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Exploiting 3-Methyleneisoindolinones as In Situ Generated Reactive Intermediates in the Synthesis of Tetrasubstituted Carbon Center

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In this study, we describe the operationally simple construction of a tetrasubstituted carbon center utilizing in situ formation of 3-methyleneisoindolinones as reactive intermediates. An acid-catalyzed Meyer–Schuster rearrangement of isoindolinone-based propargylic alcohols followed by an intermolecular Friedel–Crafts alkylation assembles the 3,3-disubstituted isoindolinones, an important architectural motif found in numerous biologically active compounds. Highly inert to structural changes in building blocks, this robust transformation allows the quick build-up of a library of compounds with an embedded isoindolinone core. In addition, the in situ formation of the activated intermediate allows a selective installation of various structural characteristics .

Functionalization at the C3 position of isoindolinones is undeniably the most explored method for the construction of isoindolinones containing a tetrasubstituted carbon center. To a large extent, this encompasses strategies based on either nucleophilic substitution of parent 3-hydroxyisoindolinones or electrophilic transformation at the α-position of 3-substituted isoindolinones.[1] While the formation of highly reactive *N*(acyl) ketimine is a prerequisite for the nucleophilic attack of various types of nucleophiles, electrophilic functionalization requires an electron-withdrawing group at the C3 position, thus imposing severe limitations on product variability and subsequent selective modifications. Although these methods still stand at the forefront of the synthesis of isoindolinones containing a tetrasubstituted carbon atom, the development of efficient alternatives that would bridge over the current limitations and at the same time allow quick build-up of molecular complexity is highly desirable.[2,3] In particular, the direct transformation of compounds with embedded isoindolinone core is an attractive method as it provides an impetus for the development of new methodologies, but at the same time avoids complex synthetic manipulations. In this respect, we considered the catalytic transformations of 3-methyleneisoindolinones as a viable method for the construction of 3,3-disubstituted isoindolinones. The presence of an activated exocyclic double bond in 3-methyleneisoindolinones has been previously exploited in several mechanistically different reactions.[4–6] But surprisingly, there are only a few reported examples of such an approach in the construction of C3-disubstituted isoindolinones. Existing examples include an enantioselective photoinduced Povarov reaction of *N*-aryl α-amino acids with 3-methyleneisoindolinones,[7] providing tetrahydroquinoline-based products and intramolecular Pictet–Spengler type annulation of indole-linked 3-methyleneisoindolinones.[8] Another example is the enantioselective formal (4 + 2) cycloaddition resulting in spiro chroman-isoindolinones.[9] Although 3-methyleneisoindolinones hold high synthetic value and serve as a promising platform for further research, their scarce utility could be attributed to a lack of a general protocol that would allow their rapid synthesis and concomitant conversion into 3,3-disubstituted isoindolinones. Much of the work on the synthesis of 3-methyleneisoindolinones has focused on the metal-catalyzed annulations, with only a handful of examples of their metal-free counterpart emerging in the last decade.[10–14] Thus, we wondered if an acid-catalyzed in situ generation of 3-methyleneisoindolinones, followed by direct functionalization at the C3 position, would allow structural variability unreachable within the currently available synthesis pool.

In light of the absence of a more general protocol for the synthesis of tetrasubstituted carbon centers within an isoindolinone skeleton based on the functionalization of 3-methyleneisoindolinones and encouraged by our work in this field,[15] we devised a method that exploits a Meyer–Schuster rearrangement of isoindolinone-based propargylic alcohols (Scheme 1).

The Meyer–Schuster rearrangement represents an acid-catalyzed disposition and transformation of the hydroxyl group in propargylic alcohols,[16–25] resulting in α,β-unsaturated carbonyl compounds.[26–28] Applying this transformation to 3-alkynyl-3-hydroxyisoindolinones gives 3-methyleneisoindolinone

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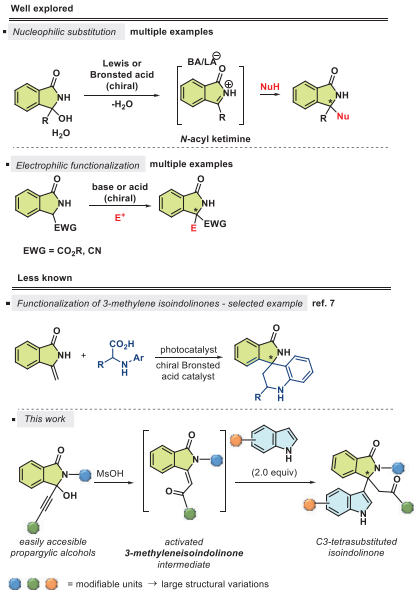


