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Brønsted acid catalysed synthesis of 3-(2-alkoxyethyl)indoles from α- arylaminocyclobutanones and alcohols

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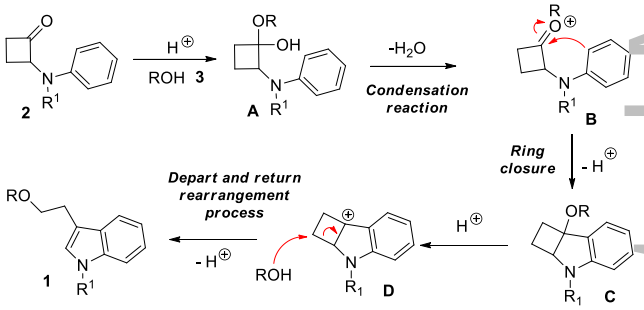
Abstract. A new Brønsted acid catalysed solvent-free cascade reaction consisting of a ring closure–ring fission process between α- arylaminocyclobutanones and alcohols has been established, providing highly functionalised 3-(2-alkoxyethyl)indoles in good to excellent yields.

Keywords: alcohols; carbocycles; nitrogen heterocycles; cascade reaction; synthetic methods

Functionalized indoles constitute an important class of nitrogen heterocycles with widespread applications.[1] These heterocycles are found in numerous natural products possessing diverse biological activities[2] and represent useful building blocks for the synthesis of other nitrogen-containing heterocycles.[3] 3-(2-Alkoxyethyl)indoles **1** are an important sub-group of this family, serving as valuable intermediates for the preparation of indole-fused polycyclic compounds,[4] including therapeutics such as pemedolac[5] and etodolac.[6] Classical methods for the synthesis of 3-(2-alkoxyethyl)indoles usually require multistep procedures – typically 4-5 steps starting from an aniline or a phenylhydrazine – and harsh conditions.[7] More efficient methods for their preparation from readily available precursors are clearly desirable. Recently,[8] we established Brønsted acid catalysed cascade reactions allowing rapid access to diversely substituted tryptamines from anilines in one-pot metal-free processes employing α-hydroxycyclobutanone as a key four-carbon synthon.[9,10]

We felt that this unusual synthetic paradigm for the construction of substituted indoles could be further exploited to provide an expedient access to 3-(2-alkoxyethyl)indoles **1**. We envisaged that the acid-mediated condensation of a readily available α-arylaminocyclobutanone **2**[11] with an alcohol **3** would lead to oxonium ion **B** (via **A**) which would undergo ring closure to give tricyclic intermediate **C** followed by an acid-induced ''depart-and-return'' rearrangement process, via **D**, to provide the target structure **1** directly (Scheme 1). We report here on the successful achievement of this objective.



Scheme 1. Proposed reaction sequence for the direct synthesis of (2-alkoxyethyl)indoles **1**.

We began our investigations by treating a solvent-free mixture *N*-methyl-*N*-phenyl-α-aminocyclobutanone **2a** and ethanol **3a**, in a sealed tube, with a panel of Brønsted acids (Table 1). In the presence of 20 mol% *p*-toluenesulfonic acid (TsOH), the reaction barely advanced over 2 days at room temperature or at 40 °C (entries 1,2). Increasing the temperature to 60 °C was propitious, however, providing **1a** in 67% yield (entry 3) and at 80 °C the yield improved further to a very satisfying 88% (entry 4). When other Brønsted acid catalysts – HI, HBr, HCl, CF_3COOH, MsOH and $(PhO)_2POOH$ – were employed in the same conditions, the yield of **1a** decreased partially or substantially (entries 5-10).

