

Towards the Synthesis of Polymeric Ladderphanes from Cyclopropene Monomers: A Model Study

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Abstract

Ladderphanes are special types of polymers characterized by parallel, covalently connected backbones that show exceptional rigidity, anisotropy, and potential utility in advanced materials. The work described here is a model strategy for the synthesis of a strain cyclopropene monomer, followed by Ring-Opening Metathesis Polymerization (ROMP) polymerization. The synthesis of strain cyclopropene is challenging and faces various difficulties, which are successfully overcome using different modification techniques. Due to their characteristic ring strain and high reactivity, cyclopropenes serve as attractive precursors for controlled polymerization. This study will help to design the synthesis of different cyclopropane derivatives, which may be highly useful for many organic transformations.

Keywords: Cyclopropene; Ladderphanes; Metathesis; Organic Synthesis; ROMP; Strained Hydrocarbons

Introduction

Ladderphane is defined as a double-stranded polymer composed of multiple layers of cyclophanes, in which the tethers are part of the polymeric backbone (Figure 1) (Luh *et al.*, 2023). Interestingly, if one considers the DNA molecule as an illustrative example of a ladder-like architecture, it can be viewed as a specialized form in which complementary base pairs are held together through hydrogen bonding interactions and connected perpendicularly to the deoxyribose phosphate backbones. In this structure, the sugar-phosphate backbones are stereochemically isotactic in nature. The spacing of adjacent base pair layers is about 3.4 Å, and this distance indicates that there should be strong interactions between adjacent layers of base pairs. These features could be advantageous in the plan and preparation of double-stranded DNA-like polymeric ladderphanes.

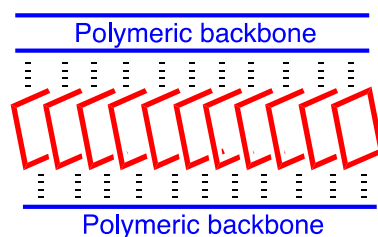


Figure 1: Polymeric Backbone of Ladderphane

Generally, three different methods have been used for the synthesis of polymeric ladderphanes (Figure 2). The initial strategy involves the simultaneous polymerization of a monomer bearing two reactive polymerizable units bridged by a rigid spacer. In this strategy, symmetric ladderphanes are obtained. The second and third protocols involve the synthesis of unsymmetrical ladderphanes where the two polymeric backbones are not identical but complementary. The latter method has been employed for replication to synthesize a range of condensation polymers with precisely controlled chain lengths and low dispersity.

