Highly Regioselective Synthesis of Substituted Isoindolinones via Ruthenium-Catalyzed Alkyne Cyclotrimerizations

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Received: January 21, 2013; Revised: May 23, 2013; Published online: August 12, 2013

Supporting information for this article is available on the WWW under http://dx.doi.org/10.1002/adsc.201300055.

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Abstract: (Cyclooctadiene)(pentamethylcyclopentadiene)ruthenium chloride [Cp*RuCl(cod)] has been used to catalyze the regioselective cyclization of amide-tethered diynes with monosubstituted alkynes to give polysubstituted isoindolinones. Notably, the presence of a trimethylsilyl group on the diyne generally led to complete control over the regioselectivity of the alkyne cyclotrimerization. The cyclization reaction worked well in a sustainable non-chlorinated solvent and was tolerant of moisture. The optimized conditions were effective with a diverse range of alkynes and diynes. The 7-silylisooindolinone products could be halogenated, protodesilylated or ring opened to access a range of usefully functionalized products.

Keywords: alkyne; amide tether; cyclotrimerization; isoindolinones; ruthenium; trimethylsilyl group

Introduction

Substituted isoindolinones have recently generated considerable interest because of their diverse biological activities, including the inhibition of angiogenesis,[1] tumour necrosis factor production,[2] MDM2-p53 protein-protein interactions,[3] hypoxia-inducible factor-1α,[4] and histone deacetylase.[5] The majority of existing protocols for isoindolinone synthesis require the construction of a γ-lactam adjacent to a preformed aromatic core.[6] Recent examples include the one-pot transformation of 2-halobenzaldimines into chiral 3-substituted isoindolinones and the Ni-mediated cyclization of N-benzoyl aminals in the presence of a stoichiometric Lewis acid.[7,8] However, the inevitable limitation of these approaches is the accessibility of the arene starting material itself. The synthesis of polysubstituted arenes is often non-trivial, frequently requiring numerous steps, the use of protecting group strategies and/or functional group interconversions.

The transition metal-catalyzed [2+2+2] cyclotrimerization of alkynes is emerging as an elegant, atom efficient and convergent approach to the synthesis of highly substituted arenes.[9] The strategy allows for the regioselective synthesis of compounds that would be extremely difficult to make via traditional aromatic chemistry. The regioselectivity of a cyclotrimerization is normally controlled by tethering two or three of the alkyne components together, so this strategy is best suited to the synthesis of bicyclic and tricyclic ring systems. This allows for the assembly of substituted multiple-ring aromatic compounds from alkyne precursors in a single step.

Yamamoto and co-workers have previously recognized the potential of alkyne cyclotrimerizations for the synthesis of isoindolinones bearing substituents on the aromatic ring.[10] They reported the cyclization of amide-tethered diynes 1 with monoynes 2 using Cp*RuCl(cod) 3 as the catalyst to give regioisomeric isoindolinones 4 and 5 (Scheme 1). In general the regioselectivity of the cyclotrimerization was poor to moderate, with the exception of a single example bearing a methyl group at R1. In addition, a significant limitation of this method is the use of 1,2-dichloroethane (DCE) as solvent, a substance which is potentially detrimental to human health and is generally avoided within industry.[11]
The aim of this study was to explore the regioselective synthesis of polysubstituted isoindolinones using more industrially viable reaction conditions, to establish the general applicability of the reaction, and to develop the synthetic potential of the cyclized products. On the basis of previously reported cyclizations we envisaged that the introduction of a trimethylsilyl group at $R_1$ in diyne $1$ would direct the regioselectivity of the cyclisation reaction effectively with a broad range of monoynes. The arylsilane unit present in the isoindolinone product could then be transformed using standard chemical techniques to access a variety of 7-substituted derivatives.

Results and Discussion

Diyne Synthesis

Initially several amide-tethered diynes $6$ were prepared by the coupling of propargylic amines $7$ with 3-(trimethylsilyl)propionic acid $8$, via the corresponding acid chloride (Scheme 2). Where necessary the corresponding amines were prepared using literature procedures.