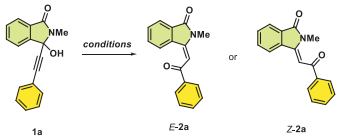
Scheme 1. Current strategies toward 3,3-disubstituted isoindolinones.

derivatives capable of undergoing Friedel–Crafts alkylation, resulting in 3-aryl(alkyl)-3-alkyl isoindolinones. The resulting products bearing a 2-oxo pendant chain have structural similarities with various biologically active compounds, thus serving as a base for further molecular decoration.[29–45]

Although the Meyer–Schuster rearrangement has been a synthetically well-explored method, its application to isoindolinone-derived propargylic alcohols is unknown. Thus, at the beginning, we set out to screen the conditions to access 3-methyleneisoindolinone, a precursor for further transformation. All reactions were performed using *N*-methyl substituted 3-alkynyl-3-hydroxy isoindolinone **1a** as a model compound (Table 1). A set of Bronsted acids was tested (entries 1–8), out of which MsOH, phenylphosphinic acid (PPA), and diphenyl phosphite (DPP) showed greater capacity to catalyze the Meyer–Schuster rearrangement. Compared to these catalysts, the used Lewis acids did not initiate the reaction at all (entries 9 and 10), or the product was formed in a lesser amount (entries 11 and 12). Switching from acetonitrile to other solvents had a detrimental effect on the yield of the reaction (entries 13–17). In the same line, a lower temperature (entry 18) is not sufficient to activate the rearrangement. On the other hand, reducing the amount of the MsOH catalyst gave the desired product in high yield (entries 19–20) while the presence of a drying agent, as expected, suppressed the rearrangement (entry 21).

The reaction with stronger acids such as *p*-TsOH and MsOH in acetonitrile gave exclusively an *E*-stereoisomer, whereas an *E*:*Z* mixtures were obtained with other acids and solvents. The observed stereoisomeric distribution could be attributed to the smaller steric clash between a carbonyl group and the *N*-Me substituent in *E*-**2a** compared to the opposite stereoisomer *Z*-**2a**. These results indicate the dynamic shift between the two isomers, most probably through *N*(acyl) ketimine intermediate, which is also a good electrophile capable of undergoing a subsequent Friedel–Crafts alkylation with a suitable nucleophile. This encouraged us to screen the various types of electron-rich

Table 1. Screening of reaction conditions[a)]



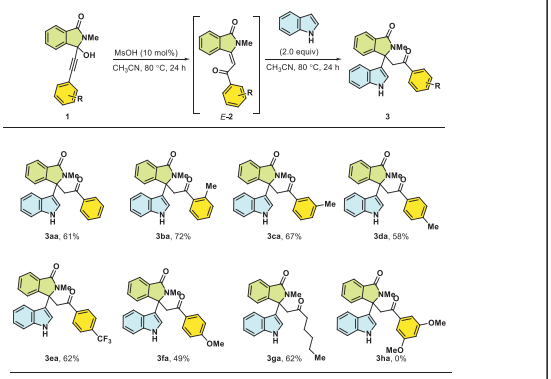
Entry	Catalyst	Solvent	Temperature (°C)	Time (h)	Yield/%	*E*:*Z*
1	TsOH	CH_3CN	25	24	55	>20:1
2	TsOH	CH_3CN/H_2O	25	48	6	–
3	TsOH	CH_3CN/H_2O	80	48	Traces	–
4	MsOH	CH_3CN	80	24	91	>20:1
5	PPA[b)]	CH_3CN	80	24	95	8:1
6	DPP[c)]	CH_3CN	80	24	91	10:1
7	$PhCO_2H$	CH_3CN	80	24	Traces	–
8	AcOH	CH_3CN	80	24	Traces	–
9	$AlCl_3$	CH_3CN	80	24	n.r.	–
10	TMSCl	CH_3CN	80	24	Traces	–
11	BF_3xEt_2O	CH_3CN	80	24	43	4:1
12	$SnCl_2$	CH_3CN	80	24	41	5:1
13	MsOH	Cyclohexane	80	24	74	8:1
14	MsOH	DCE	80	24	39	4:1
15	MsOH	Toluene	80	24	61	4:1
16	MsOH	$CHCl_3$	80	24	26	3:1
17	MsOH	Hexane	80	24	83	8:1
18	MsOH	CH_3CN	40	24	Traces	–
19[d)]	MsOH	CH_3CN	80	24	95	>20:1
20[e)]	MsOH	CH_3CN	80	24	80	>20:1
21[f)]	MsOH	CH_3CN	80	24	43	8:1

[a)] Reactions were performed using **1a** (0.2 mmol) and 20 mol% of catalyst in 2.0 mL of solvent, [b)] Phenylphosphinic acid, [c)] Diphenyl phosphite [d)] 10 mol% of MsOH, [e)] 5 mol% of MsOH, [f)] 4 Å molecular sieves were used as an additive.

aromatics and heteroaromatics to construct the tetrasubstituted carbon center.