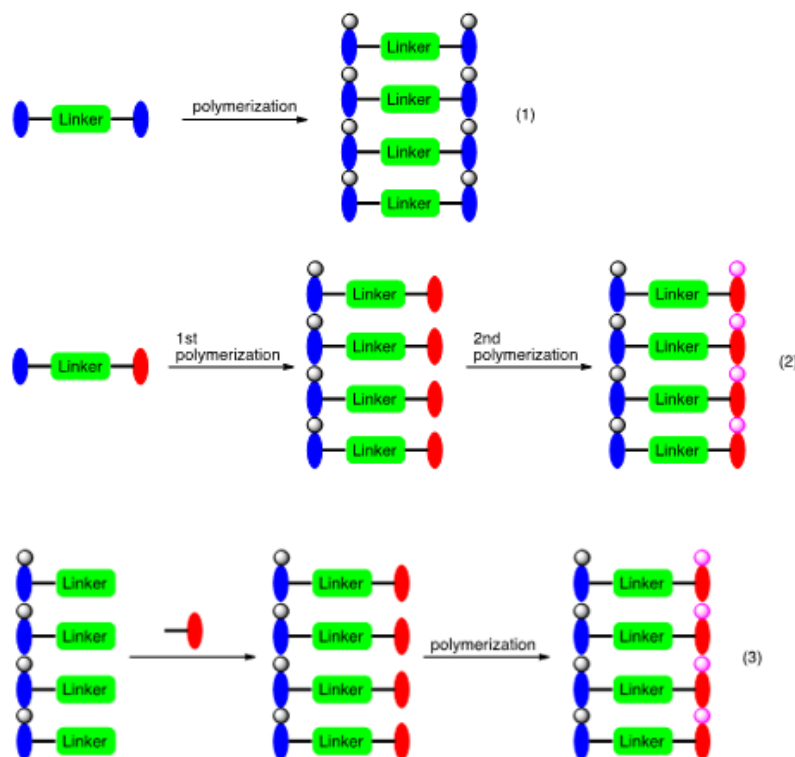


Figure 2: Different Strategies for the Synthesis of Polymeric Ladderphanes

Ring-opening metathesis polymerization, commonly abbreviated as ROMP, holds a leading position in polymer chemistry and enables the synthesis of structurally diverse polymers with tailored architectures and functional properties (Nomura & Jaiyen, 2026). In ROMP, the driving force for polymer formation arises from the relief of ring strain in the cyclic olefin monomer (negative ΔH), although a reduction in entropy accompanies this process (Lessard *et al.*, 2024). It has been found that living ROMP is an effective method to produce a large number of different polymers and block copolymers having a particular molecular weight range and lower polydispersity, as well as unique properties. Structures of different catalysts commonly used in the ROMP process are shown in Figure 3. Five-membered cyclic olefins, e.g., norbornene and its derivatives, are widely employed as monomers because of their high reactivity in ROMP and the ease with which functional groups can be introduced onto the ring framework (Alentiev & Bermeshev, 2022). Polynorbornenes generated through metal-catalyzed ROMP of functionalized norbornene derivatives have shown broad utility in areas such as catalysis (García-Loma & Albéniz, 2019), light-harvesting systems (Boyd & Schrock, 1999), photoinduced electron transfer, ion transport (Liu *et al.*, 2026), and optoelectronic materials (Sutthasupa *et al.*, 2025). Polynorbornene is also used for the target gas separation (Wang *et al.*, 2021). Luh and co-workers (2008) demonstrated that when norbornene derivatives bearing a 5,6-endofused *N*-arylpyrrolidine unit undergo ROMP in the presence of the first-generation Grubbs catalyst, the resulting polymers exhibit a high degree of stereochemical order. Specifically, the process yields isotactic, single-stranded polynorbornenes characterized by uniformly trans-configured double bonds and a consistent alignment of pendant groups along one direction, reflecting a remarkable level of structural control during polymer growth. It is plausible that π - π stacking interactions among the pendant aryl substituents occur during the polymerization process, thereby influencing and directing the observed stereoselective outcome. The same group also revealed that in a contrasting scenario, substituting the *N*-aryl group with an *N*-cyclohexyl moiety leads to a loss of stereocontrol, producing polynorbornenes with a mixed cis/trans double-bond distribution (21:79) and only weakly defined tacticity (Wang *et al.*, 2009).

Interestingly, this strategy has also been effectively applied to construct relatively rigid, polynorbornene-derived double-stranded ladderphane architectures (Luh, 2013) and has further enabled the templated replication of a single-stranded polynorbornene into its complementary counterpart. In polynorbornenes, the distance between adjacent repeating units is roughly 5.5 Å, a spacing that allows significant interactions to arise between neighboring pendant groups or between linkers in ladderphane structures.