Optimization

Various conditions were screened for the cyclotrimerization of diyne $6a$ with 1-hexyne $9a$ to form isoindoline none $10a$, and the results are summarized in Table 1. All reactions were conducted for 16 h at which point

![Scheme 1. Isoindolinone synthesis as reported by Yamamoto and co-workers.](image1.png)

![Scheme 2. Synthesis of diynes $6a$–$e$.](image2.png)

Table 1. Optimization of the cyclotrimerization of $6a$ and $9a$.

| Entry | Solvent | Equivalents of $9a$ | Catalyst | Catalyst loading [mol%] | Conversion $^{[a,c]}$ [%] | Ratio $^{[e]}$ |
|-------|---------|----------------------|----------|-------------------------|--------------------------|-------------|
| 1     | PhMe    | 4                    | RhCl(PPh$_3$)$_3$ | 5                       | $<5$                     | 9:1         |
| 2     | PhMe    | 4                    | Co$_2$(CO)$_8$  | 10                      | $<5$                     | 9:1         |
| 3     | CH$_2$Cl$_2$ | 4                  | Grubbs I       | 5                       | 5                        | n.d.        |
| 4     | DCE     | 4                    | Cp*RuCl(cod)  | 1                       | 5                        | n.d.        |
| 5     | neat    | 4                    | Cp*RuCl(cod)  | 3                       | 50                       | 3:2         |
| 6     | neat    | 4                    | Cp*RuCl(cod)  | 1                       | 100                      | 3:1         |
| 7     | CPME    | 4                    | Cp*RuCl(cod)  | 3                       | 100                      | 5:1         |
| 8     | CPME    | 4                    | Cp*RuCl(cod)  | 1                       | 60                       | 4:1         |
| 9     | CPME    | 2                    | Cp*RuCl(cod)  | 3                       | 100                      | 2:1         |
| 10$^{[e]}$ | CPME  | 4                    | Cp*RuCl(cod)  | 3                       | 100                      | 8:1         |
| 11$^{[e]}$ | CPME  | 2                    | Cp*RuCl(cod)  | 3                       | 100                      | 8:1         |
| 12$^{[e]}$ | CPME | 1.1                   | Cp*RuCl(cod)  | 3                       | 100                      | 9:1         |
| 13$^{[e]}$ | CPME | 2                    | Cp*RuCl(cod)  | 3                       | 100                      | 5:2         |
| 14$^{[e]}$ | CPME | 2                    | Cp*RuCl(cod)  | 3                       | 90                       | 5:1         |
| 15$^{[e]}$ | CPME/10% water | 2                  | Cp*RuCl(cod)  | 3                       | 70                       | 3:1         |
| 16$^{[e]}$ | water | 4                    | Cp*RuCl(cod)  | 3                       | 30                       | 3:1         |

$^{[a]}$ Determined by analysis of the crude $^1$H NMR spectrum.

$^{[b]}$ Conversion of $6a$ into $10a$ and $11$ (determined by crude $^1$H NMR without the use of an internal standard).

$^{[c]}$ Solvent dried over activated 4 Å molecular sieves and degassed.

$^{[d]}$ Cp*RuCl(cod) 3 was added to the reaction mixture at 0°C, which was then allowed to reach room temperature.

$^{[e]}$ Diyne $6a$ in CPME was added dropwise over 3 h to a stirring solution of $9a$ and 3 in CPME.
conversion and selectivity were determined by analysis of the crude \(^1\text{H}\) NMR spectrum.

The cyclotrimerization of diyne 6a and alkyne 9a was examined using four different literature procedures. Neither RhCl(PPh\(_3\))\(_3\), nor Co\(_2\)(CO)\(_8\) were effective in catalyzing the alkyne cyclotrimerization, with no measurable conversion of diyne 6a (entries 1 and 2).\(^{[16]}\) Treating diyne 6a with 5 mol\% Grubbs’ first generation catalyst and 4 equivalents of 1-hexyne 9a in dried, degassed CH\(_2\)Cl\(_2\) resulted in formation of the target isoindolinone 10a with only 5% conversion (entry 3).\(^{[17]}\) Treating diyne 6a with 1-hexyne 9a and 1 mol\% Cp*RuCl\(_2\)(cod) in dried, degassed DCE also gave isoindolinone 10a, again with 5% conversion of 6a (entry 4).\(^{[10]}\) Given that the latter procedure gave a similar conversion with a lower catalyst loading, Cp*RuCl\(_2\)(cod) was selected for subsequent optimization.

