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Graphical Abstract





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Synthesis of Star-Shaped Pyrrole-Based C₃-Symmetric Molecules *via* Ring-Closing Metathesis, Buchwald–Hartwig Cross-Coupling and Clauson–Kaas Pyrrole Synthesis as Key Steps

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ABSTRACT

We have demonstrated three contrasting synthetic strategies to assemble pyrrole-based C_3 -symmetric molecule. Here, ring-closing metathesis (RCM), Buchwald–Hartwig cross-coupling and Clauson–Kaas pyrrole synthesis have been used as key steps. Interestingly, readily available starting materials such as 4-aminoacetophenone, 4-bromo acetophenone and 4-nitro acetophenone have been used as starting materials. Grubbs' first generation catalyst (G-I) is useful for one-pot RCM and aromatization sequence without the involvement of additional reagents. We also report photophysical properties of these star-shaped molecules.

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Introduction

Trimerization of carbonyl compounds is instrumental to assemble C_3 -symmetric star-shaped molecules and they are found to be useful core units in conjugated dendrimers¹ and branched oligomers.¹⁻² Moreover, conjugated star-shaped molecules have been used in liquid crystals,³ photovoltaics,⁴ organic field-effect transistors (OFETs),^{1b,5} organic light-emitting diodes (OLEDs),^{1b,6} electroluminescent devices^{2a} and organic solar cells.^{6a,7} Several C_3 -symmetric molecules containing heterocycles (eg: carbazole, and N-heterocyclic carbenes) have been synthesized and utilized in electro-optical materials, and separation science.⁸ As part of our major program aimed at designing star-shaped C_3 -symmetric molecules, ^{1a,9} here, we conceived new strategies to pyrrole-based C_3 -symmetric molecules.

Synthesis of pyrrole derivatives is of great interest due to their applications in material science and pharmaceutical area. In addition, a number of biologically important molecules contain pyrrole as a core unit (Figure 1).¹⁰ For example, pyrrole moieties have played an important role in haemoglobin function to carry the oxygen in living organisms.¹¹ Pyrrole derivative, atorvastatin is used for reducing the cholesterol level and ketorolac has been used as nonsteroidal anti-inflammatory drug (NSAID).¹²



Figure 1. Bio-active molecules containing pyrrole moieties.

The metathesis strategy has emerged as one of the most useful tool for the carbon-carbon bond formation in synthetic organic chemistry. Ring-closing metathesis (RCM) is well suited to prepare the cyclic olefins in high yield.¹³ With the advent of structurally well-defined ruthenium based metathesis precatalysts (Figure 2) during the last two decades, assembly of heteroaromatics by intra and intermolecular C-C bond formation has gained momentum.¹⁴



Figure 2. Selected list of ruthenium-based metathesis catalysts.

In view of the importance of pyrroles, we conceived several strategies to C_3 -symmetric molecule **4** containing three pyrrole units. To the best of our knowledge there is no report available

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in the literature to the synthesis of pyrrole-based C_3 -symmetric star-shaped molecules. In our approach, pyrrole-based C_3 -symmetric star-shaped molecules are assembled by RCM, Buchwald–Hartwig cross-coupling¹⁵ and Clauson–Kaas pyrrole synthesis¹⁶ as key steps. We also studied photophysical properties of these C_3 -symmetric molecules.

Strategy

The retrosynthetic strategy to pyrrole-based star-shaped compound 4 is shown in Figure 3. C_3 -Symmetric star-shaped compound 4 could be assembled from hexa-allyl derivative 3 via RCM followed by aromatization. The hexa-allyl derivative 3 could be assembled from di-allyl ketone 2 by trimerization sequence which can be derived from readily available 4-amino acetophenone (1) (path A). Alternatively, C_3 -symmetric product 4 could be assembled from C_3 -symmetric tri-bromide 5 (Figure 3) via Buchwald-Hartwig cross-coupling reaction. However, the tri-bromo derivative 5 can be derived from a readily available 4-bromo acetophenone (6) by trimerization sequence. These three distinctly different approaches provide several opportunities to expand the chemical space of C_3 -symmetric pyrrole derivatives. Finally, application of three fold Clauson-Kaas pyrrole synthesis starting with 5 can generate the target compound 4.



Figure 3. Retrosynthetic routes to pyrrole-based C_3 -symmetric star-shaped molecule **4**.

Results and Discussion

To realize the strategy shown in Figure 3 (Path A), commercially available 4-amino acetophenone (1) was treated with allyl bromide in the presence of sodium hydride (NaH) in dimethylformamide (DMF) at room temperature for 1 h which delivered the N,N'-diallyl product 2 (68%). Then, the di-allyl compound 2 was subjected to trimerization sequence with thionylchloride (SOCl₂) in EtOH under reflux condition to generate the trimerized product 3 in 42% yield. To improve the yield of the compound 3, we choose another route. In this regard, we prepared tri-amine derivative 9 by using literature procedure.¹⁷ Star-shaped tri-amine derivative 9 was subjected to N-allylation by using allyl bromide in the presence of NaH in DMF at room temperature for 2 h to give the hexa-allyl product 3 in 72% yield. Later, it was treated with Grubbs first generation catalyst (G-I) in CH₂Cl₂ at room temperature for 2 h to obtain the ring-closing metathesis (RCM) product 4 (84%) which underwent aromatization. The major driving force for facile dehydrogenation sequence under the reaction conditions is the gain in aromatization energy (Scheme 1). Such type of non-metathetic behaviour of Grubbs catalyst is known in the literature.¹⁸ Notably, earlier approach to pyrroles¹⁹ involving

RCM require separate step for aromatization. Interestingly, our approach involving G-I catalyst does not require aromatization step which is better sustainable sequence.