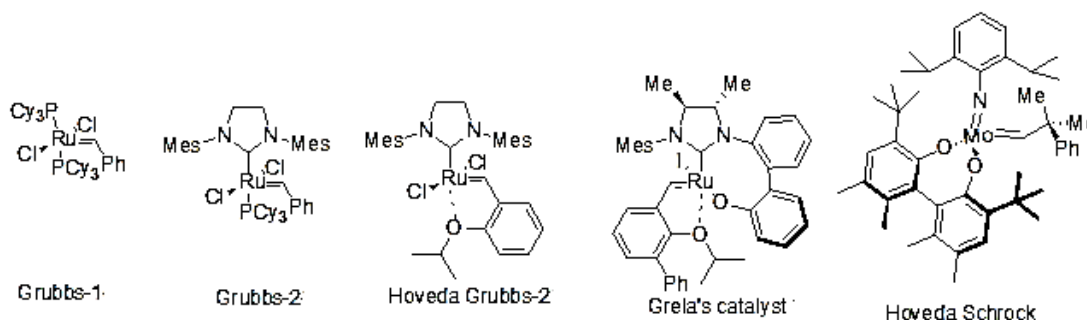


Figure 3: Commonly Used Catalysts in Metathesis Reactions

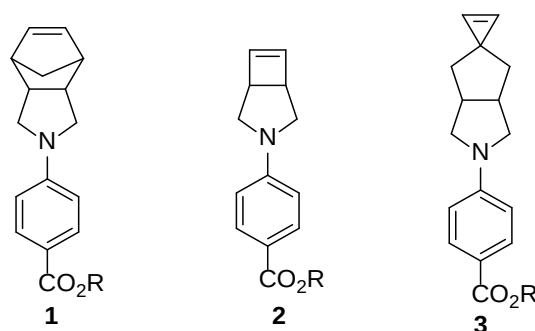


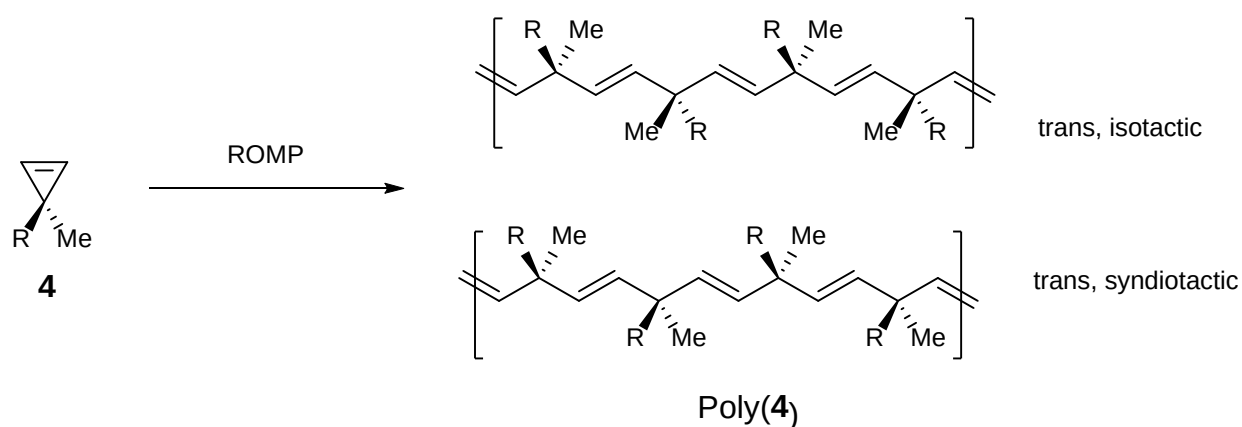
Figure 4: Different Cyclo-Ene Monomers

Comparatively, ROMPs of cyclobutene derivatives have gained less interest, and the reactions generally are nonselective, forming polymers formed as a mixture of both Z- and E-double bonds, unless specifically designed substrates are used (Lin *et al.*, 2013). It is expected that the separation of each of the monomeric units in polycyclobutenes would be somewhat shorter than polynorbornenes due to the absence of bridging methylene groups. Moving from cyclobutene to cyclopropene will bring joy and challenges in the ROMP process. Cyclopropene possesses a substantial ring strain of approximately 228 kJ mol⁻¹. Owing to its distinctive status as the smallest unsaturated cyclic framework, cyclopropene derivatives have long captured the interest of both theoretical and experimental chemists. In an early investigation, Allen and co-workers demonstrated that the vinylic carbon atoms in cyclopropene employ sp^{1.19} hybrid orbitals for bonding with substituents, whereas sp^{2.68} hybrid orbitals are utilized for bonding within the ring σ framework (Allen, 1982). The increased s-character (ca. 43%) of the cyclopropene C–H bonds enable cyclopropene to occasionally exhibit reactivity similar to that of a terminal alkyne, thereby serving as a versatile three-carbon building block.