Interestingly, treating diyne 6a with 1-hexyne 9a and 1 mol\% Cp*RuCl\(_2\)(cod) with no solvent (neat) at 0°C gave isoindolinone 10a with a 50% conversion (entry 5). This suggests that using DCE as a solvent for this reaction is actually detrimental. In addition to the desired isoindolinone 10a, dimer 11 was also formed as a significant by-product.\(^{[12]}\)

Crucially, regioisomeric cyclotrimerization product 12 was not observed at all in the crude \(^1\text{H}\) NMR spectrum. The reaction under neat conditions reached completion within 16 h when 3 mol\% of catalyst 3 was used, and with a significant reduction in the proportion of homo-coupled product 11 produced (entry 6).

We were interested in using cyclopentyl methyl ether (CPME) as a solvent for this cyclization as it has been recently established as a safer and more environmentally benign alternative to many traditional organic solvents.\(^{[18]}\) As shown in entry 7, when the reaction was conducted in CPME with 3 mol\% of catalyst 3, diyne 6a was completely consumed within 16 h and an improved selectivity for the cross-coupled product 10a was observed. By comparison, the same reaction using only 1 mol\% catalyst resulted in a comparable level of selectivity, but a lower conversion (entry 8). Reducing the number of equivalents of 1-hexyne 9a to two resulted in the complete consumption of diyne 6a but also a significantly increased level of homo-coupling.

In an attempt to minimize the formation of dimer 11, diyne 6a was added dropwise over 3 h to a stirring solution of monoyne 6a and catalyst 3\(^{[19]}\) and this proved to be highly effective (entry 10). When using the 3-hour dropwise addition it was possible to reduce the number of equivalents of 1-hexyne 9a from four to two with no increase in homo-coupling (entry 11). A further reduction to 1.1 equivalents of 1-hexyne 9a did result in increased homo-coupling, but target isoindolinone 10a was still the major product (entry 12).

The cyclization of 6a and 9a was also effective when 2-MeTHF or MTBE were used as solvents, but in both cases a greater degree of homo-coupling of 6a was observed than with CPME (entries 13 and 14). The reaction proved to be relatively water tolerant, with a significant conversion and a reasonable selectivity observed when the reaction was conducted in the presence of 10% water (entry 15). Cyclization was even observed when the reaction was conducted in water as solvent (entry 16). This is important as it could enable the extension of the reaction to aqueous conditions for reactions of water-soluble substrates.

Following the optimization study the conditions described in entry 11 were taken as the “optimized” cyclization conditions as they required a reduced excess of monoyne and minimized the formation of dimer 11. Crucially this protocol did not require the CPME solvent to be either degassed or dried. This, together with the environmental benefits of CPME, makes this reaction a very practical method for the synthesis of isoindolinones. Dimer 11 could be readily separated from the desired product by flash column chromatography, and the optimized conditions described in entry 11 gave the target isoindolinone 10a in 81% isolated yield (Table 2, entry 1). This reaction was also scaled up to a 500-mg scale and isoindolinone 10a was isolated in 66% yield (428 mg product).

\textbf{Monoyne Scope}

The cyclization of 6a was then examined with a variety of monoynes using the optimized conditions described above to determine how robust the reaction was for a range of different substrates. Diyne 6a cyclized with a wide range of monoynes 9 as detailed in Table 2. Crucially, no evidence for the formation of regioisomeric isoindolinones was observed in any of the cyclization reactions. Alkyl monoynes 9a–e cyclized efficiently with 6a to give the corresponding isoindolinones 10a–e in good isolated yield (entries 1–5, 66–83%). Little formation of the undesired dimer 11 was observed, except in the reaction of tert-butylacetylene 9h, presumably due to high steric crowding about the monoalkyne. Carbamate 9f cyclized with 6a to give 10f in reasonable yield and with modest levels of homo-coupling (entry 6).