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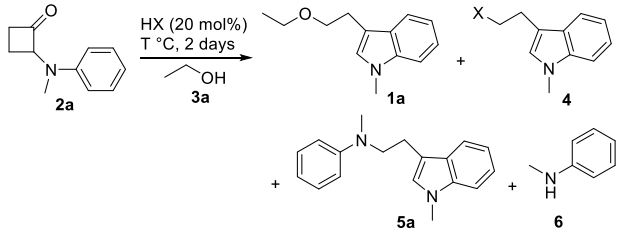
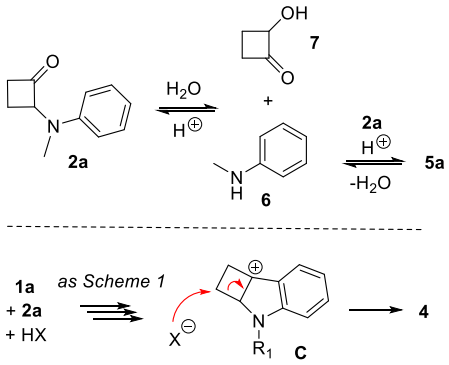
Interestingly, three by-products were detected in variable amounts in these test reactions: a haloethyl derivative **4**, tryptamine **5a** and *N*-methylaniline **6**. The formation of these compounds was rationalized in terms of two reactions pathways which compete with the proposed mechanism (Scheme 1) for the formation of **1a**. Under acidic conditions, the α-aminocyclobutanone **2a** may decompose into *N*-methyl aniline **6** and α- hydroxycyclobutanone **7** following a known equilibration;[8] subsequent condensation between **6** and a second equivalent of **2a** followed by the tandem ring closure-ring fission process[8] would lead to **5a** (Scheme 2). The formation of **4** was only significant for the halogen acids, whose conjugate bases have more nucleophilic character than the other acids investigated. For this reason, competition may exists for the "return" part of the depart-and-return mechanism, whereby halide anion adds to intermediate **C**, leading to **4** instead of **1a** (Scheme 2).

Table 1. Screening of a panel of Brønsted acids.[a]



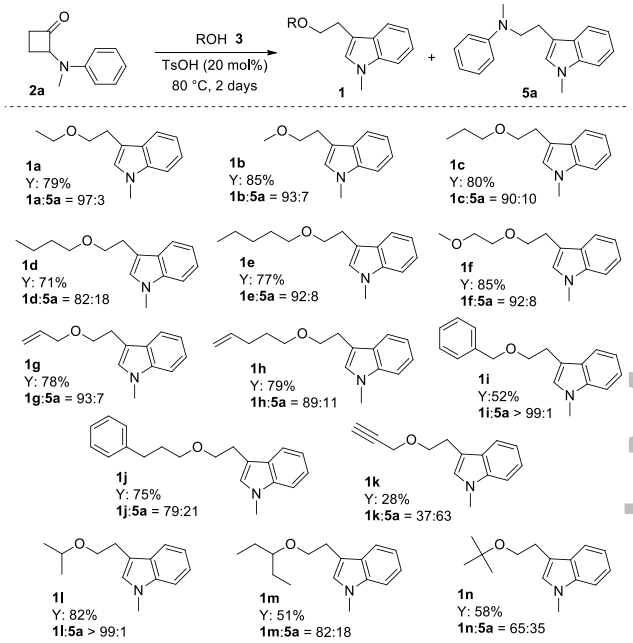
Entry	HX	T (°C)	**1a** (%)[b]	**4** (%)[b]	**5a** (%)[b]	**6** (%)[b]	**2a** (%)[b]
1	TsOH	R. T.	1	0	6	0	93
2	TsOH	40	3	0	6	0	91
3	TsOH	60	67	0	23	0	10
4	TsOH	80	88(79)[c]	0	3	9	0
5	HI	80	52	14	30	0	4
6	HBr	80	44	9	37	2	8
7	HCl	80	8	28	49	6	9
8	CF_3COOH	80	77	1	12	2	8
9	MsOH	80	50	0	0	41	9
10	$(PhO)_2POOH$	80	43	0	53	0	4

[a] Reaction conditions: **2a** (0.571 mmol), **3a** (1 mL), HX (0.114 mmol). [b] Determined by GC-MS. [c] Isolated yield.



Scheme 2. Proposed competing pathways leading to the formation of **5a** and **4**.