Notably, no reports describing the ROMP of cyclopropene appeared in the literature until Singh and co-workers disclosed the successful ROMP of 3,3-disubstituted cyclopropenes in 2006. Two years later, the same group reported that the highly tactic trans-polycyclopropene was prepared through enantiomorphic site control using initiators closely related to those previously employed in asymmetric metathesis reactions (Singh & Schrock, 2008). The reason behind its living nature likely arises because the alkylidene unit in the propagating species resembles a neopentylidene or neophylidene moiety, both of which are recognized as

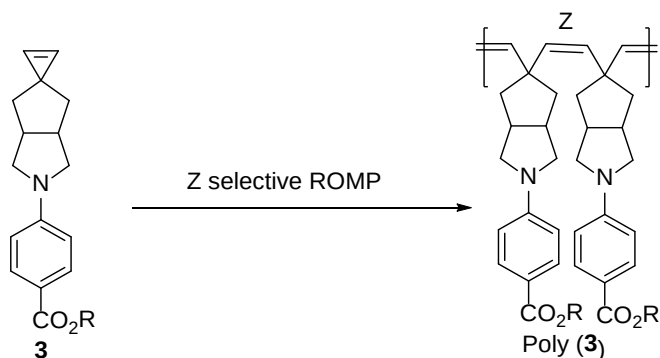
Synthesis of Polymeric Ladderphanes from Cyclopropenes

the most stable terminal alkylidenes in the $\text{Mo}(\text{NR})(\text{CHR}')(\text{OR}'')_2$ family with respect to bimolecular decomposition. The small ring metathesis is really an interesting and growing topic in organic chemistry (Panda, 2018). It was proposed that trans linkages between adjacent repeat units in poly(**4**) arise from steric congestion within the molybdabicyclopentane intermediate (Scheme 1). When the substituents at C(3) of the cyclopropene are different, as in compound **4**, the resulting polymer can display tacticity. Shown below are the two regular structural arrangements possible for trans-poly(**4**). The spacing between adjacent pendants in polycyclopropenes would therefore be shorter compared to polynorbornenes and even shorter than polycyclobutenes. Accordingly, the interactions among pendant groups in polycyclopropenes are expected to differ from those observed in polynorbornenes or polycyclobutenes. Therefore, the introduction of a congener of norbornene and cyclobutene, cyclopropene fused with N-arylpyrrolidine, will open a new arena in template-assisted duplex polymer synthesis. The close proximity of aryl pendants may show the strong π - π interactions, which will contribute to the well alignment for the formation of ladderphanes. In 2019, Peng and co-workers reported a stereospecific synthesis of polymers from cyclopropene monomers by ROMP method.



Scheme 1: ROMP of Cyclopropane Monomer

As with polynorbornenes, in polycyclopropenes, face-to-face long-range π -stacking that promotes a well-organized layered arrangement of aromatic rings may give rise to strong electronic coupling between these π -conjugated systems. (Scheme 2). Charge-carrier mobility would be strongly influenced by these intermolecular interactions, as efficient orbital overlap and enhanced electronic communication between adjacent π -conjugated units can facilitate the transportation of charge through the material. It has been anticipated that the self-organization of multichromophoric building blocks designed for light-harvesting and photophysical studies will lead to intriguing outcomes. In this context, supramolecular frameworks such as double-stranded ladderphanes could provide an alternative strategy for arranging these planar π -systems in a face-to-face manner. The polymeric backbone can also function as a barrier, effectively preventing interactions between neighboring arene arrays. Beyond covalently linked ladder-type polymers, double-stranded oligomeric or polymeric architectures can be constructed by associating two single strands through various noncovalent interactions, including metal coordination, hydrogen bonding, π - π stacking, ionic interactions, or host-guest inclusion (Elling & Neary, 2026). From another perspective, replication may be viewed as a templated process in which a single-stranded polymer directs the formation of a complementary strand, ultimately yielding an unsymmetrical double-stranded ladderphane. If the polymerization degree and polydispersity of the template are precisely regulated, this strategy could enable the synthesis of daughter polymers exhibiting controlled chain lengths and narrow polydispersity.