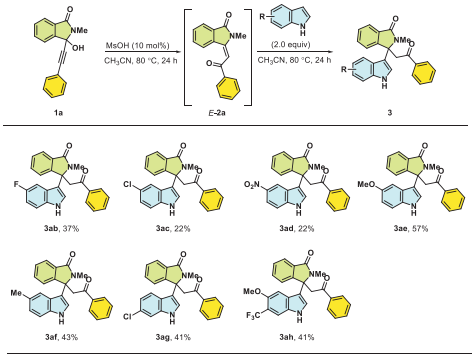
The successful addition of various nucleophiles, especially indoles, to in situ formed *N*(acyl) ketimines directed our attention to the extent of the proposed strategy.[46] As a first choice of nucleophile, indole was used throughout the series of *N*-Me propargylic alcohols **1** with varying substituents at the acetylenic part (Table 2). Two equivalents of indole were enough for the full consumption of the enone intermediate *E*-**2** within 24 h. The successful formation of **3aa** in good yield over two steps has proven the initial hypothesis about the 3-methyleneisoindolinones as suitable precursors in the formation of the C3-tetrasubstituted carbon center. Disposition of the methyl group around the phenyl ring with concomitant formation of products **3ba**–**3da** had a negligible effect on the effectiveness of the process. On the other hand, a comparison of the reactivity of **1ea** bearing an electron-withdrawing group with methoxy-containing **1fa** showed a slight preference for the diminished electron charge distribution around the center of reactivity. An alkyl group is also well tolerated in this transformation, as exemplified by

Table 2. Substrate scope–***acetylenes***.



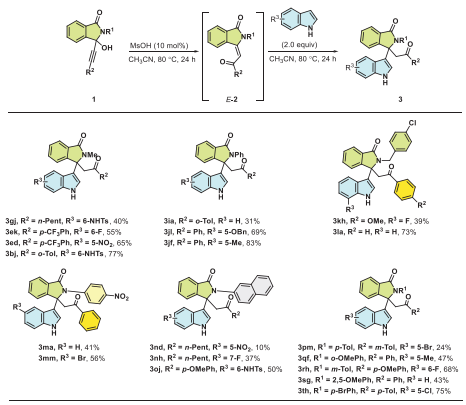
Reactions were performed using **1** (0.2 mmol) and 10 mol% of catalyst in 2.0 mL of acetonitrile.

Table 3. Substrate scope –***indoles***.



Reactions were performed using **1** (0.2 mmol) and 10 mol% of catalyst in 2.0 mL of acetonitrile.

Table 4. Substrate scope–***variations***.



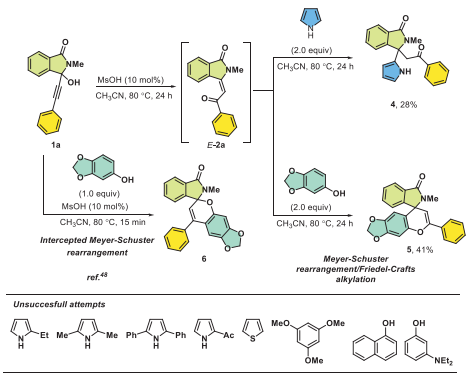
Reactions were performed using **1** (0.2 mmol) and 10 mol% of catalyst in 2.0 mL of acetonitrile.

the formation of **3ga**. The only unsuccessful attempt was with 3,5-dimethoxy substituted propargylic alcohol **1ha**, where not only was the final product not observed, but even the Meyer–Schuster rearrangement did not give enone intermediate. This substitution pattern was also troublesome in other mechanistically distinct reactions under similar reaction conditions.[47]