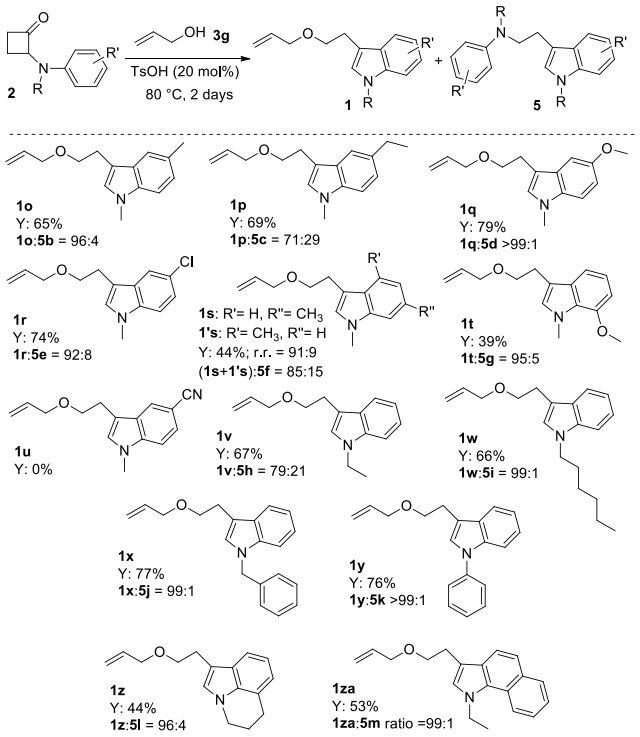
Having established the optimized conditions, the broad scope of the reaction was then demonstrated using a selection of alcohol substrates **3** in reaction with **2a** (Scheme 3). Under the standard conditions, all reactions implicating a primary alcohol **3a-j** were successful, highlighting the broad generality of this protocol: good-to-excellent product selectivity (**1**:**5a** ratio from 79:21 to >99:1) was observed, leading to good-to-high isolated chemical yields (52-85%) of derivatives **1a-j**. Propargylic alcohol **3k** also participated in the reaction, albeit less efficiently, to provide indole **1k** (28% yield; **1k**:**5a** ratio 37:63). The reaction also worked well with secondary alcohols **3l** and **3m**, giving **1l** (82% yield; **1l**:**5a** ratio > 99:1) and **1m** (51% yield; **3m**:**5a** ratio 82:18) respectively. Even the sterically congested *tert*-butyl alcohol **3n** reacted to afford the corresponding indole **1n** (58% yield; **1n**:**5a** ratio 65:35).



Scheme 3. Substrate scope for alcohols. Reaction conditions: **2a** (0.571 mmol), alcohol **3** (1 mL), TsOH (0.114 mmol). Isolated yields for **1** are given. The **1:5a** ratio was determined by GC-MS.

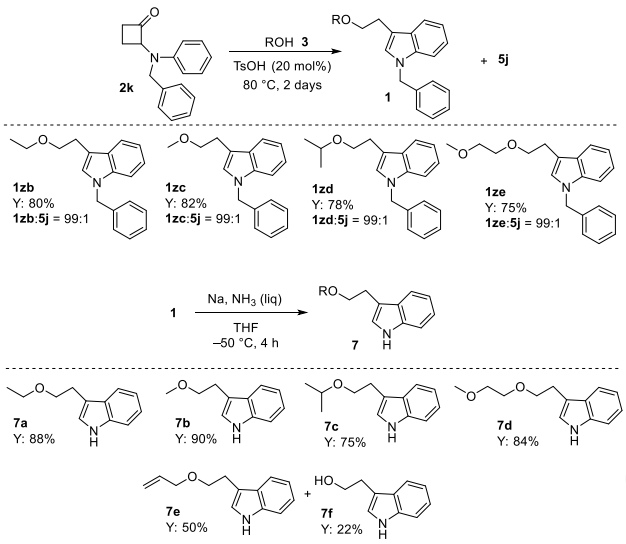
Next, the scope of the reaction was probed using different *N*-alkyl-*N*-aryl-α- aminocyclobutanones **2** in reaction with allyl alcohol **3g**. *N*-methyl substrates **2o-q**, whose *N*-aryl group bore an electron-donating group at the *para* position, performed well and furnished the corresponding 3-(2-allyloxyethyl)-1-methylindoles **3o-q** in 65-79% yield (**3**:**5** ratio from 71:29 to >99:1). Substrate **2r**, bearing a *p*-chlorophenyl group, was also transformed successfully into **1r** (74% yield; **31r**:**5e** ratio 92:8), although the substrate with a *p*-cyanophenyl group, **1u**, failed to react. The *meta* substituted substrate **2s** gave a mixture of the two regioisomers **3s** and **3's** (r.r. = 91:9) in a moderate 44% yield (**2s**+**2's**):**5g** ratio 85:15). The reaction also succeeded with substrate **2t**, bearing

a substituent at the *ortho* position, to provide indole **1t** in 39% yield (**1t**:**5h** ratio=95:5).
Other *N*-alkyl-*N*-aryl-α- aminocyclobutanones were examined: substrates **2v-y**, with *N*-ethyl, hexyl, benzyl and phenyl substituents, respectively, were all transformed efficiently into the corresponding indoles **1v-y**, in 66-76% yield (**3**:**5** ratio from 79:21 to >99:1). Substrate **2z**, a tetrahydroquinoline derivative, also participated in the reaction to furnish the tricyclic product **1z** in 44% yield (**1z**:**5l** ratio 94:6). Moreover, *N*-ethyl-*N*-(α-naphthyl)-α-aminocyclobutanone **2za** was transformed into the 1*H*-benz[*g*]indole derivative **1za** in a respectable 53% yield (**1za**:**5m** ratio 99:1).