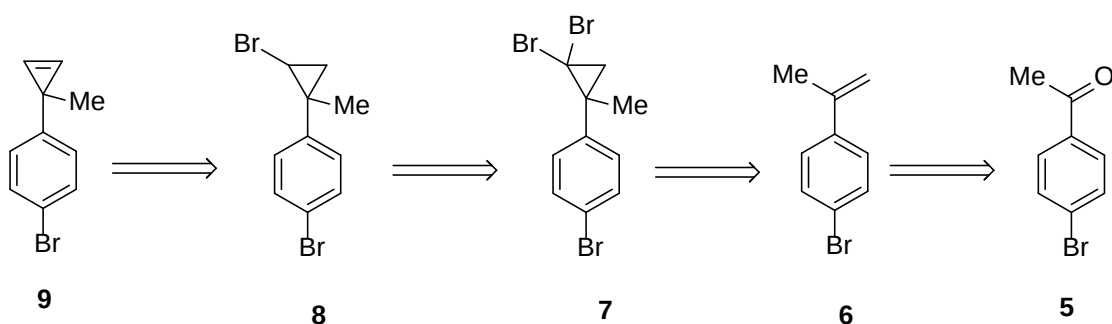


Scheme 2: Z-Selective ROMP of Pyrrolidine Containing Cyclopropene 3

Results and Discussion

For the preliminary study towards the synthesis of polymeric ladderphanes from cyclopropene monomer, 3-*p*-bromophenyl, 3-methyl cyclopropene (**9**) was selected as a model compound for the investigation of ROMP of cyclopropene moiety using different ruthenium and molybdenum-based catalytic systems.

Similar to its application in total synthesis, retrosynthetic analysis serves as a highly effective strategy for determining the synthetic route of any target molecule (Panda, 2019). Thus, with a view to synthesizing 3-*p*-bromophenyl, 3-methyl cyclopropane **5**, the retro-synthetic analysis is presented in Scheme 3. It has been envisaged that the conversion of 4-bromo acetophenone **5** into α -methyl styrene **6** can easily be achieved in a single step using Wittig olefination. An alternate approach is also possible from 4-bromo acetophenones **5** through a two-step process with the addition of methyl magnesium bromide, after that the acid-catalyzed dehydration of the generated tertiary alcohols, but the preparation of olefins from this method is highly prone to acid-catalyzed polymerization. It will then involve a three-step sequence, namely, the conversion of α -methyl styrene **6** to the dibromocyclopropane **7**, partial reduction of dibromocyclopropanes will provide monobromocyclopropane **8**, which is then dehydrohalogenated to furnish the required cyclopropene derivative **9**.



Scheme 3: Retrosynthesis of Cyclopropene Derivative 9

The forward synthesis was commenced using commercially available 4-bromoacetophenone **5** (Scheme 4). The bromo substituent was incorporated into the aromatic ring because it can be readily functionalized through various cross-coupling reactions, enabling the introduction of diverse functional groups (Panda & Sarkar, 2010). Wittig olefination provides styrene derivative **6** in 92% yield. Dibromocyclopropanes **7** were synthesized through the cyclopropanation process of crude olefins **6** using dibromocarbene, produced under modified Makosza's phase transfer catalysis (PTC) conditions. Notably, employing a different addition method than the one originally described by Makosza, where an aqueous base solution was added dropwise into a rapidly stirred mixture containing the organic components, proved to be advantageous for enhancing the reaction yields. This revised method significantly suppressed resin formation, thereby enabling cleaner and more efficient isolation of the target compound.