Ether 9g and acetal 9h both underwent cyclotrimerization with 6a, but with the formation of significant quantities of dimer 11. Propargylic alcohol 9i and methoxyacetylene 9j both failed to cyclize with diyne 6a, with only starting material being recovered in both cases. In addition to aliphatic monoynes, diyne 6a cyclized effectively with a broad range of aromatic monoynes. Electron-rich (entries 12, 13, 17 and 18), electron-poor (entry 16) and sterically hindered substrates (entries 12 and 14) could all be tolerated and products
were isolated in good yields (79–93%) with low levels of diyne homo-coupling. For most of these examples longer reaction times (up to 24 h), and in some cases higher catalyst loadings, were required to drive the reaction to completion. However the reactions with ortho-substituted arylacetylenes 9l and 9n reached...
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Table 3. Cyclizations involving diynes with different N-substituents\(^{[a]}\)

| Entry | R\(^1\) | R\(^2\) | 3 [mol\%] | Time [h] | Product 13 | Yield of 13 [%]\(^{[b]}\) | Ratio of 13:14 \(^{[d]}\) |
|-------|--------|--------|-----------|---------|-----------|----------------|----------------|
| 1     | t-Bu   | n-Bu   | 9a        | 3       | 13a       | 84             | 10:1           |
| 2     | t-Bu   | Ph     | 9k        | 4       | 13b       | 89             | >10:1          |
| 3     | t-Bu   | o-tolyl| 9l        | 3       | 13c       | 94             | >10:1          |
| 4     | H      | n-Bu   | 9a        | 10      | 13d       | 51 (90%\(^{[c]}\)) | 2:1            |
| 5     | H      | o-tolyl| 9l        | 10      | 13e       | 62 (90%\(^{[c]}\)) | 7:1            |

\(^{[a]}\) Reaction conditions: a solution of 6 in CPME was added dropwise to a stirring solution of 9 and 3 in CPME over 3 h at room temperature.

\(^{[b]}\) Isolated yield.

\(^{[c]}\) Determined by the analysis of crude \(^1\)H NMR spectra.

\(^{[d]}\) Conversion of diyne 6 to 13/14 (determined by crude \(^1\)H NMR without the use of an internal standard).

The cyclization of amide-tethered diynes bearing different N-substituents was also explored (Table 4). With doubly substituted diynes 6d and 6e, no homo-coupling of the diyne was observed and dropwise addition of the diyne to the reaction was unnecessary (entries 1–3). With 10 mol% of Cp*RuCl(cod), methyl-substituted diyne 6d cyclized with 1-hexyne 9a to form a 9:1 mixture of regioisomeric isoindolinones 15a and 16a (entry 1).

Ethyl-substituted diyne 6e reacted with 1-hexyne 11a with lower regioselectivity, giving a 2:1 mixture of isoindolinones 15b and 16b (entry 2). However, diyne 6e cyclized with 2-ethylthiophene 9l, to give a 5:1 mixture of isoindolinones 15c and 16c (entry 3). Interestingly, the presence of diastereotopic benzylic protons in the \(^1\)H NMR spectrum suggests that isoindolinone...
15c is a chiral molecule, presumably due to restricted rotation about the hindered biaryl unit.

The dependence of the cyclotrimerization on an SiMe₃ regiodirecting group was also investigated. Diyne 6f with a terminal methyl substituent reacted with 1-hexyne 9a under the optimized cyclization conditions to give isoindolinone 15d in 85% yield (entry 4). Crucially, there was no trace of the regioisomeric isoindolinone 16d by crude ¹H NMR. Similarly, diyne 6f cyclized with 2-ethyl toluene 9l to give isoindolinone 15e in 94% yield, with no evidence for the formation of regioisomer 16e (entry 5).