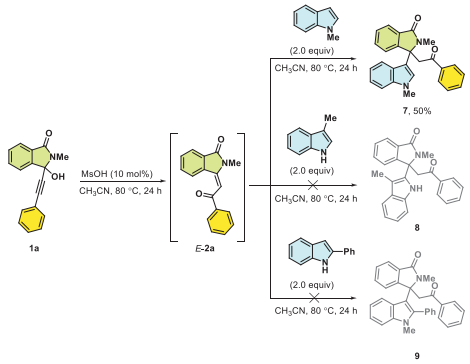
Next, we screened the influence of the steric/electron distribution change in indoles on the outcome of the nucleophilic addition (Table 3). The available indoles with substitutions at positions 5 and 6 were all tolerated in this transformation giving the final products in varying isolated yields. Electron-withdrawing groups at the C5 position had a detrimental effect on the effectiveness of the reaction as products **3ab**–**3ad** were formed in the 22%–37% isolated yield range. On the other hand, the presence of electron-donating groups increased the isolated yield of products **3ae**--**3af**. A comparable result was obtained for indoles with substitution at the C6 position, with product **3ag** isolated in 41% isolated yield. Finally, reaction with C5/C6 disubstituted indole afforded the product **3ah** in 41% isolated yield. It should be mentioned that in all cases the Meyer–Schuster rearrangement proceeded without difficulties and the limiting step was the addition of indoles to the enone intermediate, during which the intractable products were also formed, thus diminishing the isolated yields.

Finally, we performed reactions where we altered the *N*-substituent, acetylenic part, and the indole or all of them simultaneously. The products are grouped in six different categories, where in five of them the *N*-substituent is always the same with variable indole and acetylenic portion while in the last category, all three transformable moieties were changed. Table 4 summarizes the results. The products were obtained in the range between 10%–83% isolated yield, depending on the combinations of electrophile/nucleophile pairs used. The lowest amount of product was obtained for the **3nd** bearing *N*-naphthyl group and the aliphatic propargylic portion using the 5-nitro indole as a nucleophile. On the other hand, the highest-yielding combination used **1j** as the starting reagent and 5-methyl indole, a combination that gave the final product **3jf** in 83% isolated yield. Although a respectable number of C3-disubstituted products was obtained, there is no observable pattern that would aid in modeling a general conclusion about the effect of each component of the final product on the effectiveness of this transformation. Nonetheless, the variable product yield distribution and considering the endless possible combinations of two structurally modifiable reagents, it is easy to architect specific structural features of tetrasubstituted isoindolinones. For that reason, the described methodology offers unparalleled potential in the synthetic design of even more complex compounds within this class, as it avoids vigilant modeling of reaction partners.

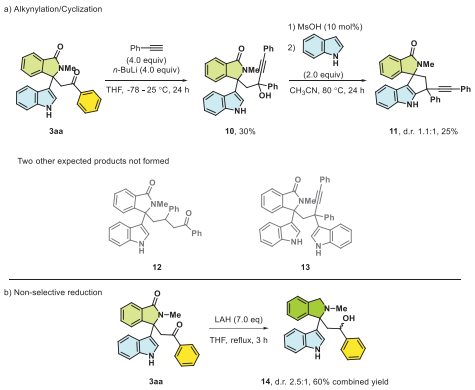
One of the main advantages of the described methodology is the in situ generation of an active electrophile, thus leaving great potential for accessing the high structural variability of the final product depending on the nucleophile used. For that reason, we turned our attention to other heteroaromatic and aromatic nucleophiles suitable to undergo the Friedel–Crafts alkylation. The seemingly wide range of possible nucleophiles turned out to be rather narrow because out of several screened nucleophiles, only pyrrole afforded the C-3 disubstituted product **4**, but in rather moderate isolated yield (Scheme 2). Any alteration in



Scheme 2. Substrate scope–***nucleophiles***.



Scheme 3. Control experiments.



Scheme 4. Product transformations.

the structure of pyrrole did not result in the addition, and only the products of Meyer–Schuster rearrangements were observed in the reaction mixtures. The same outcome was observed when thiophene was used as the nucleophile. It seems that there is a strong steric influence on the trajectory of the nucleophile approach to the sterically congested *N*(acyl) ketimine intermediate. Despite the planar structure of *N*(acyl) ketimine, the substitution of the pyrrole prevents its suitable alignment for the addition to occur. On the other hand, thiophene might not be nucleophilic enough for the reaction to occur in the first place. The unproductive trend continued with electron-rich benzene derivatives such as 1,3,5-trimethoxy benzene and 1-naphthol, otherwise applicable nucleophiles in the construction of tetrasubstituted isondolinones. Surprisingly, sesamol eventually participated in the product formation, but instead of the expected 2-oxo-bearing product, chromene **5** was isolated from the reaction mixture. Undeniably, the nucleophilic attack occurs at the most activated C-position of sesamol, but the concomitant *O*-acylation followed by dehydration leads to the chromene product. Interestingly, this process represents the complementary method for spiro-chromene synthesis based on the order of the addition of reagents. As exemplified in this work, if the Meyer–Schuster rearrangement occurs first, the following Friedel–Crafts alkylation and the *O*-acylation result in the formation of product **5**. In the case of intercepting the Meyer–Schuster rearrangement with sesamol, followed by *O*-alkylation, the isomeric chromene **6** is formed.[48] Finally, we attempted the reaction with bisalkylated hydroxy aniline, but in this case, neither the desired product nor the chromene was observed.