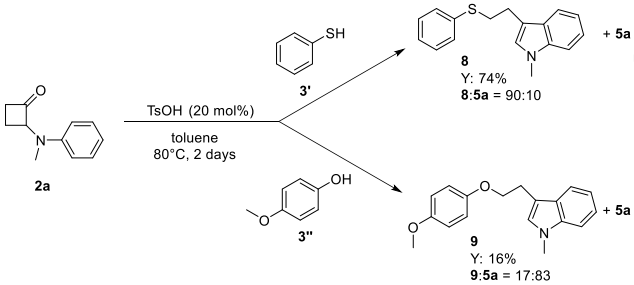
Scheme 4. Substrate scope for α- aminocyclobutanones. Reaction conditions: **2** (0.571 mmol), allylic alcohol **3g** (1 mL), TsOH (0.114 mmol). Isolated yields for **1** are given. The **1**:**5** ratio was determined by GC-MS.

The methodology was conveniently adapted to provide a viable synthesis of NH indoles in only two steps (Scheme 5).[12] A selection of primary (**3a, 3b, 3f**) and secondary (**3l**) alcohol substrates were reacted with *N*-benzyl-*N*-phenyl-α-aminocyclobutanone **2k** in the standard conditions to provide *N*-benzyl-3-(2-alkoxyethyl)indoles **1zb-ze** in high yields (75-80%). Competition from the formation of tryptamine **5f** was minimal. Selective debenzylation of each derivative **1zb-ze** was achieved via Birch reduction, through treatment with sodium and ammonia in THF at –50 °C, leading smoothly to the target NH indoles **7a-d** in good to high chemical yields (75-90%). Birch reduction of 3-(2-allyloxyethyl)-1-benzylindole **1x** gave **7e** in a lower yield (50%) due to a competing deallylation reaction, testified by the concomitant formation of 1*H*-indole-3-ethanol **7f** (22% yield).



Scheme 5. Two-step reaction sequence for the synthesis of NH indoles **7**.

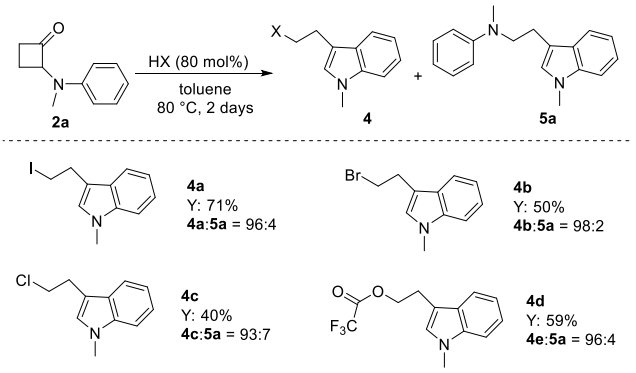
In all of the 3-(2-alkoxyethyl)indole libraries described above, the alkoxy moiety was provided in the form of an alcohol. We examined the scope of the reaction protocol further by considering the TsOH-mediated reaction of **2a** with two other types of nucleophile (Scheme 5). Pleasingly, the reaction with thiophenol **3'** provided the requisite phenylthioethyl derivative product **8** in good yield (74%). On the other hand, the reaction with *p*-methoxyphenol **3''** gave a low yield (16%) of the *p*-methoxyphenoxy derivative **9** and a significant amount of the tryptamine **5a** was formed via the competitive reaction. The lower nucleophilicity of **3''** may explain the reactivity erosion.



Scheme 6. Synthesis of indoles **8** and **9**. A small amount of toluene was added to reduce the viscosity of the reaction mixture.

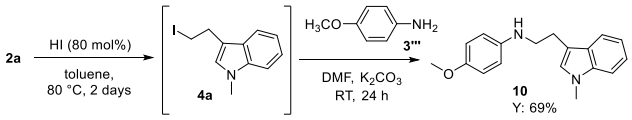
3-(2-Halogenoethyl)indoles are widely used in organic synthesis,[13] so we returned to the formation of compounds **4** from the reaction of **2a** and **3a** in the presence of halogen acids (Table 1). It appeared plausible that by increasing the amount of acid reagent and removing the alcohol, this process might provide a convenient access to compounds such as **4**. To examine this hypothesis, **2a** was treated with 80 mol% of each of the acids HI, HBr and HCl. Gratifyingly, the

corresponding 2-halogenoethyl derivatives **4a-c** were obtained in moderate-to-good yields (40-71%; Scheme 7). This procedure also provided access to 2-(1-methyl-3-indolyl)ethyl trifluoroacetate **4d** (59% yield). The reactions of TsOH and MsOH failed to produce the corresponding sulphonate esters; the only products obtained were tryptamine **5a** and *N*-methyl aniline **6**.