Synthesis of Polymeric Ladderphanes from Cyclopropenes

To manage the exothermic nature of the reaction, especially during the initial stages, the reaction was performed in a 1:1 (v/v) mixture of bromoform and dichloromethane. Refluxing this mixture was crucial for preventing overheating of the reaction mixtures and ensuring temperatures did not exceed 40–45°C. During the later stages of the process, once the intense exothermic reactions subsided, the residual dichloromethane was removed at 50°C. It is well established that the kinetics of cyclopropanation reactions under phase-transfer conditions depend significantly on the structure of the phase-transfer catalyst. Typically, the highest reaction rates and optimal conversions are achieved when employing benzyltriethylammonium (TEBA) salts. However, it was observed that the use of tetrabutylammonium (TBA) and TEBA salts introduced additional challenges, including pronounced foaming and the formation of persistent emulsions during the aqueous workup, particularly in large-scale reactions.

In contrast, when hexadecyl- or tetradecyltrimethylammonium salts were used in catalytic amounts, the biphasic solution was separated more efficiently. This led to quicker and more effective extraction, ultimately resulting in higher overall yields. The final product was isolated by straightforward filtration through a short column of silica gel. Cyclopropanation of α -methyl styrene **6** with dibromocarbene produces dibromocyclopropane **7** in 82% isolated yield. The reduction of dibromocyclopropanes **7** was followed by the established procedure with some practical adjustments necessary for scale-up (Sheshenev *et al.*, 2009). To ensure safety and efficiency, several modifications were made.

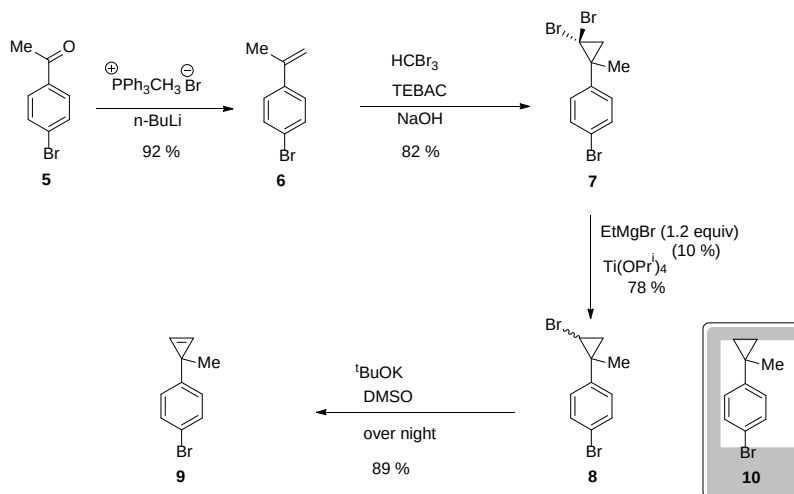
Firstly, due to the evolution of flammable gases such as ethylene and ethane, the reaction was conducted in a fume hood where ventilation is good. The reaction vessel was filled to less than one-third of its capacity and was fitted with a proficient reflux condenser. The extra space acted as a safeguard against irregular boiling and unexpected splashing, crucial especially during the quench step when a significant amount of heat and gas was generated. Secondly, the reaction exhibited an initiation phase where the initial 20 mol % of the Grignard reagent was utilized for the reduction of the Ti(IV) complex into the Ti(II) species. The generation of the catalytically active complex was indicated by a change in colour from pale yellow to dark brown. However, this colour change did not always signify the end of the activation period. The process might take more time when the addition of the Grignard reagent was too rapid, exceeding the rate of Ti(IV)/Ti(II) reduction. Therefore, there is a significant risk of the reaction mixture violently boiling and splashing. Therefore, during the early stages of the reaction, it is essential to add the Grignard reagent dropwise at a reasonably slow rate. This allows for the solvent to boil and the gaseous byproducts to evolve gradually. Additionally, close monitoring of the reaction through GC analysis is imperative to prevent the overreduction of the target monobromide **8** to the corresponding cyclopropane compound **10**.