Scheme 1. Synthesis of star-shaped compound 4 via RCM.

Later, alternate routes were developed to this star-shaped pyrrole containing compound **4**. In this regard, compound **9** was reacted with 1,4-dimethoxy tetrahydrofuran (**10**) in acetic acid under reflux condition for 6 h to deliver the pyrrole-based C_3 -symmetric compound **4** in 63% yield (Scheme 2).



Scheme 2. Synthesis of star-shaped pyrrole derivative **4** by using Clauson–Kaas pyrrole synthesis.

In another route, we prepared the tri-bromo derivative **5** by using the literature procedure from commercially available 4bromo acetophenone (**6**).²⁰ Subsequently, the tri-bromo compound **5** was subjected to Buchwald–Hartwig crosscoupling reaction with pyrrole (**11**) in the presence of potassium carbonate (K₂CO₃) and, copper powder in dimethyl sulfoxide (DMSO) at 130 °C under inert atmosphere to obtain the starshaped molecule **4** in 68% yield (Scheme 3). The approaches demonstrated here start with simple and readily available starting materials. Moreover, these approaches are imbeded with several diversity points in terms of strategies and coupling partners. Furthermore, RCM and aromatization occur in one-pot sequence unlike earlier reports.



Scheme 3. Synthesis of star-shaped pyrrole derivative **4** via Buchwald–Hartwig Cross-Coupling reaction.

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Time-resolved fluorescence measurements of star-shaped derivatives 3 and 4

Steady-state and time-resolved fluorescence measurements (Figure 4) were performed in dilute solutions contained in standard quartz cuvettes. The superimposability of excitation and absorption spectra of the samples suggests a high degree of purity of the compounds prepared. Emission spectrum for each compound is obtained, with excitation at the wavelength of the absorption maximum.



Figure 4: Lifetime decay of star-shaped pyrrole derivatives in dichloromethane. Emission monitored at $\lambda_{em} = 410$ nm, $\lambda_{ex} = 340$ nm.

Corresponding lifetime values are given in Table 1. The decay curves of compound **3** and **4** both show single exponential nature (Table 1). These two compounds were shown fluorescence studies in dichloromethane as a solvent. Here, we have observed N,N'-diallylated C_3 -symmetric product **3** showing longer lifetime than pyrrole-based star-shaped derivative **4**.

Table 1: Time-correlated single-photon counting (TCSPC) data analysis of C_3 -symmetric compounds.

compound	$\tau_1(ns)$	τ_2 (ns)	χ2
4	2.98 (100%)	-	1.04
3	4.58 (100%)	Ó	1.07

These two **3** and **4** star-shaped C_3 -symmetric derivatives have similar spectroscopic properties. Structural difference is in functionality at the end of star arm doesn't have any influence on these properties. These two symmetric molecules can be modified further in versatile way of molecular network structures.

Conclusion

We have demonstrated three useful methods to pyrrole-based star-shaped C_3 -symmetric molecule 4 by different routes. To this end, we used RCM, Buchwald-Hartwig crosscoupling reaction and Clauson-Kaas pyrrole synthesis as key steps. Readily available and simple starting materials such as 4amino acetophenone 1, 4-bromo acetophenone 6 and 4-nitro acetophenone 7 have been used. It is worth mentioning that 1st route involve three steps (Scheme 1) and gave 23% overall yield. 2nd Route rely on Clauson-Kaas pyrrole synthesis (Scheme 2) also involve same number of steps (overall yield 33%). Buchwald-Hartwig cross-coupling route (Scheme 3) gave better overall yield 46%, which involve two steps synthetic sequence. Metathesis route use expensive catalyst. We have employed G-I catalyst for one-pot RCM and aromatization sequence. The tactics and strategies developed here would provide an easy access to new chemical space in pyrrole area.

We also reported the photophysical properties of these starshaped molecules. Here, we have observed N,N'-diallyl C_3 symmetric product **3** showing more lifetime than pyrrole-based star-shaped derivative **4**.

Steady-state and time resolved fluorescence

Absorption and fluorescence spectra were recorded on a Jasco V530 spectrophotometer and a Varian Cary Eclipse fluorometer, respectively. Band widths of 5 nm were used on the excitation and emission sides for fluorescence measurements. The samples were excited at 340 nm. TCSPC measurements were performed on an IBH Fluorocube time resolved fluorescence spectrophotometer. A Nanoled emitting at 340 nm was used to excite the samples. Instrument response function was 800 ps. Further detail about the instrument is available elsewhere.²¹ The lifetime values were obtained by fitting the fluorescence decays to multiexponential functions²² by an iterative reconvolution technique using reduced χ^2 as the parameter for goodness of fit ($\chi^2 < 1.2$ for a good fit). The fitting function is as follows:

$$I(t) = I(0) \sum_{i} A_i e^{-t/\tau_i}$$

Where I(t) and I(0) denote the fluorescence intensities at time t and time zero after excitation, respectively. A_i and τ_i denote the amplitude and lifetime, respectively, of the i^{th} component of the decay.

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Supplementary Material

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/ ...

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Highlights

Ring-closing metathesis product which underwent aromatization.

Photophysical properties of star-shaped molecules are studied.

Accepter N,N'-Diallyl C_3 -symmetric product showing more lifetime than pyrrole-based star-shaped derivative.