and selectivity investigations on this probe for fluoride are underway.

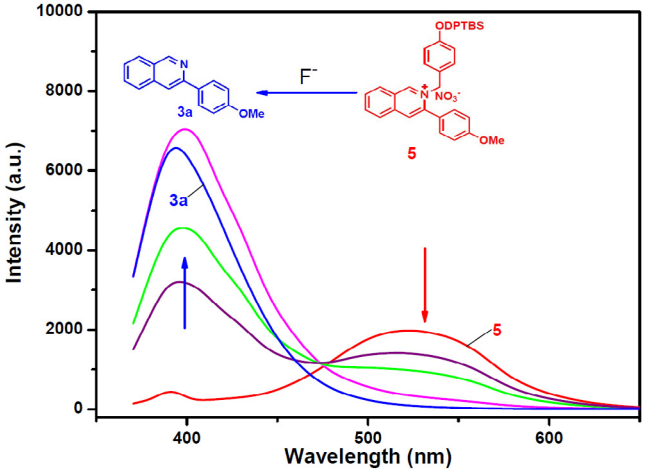


Fig. 1. Fluorescent emission of compounds **5**, **3a** and the emission changes of **5** (10 μmol L^{-1}) in the presence of increasing amounts of F^-.

The possible reaction pathway of this tandem reaction was demonstrated by monitoring reaction intermediates using

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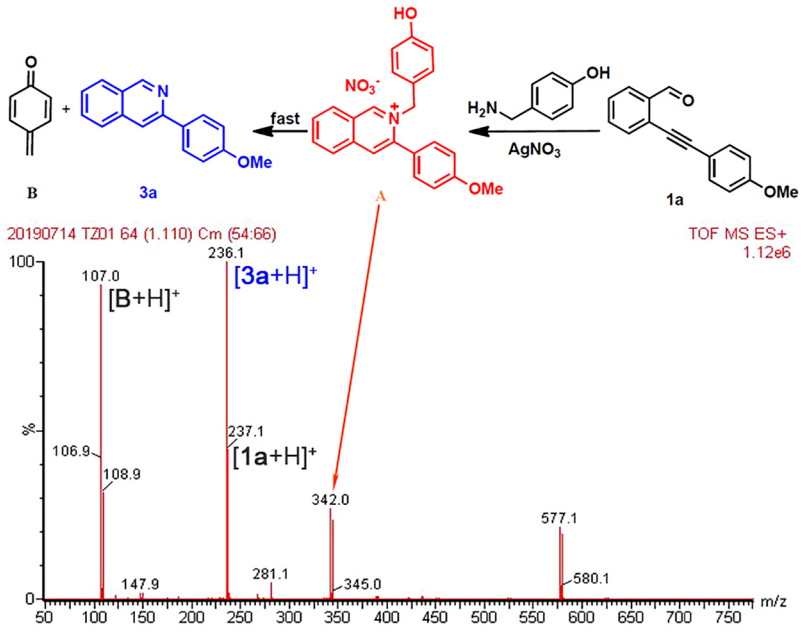

Fig. 2. The reaction pathway of this tandem reaction was confirmed by TOF-MS.

TOF-MS. A small amount of the reaction solution was decanted for MS monitoring. As shown in Fig. 2, the $AgNO_3$-catalyzed reaction of *o*-alkynylarylaldehyde **1a** with 4-hydroxybenzylamine produced a cationic intermediate **A**, corresponding to the signal at 342.0. Because of the driving force of aromatisation induced by F^-, the intermediate immediately decomposed into isoquinoline and methylene benzoquinone **B**, corresponding to the peaks at 236.1 and 107.0, respectively. This reaction process was consistent with the proposed reaction pathway.

In summary, an efficient silver nitrate-promoted 6-*endo*-dig cyclisation followed by oxidation/elimination of *o*-alkynylarylaldimines with 4-hydroxybenzylamine for the efficient synthesis of isoquinolines in moderate to good yields is reported. A novel fluorescent probe is designed and synthesised for recognition of fluoride, and this process further confirms the reaction pathway. Further study on the efficiency of isoquinolinium salt **5** for determination of fluoride is underway.

Acknowledgments

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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.2019.151187.

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