It is crucial to use the Grignard reagent having exact strength; otherwise, if the Grignard reagent is not titrated before use, there is a possibility of forming the undesired byproducts in a considerable amount. To address this, approximately 1.1 equivalents of EtMgBr were added dropwise. The reaction mixture was then allowed to stir for 15 minutes and studied using GC. The requirement of any additional amount of Grignard reagent was accurately determined based on the GC conversion.

Under typical conditions, the crude reaction mixture was found to contain 98–99% bromocyclopropane **8**, with residual starting material **7** accounting for no more than 1%. Considering the subsequent step, it is preferable to retain some dibromide **7** instead of risking overreduction into compound **10**. This preference arises because dibromide **7** will ultimately be eliminated by the treatment of strong base *t*-BuOK through the β -elimination process. In contrast, the separation of compounds **9** and **10** is exceedingly challenging due to their similar polarity. Hence, if a significant amount of the over-reduced product **10** is generated, it becomes necessary to purify monobromide **8** through column chromatography.

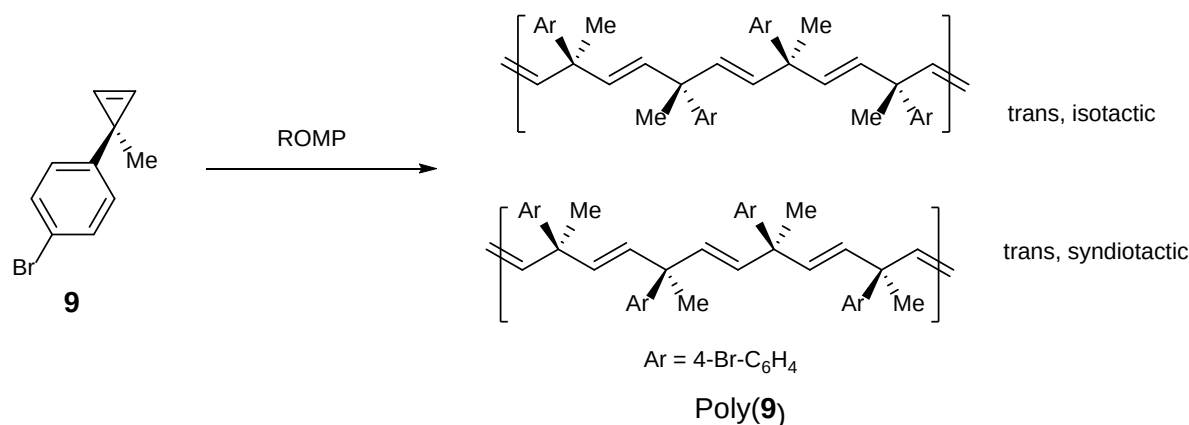
In the synthesis of cyclopropenes, diastereoselectivity does not play a significant role because both diastereomeric forms of compound **8** are capable of undergoing dehydrohalogenation under the reaction conditions. Cyclopropenes **9** were synthesized from monobromocyclopropanes **8** by carrying out the

reaction in anhydrous DMSO in the presence of a slight excess of *t*-BuOK. Under these conditions, the base-promoted dehydrohalogenation of the bromocyclopropane intermediates proceeded efficiently to afford the corresponding cyclopropenes. Because this reaction is highly sensitive to moisture and oxygen, it requires careful handling and precautions to prevent degradation or failure. For this reason, potassium *tert*-butoxide was kept and used in a nitrogen-filled glovebox.