Scheme 7. Substrate scope of indoles **4**. Reaction conditions: **2a** (0.571 mmol), toluene (0.7 mL), HX (0.457 mmol). Isolated yield for **4** are given. The **4:5a** ratio determined by GC-MS. A small amount of toluene was added to reduce the viscosity of the reaction mixture.

Finally, to demonstrate the synthetic utility of this protocol, **4a** was prepared as above from **2a** and HI and was reacted directly with *p*-methoxyaniline **3'''** to furnish the tryptamine derivative **10** in good yield (69% for two steps; Scheme 8).



Scheme 8. A two-step preparation of tryptamine **10**.

In summary, we have developed a rapid and efficient methodology for the construction of highly functionalized *N*-alkyl-3-(2-alkoxyethyl)indoles via a Brønsted acid catalysed cascade reaction of α-arylaminocyclobutanones with alcohols. Minor adaptations of the protocol allow access to NH indoles as well as representative 2-arylthioethyl-, 2-aryloxyethyl-, 2-halogenoethyl- and 2-trifluoroacetoxyethyl- derivatives through an expanded substrate scope. This new synthetic method offers significant modularity, allowing expedient access to a wide range of complex indole-based heteroaromatic frameworks.

Experimental Section

General procedure for the preparation of indoles 1: A solution of **2** (0.571 mmol) and TsOH (0.114 mmol) in the alcohol of choice (1 mL) was stirred in a sealed tube reactor at 80 °C for 48h. The crude mixture, without aqueous work- up, was directly purified by flash column chromatography (SiO_2, petroleum ether/ether = 10:1→1:1) to give compound **1**.

General procedure for the preparation of indoles 4: A solution of **2a** (0.571 mmol) and the acid (0.456 mmol) in toluene (0.7 mL) was stirred in a sealed tube reactor at 80 °C for 48 h. The resulting reaction mixture was concentrated in a vacuum, diluted with ethyl acetate and washed with saturated $NaHCO_3$ aqueous solution. The organic layer was dried over anhydrous Na_2SO_4, filtered and concentrated to give the crude product **4** which was purified by column chromatography (SiO_2, petroleum ether/ether = 10:1→1:1).

General procedure for the preparation of indoles 7: A solution of *N*-benzylindole **1** (0.24 mmol) in THF (2 mL) was added dropwise to a mixture of THF (2 mL) and ammonia (3 mL) containing sodium (1.4 mmol) at –78 °C. The mixture was stirred at –50 °C for 4 h then cooled again to –78 °C and quenched by addition of saturated aqueous NH_4Cl solution. The solvent was evaporated under reduced pressure, water was added and the mixture was extracted with ether. Combined organic layers were dried over anhydrous Na_2SO_4, filtered and concentrated. The residue was purified by column chromatography (SiO_2, petroleum ether/ether = 10:1→1:1) to give indole **7**.

General procedure for the preparation of indoles 8 and 9: A solution of **2a** (0.571 mmol), TsOH (0.114 mmol) and thiophenol **3'** or *para*-methoxy phenol **3''** (0.856 mmol) in toluene (0.7 mL) was stirred in a sealed tube reactor at 80 °C for 48 h. The crude mixture, without aqueous work- up, was directly purified by flash column chromatography (SiO_2, petroleum ether/ether = 10:1→1:1) to give compound **8** or **9**. Yields refer to chromatographically pure materials.

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[12] This methodology cannot be evaluated at present for the direct preparation of NH-indoles from the NH-bearing substrates *N*-aryl-α-aminocyclobutanones, due to the absence of a convenient access to such starting materials in the literature. The standard entry to the α-aminocyclobutanone core employs the condensation of the requisite amine or aniline with α-hydroxycyclobutanone or its bis(trimethylsilylated) enol ether derivative; this chemistry is restricted principally to secondary anilines and does not work satisfactorily with primary anilines. See references 11c, 11e and 11g.

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UPDATE

Brønsted acid catalysed synthesis of 3-(2-alkoxyethyl)indoles from α-arylaminocyclobutanones and alcohols

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