Since the indole and its derivatives, together with pyrrole, were found to be the most effective nucleophiles, we performed the control experiments to explore the influence of the indole *N*-, C2-, and C3 substitution on the addition to the enone *E*-**2a** (Scheme 3). Despite the reduced nucleophilicity of *N*-methyl indole, the addition occurred at the C3 position, thus giving the product **7** in 50% isolated yield. On the other hand, in reaction with the skatole, addition through the C2 position did not occur to give the expected product **8**. Finally, C2-substituted indole was also unreactive in the same process, most presumably because of the high steric crowding around the nucleophilic site.

Since the formed products bear transformable functionalities, we exploited them in the product utility transformations (Scheme 4). Alkynylation of **3aa** afforded the propargylic alcohol **10** in 30% isolated yield. Even though only one diastereoisomer was isolated from the reaction mixture, we cannot rule out the possibility that both are formed during the reaction, with only one of them being isolated in larger quantity. As product **10** bears a propargylic unit, we envisioned three possible subsequent transformations using strong nucleophiles under acid-catalyzed conditions. In the first instance, either the ensuing Meyer–Schuster rearrangement will precede the Friedel–Crafts alkylation, giving the product **12**, or the intermolecular nucleophilic substitution of the hydroxyl group will be more favourable, thus yielding the bis-indole **13**. This type of substitution has been previously observed, and it was speculated that the strength of the nucleophile greatly impacts the type of product formed.[48] The third scenario is an intramolecular substitution through the C2 position of the indole moiety already embedded within the propargylic alcohol **10**. Indeed, subjecting it to acidic conditions in the presence of indole resulted in an intramolecular substitution product **11** as a 1.1:1 diastereoisomeric mixture. Formation of this tricyclic spiroisoindolinone represents a not-so-trivial task of construction of tetrasubstituted and quaternary carbon atoms, and additionally, it showcases the overall synthetic applicability of the described methodology in the construction of a tetrasubstituted center of chirality. Finally, subjecting the **3aa** to reaction with LAH resulted in a non-selective reduction. Both amide and ketone groups were reduced to the corresponding amino alcohol **14** as a separable mixture of two diastereoisomers in a 2.5:1 ratio and 60% combined yield.

In conclusion, we have developed a methodology for the construction of C3-disubstituted isoindolinones by exploiting the transformable character of in situ generated 3-methyleneisoindolinones, a transient species toward highly electrophilic *N*(acyl) ketimines. The synthetic pathway consisting of acid-catalyzed Meyer–Schuster rearrangement followed by intermolecular Friedel–Crafts alkylation afforded a series of products bearing a carbon atom substituted with four different chemical entities: heteroatom, aromatic, heteroaromatic, and aliphatic group. The structural variability of easily accessible propargylic alcohols combined with a large number of commercially available indoles provides a platform for the construction of diverse isoindolinones. Despite being limited to the heteroaromatic nucleophiles, this operationally simple two-step process, combined with exemplified product utility, may serve as a seed for future growth of the area in the synthesis of multiple and multisubstituted centers of chirality.

1. Experimental Section

General procedure for Meyer-Schuster/Friedel-Crafts relay: To a dispersion of 3-hydroxy-3-alkynylisoindolin-1-one **1** (0.20 mmol) in dry acetonitrile (2.0 mL), MsOH (0.0013 mL, 0.02 mmol) was added, and the resulting reaction mixture was vigorously stirred at 80 °C (oil bath) for 24 h. After complete consumption of the starting material (indicated by TLC), nucleophile (0.40 mmol, 2.0 equiv) was added and the resulting reaction mixture was stirred additional 24 h at the same temperature. Then, flash column chromatography on silica gel afforded the desired 3,3-disubstituted isoindolinones **3** (Supporting Information).

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Conflict of Interests

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available in the Supporting Information of this article.

Keywords: Acid catalyzed · Friedel–Crafts alkylation · Isoindolinones · Meyer–Schuster rearrangement · Tetrasubstituted carbon atom

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