Scheme 4: Synthesis of Cyclopropane 9

After successful synthesis of cyclopropane **9**, the ROMP reaction was investigated under different ruthenium-based catalytic conditions (Scheme 5). Both Grubb's 1st generation catalyst and Grubb's-Hoveyda 1st generation catalysts were proven to be less effective in ROMP polymerization of cyclopropane **9**. It has been found that using 10 mol % of catalysts and stirring at room temperature for up to 36 hours shows very little conversion of starting material. However, other ruthenium-based catalysts, such as Grubb's 2nd generation catalyst, proved to be efficient on ROMP reactions, as using 12 mol % of catalyst, full conversion of the starting material was observed within 24 hours of stirring at ambient temperature. The polymer's NMR spectrum displays a broadened proton signal in the 5–6 ppm region. The gel permeation chromatography (GPC) analysis shows the low polydispersity of the formed polymer (Figure 5). Gel permeation chromatography (GPC) analysis of the polymer exhibited a single, well-defined peak with a narrow polydispersity (PDI = 1.06), consistent with characteristics of a living polymerization process.



Scheme 5: ROMP of Cyclopropane 9

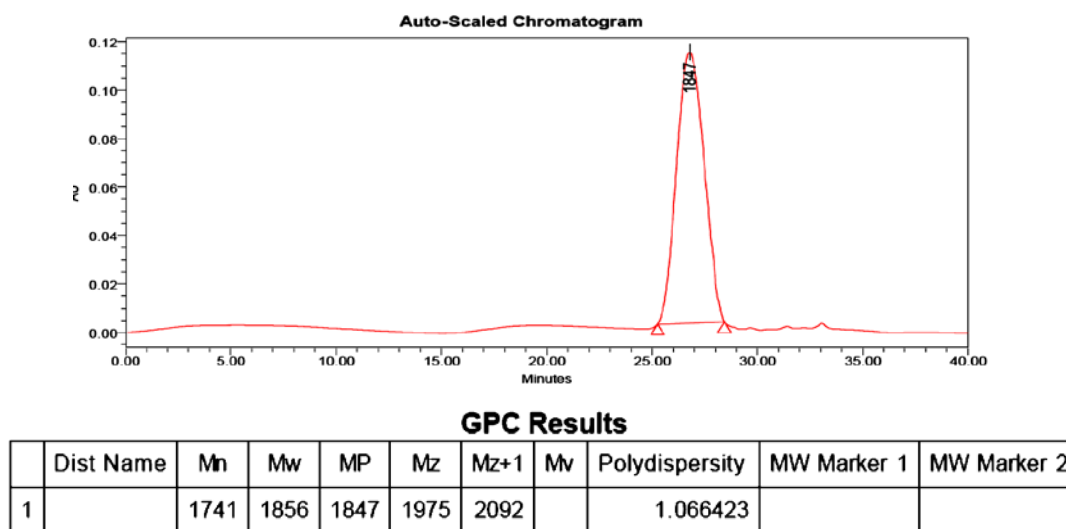


Figure 5: Gel Permeation Chromatogram

Following the successful synthesis of the cyclopropene polymer poly (**9**), the next goal was to prepare polymeric ladderphanes from monomer **3**, a project that is currently underway in the laboratory. The synthetic methods discussed above provide an understanding of how to plan a synthesis using a retrosynthetic pathway. In forward synthesis, various problems may arise that must be solved sequentially to achieve the target molecule. Thus, target-oriented and total synthesis have expanded the knowledge and built the expertise necessary to successfully execute various chemical reactions (Panda & Sarkar, 2013). Additionally, modern techniques such as cross-coupling reactions or C-H activations, as well as the development of various catalytic transformations, make this target-oriented synthesis more efficient compared to classical methods in organic synthesis.

Conclusion

The strain cyclopropene monomer was successfully synthesized, and then it was used for a ring-opening metathesis polymerization reaction. It gives the desired polymer in good yield and low polydispersity. This study will help to build the strategy for the synthesis of polymeric ladderphanes from cyclopropene monomers with low polydiversity. Since the span of each monomeric unit of the polymer derived from cyclopropene is less, it may show strong and better activity and will be helpful in different photophysical studies. This model system creates a conceptual framework for future efforts intended for synthesizing fully conjugated and structurally precise ladderphanes from cyclopropene monomers.

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Synthesis of Polymeric Ladderphanes from Cyclopropenes

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