### Functional Group Manipulation of Cyclized Products

Conversion of the cyclized isoindolinone products into a number of synthetically interesting motifs was examined. Isoindolinone 10a was converted to aryl halides 17 and 18, in 79% and 90% yields, respectively, via an ipso substitution of the silyl group (Scheme 3).[22] Treatment of N-t-butyliisoindolinone 13a with triflic acid resulted in a simultaneous deprotection of the lactam and protodesilylation within 30 min to give N-H isoindolinone 20 in good yield.[23] Alternatively, treatment of 13a with iodine monochloride followed by deprotection with triflic acid gave 7-idoisoindolinone 19 in 83% yield. Thus, an N-t-Bu diyne can be used as an indirect method for the synthesis of N-H isoindolinones via this acid-mediated deprotection.

It was also possible to access a tetrasubstituted monocyclic benzene. Treatment of N-H isoindolinone 19 with di-i-ert-butyl dicarbonate gave N-Boc isoindolinone 21, which could be reduced with lithium borohydride to form N-Boc protected amino alcohol 22.

### Table 4. Cyclizations involving diynes with different alkyne substituents[a]

| Entry | Diyne 6 | R¹ | R² | R³ | [mol%] | Time [h] | Isolated products | Yield of (15 + 16) [%][b] | Ratio of 15:16[c] |
|-------|---------|----|----|----|--------|----------|------------------|--------------------------|-----------------|
| 1     | 6d      | SiMe₃ | Me | n-Bu 9a | 10  | 24       | 15a/16a         | 69                     | 9:1             |
| 2     | 6e      | SiMe₃ | Et | n-Bu 9a | 10  | 24       | 15b/16b         | 57                     | 2:1             |
| 3     | 6e      | SiMe₃ | Et | o-tolyl 9l | 10 | 24       | 15c/16c         | 73                     | 5:1             |
| 4     | 6f      | Me   | H  | n-Bu 9a | 3   | 16       | 15d[e]          | 85                     | > 20:1          |
| 5     | 6f      | Me   | H  | o-tolyl 9l | 3 | 16       | 15e            | 94                     | > 20:1          |

[a] Reaction conditions: A solution of 6 in CPME was added to a stirring solution of 9 and 3 in CPME over 1 min at room temperature.

[b] Isolated yield.

c] Determined by the analysis of crude ¹H NMR spectra.

d] Diyne 6f in CPME was added dropwise over 3 h to a solution of 9 and 3 in CPME.

e] Evidence of limited homo-coupling of 6f was observed in the crude ¹H NMR spectrum.
together with cyclic aminol 23, in a combined yield of 78% (Scheme 4). The preparation of mono-cyclic substituted arenes via tethered alkyne cyclotrimerizations has little precedent and such systems are somewhat difficult to access via traditional aromatic substitution reactions, highlighting the value of this strategy.[24]

Conclusions

In summary, we have demonstrated the regioselective synthesis of polysubstituted isoindolinones via the Cp*RuCl(cod)-catalyzed cyclotrimerization of amide-tethered diynes and monoynes. This cyclization is effective with a wide range of structurally diverse monoynes and was demonstrated to work with a variety of different diynes. We have also demonstrated that the cyclization products could be converted into a range of functionalized isoindolinones and a tetrasubstituted benzene derivative.

Experimental Section

Full experimental details are provided in the Supporting Information.

Cp*RuCl(cod)-Catalyzed Cyclization of a Diyne and a Monoyne

A solution of 6a (500 mg, 1.86 mmol) in CPME (11 mL) was added dropwise over 3 h to a stirring solution of 1-hexyne 9a (0.43 mL, 300 mg, 3.7 mmol) and Cp*RuCl(cod) (21 mg, 3 mol%) in CPME (7.7 mL) at room temperature. The reaction mixture was stirred for a further 13 h before being filtered through a silica pad, eluting with ethyl acetate. The solvent was removed under vacuum to give the crude product, which was purified by flash column chromatography (13:1 petrol:ethyl acetate) to give 2-benzyl-5-butyl-7-(trime-

Acknowledgements

This work was supported by the Engineering and Physical Sciences Research Council (Advanced Research Fellowship EP/E027289/1), together with GlaxoSmithKline (Industrial CASE Award) and the UCL PhD program in Drug Discov-

ery. We would also like to acknowledge Simon Peace (GSK) for helpful discussions.

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