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Chemical Reduction of 9,10-Anthraquinone: A Route to Anthracene

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Abstract: *Anthracene and anthracene derivatives have been extensively studied over the years because of their interesting photophysical, photochemical, and biological properties. They are currently the subject of research in several areas, which investigate their use in the biological field and their application in OLEDs, OFETs, polymeric materials, solar cells, and many other organic materials. Their synthesis remains challenging, but some important preparative methods have been reported, especially in the last decade. This review presents an update of the recent strategies that have been employed to prepare anthracene derivatives. It encompasses papers published over the last twelve years (2008–2020) and focuses on direct and indirect methods to construct anthracene and anthraquinone frameworks.*

The preparation of anthracene from 9,10-anthraquinone involves a catalytic reduction process, where the quinone is reduced to anthracene through the use of reducing agents. A common method is the catalytic hydrogenation or the use of metal-based reducing agents like iron (Fe) or zinc (Zn) in the presence of an acid. In this reaction, 9,10-anthraquinone undergoes reduction of its carbonyl groups to form the corresponding hydrocarbon, anthracene. The process requires careful control of temperature and reaction time to ensure complete reduction and the formation of pure anthracene. This transformation is often carried out in a solvent like ethanol or another appropriate medium to aid in the dissolution and proper reaction of the reactants. The product is then isolated, typically by recrystallization, to obtain anthracene in high purity.

The preparation of anthracene from 9,10-anthraquinone typically involves a reduction reaction. The reduction of 9,10-anthraquinone to anthracene is commonly achieved through catalytic hydrogenation or chemical reducing agents like zinc or sodium in the presence of an alcohol.

Keywords: Reduction, Anthracene, 9,10-anthraquinone, Zinc, Iron, OLEDs, OFETs, polymeric materials, solar cells

I. INTRODUCTION

Anthracene is a pale yellow colored stable carbon compound. It is formed by joining three benzene rings.Anthraquinone is derived from anthracene.The compound is obtained by oxidation. Many pigments are produced from angraquinones called angraquinone dyes. Anthracene is a carbon semiconductor in pure form. They are used to detect high energy photons, electrons and alpha particles. Since anthracene, which is colorless, glows blue under UV light, it is used as an ultraviolet (UV) detector.Coal tar contains 1.5% anthracene. It is produced in the form of by-products by heating coal used in the manufacture of iron.

Anthracene is an important aromatic hydrocarbon consisting of three linearly fused benzene rings. Because of their extended aromatic and conjugated π-system, anthracene derivatives possess interesting photochemical and photophysical properties [1-3], as well as gelling ability [4]. These important properties make them relevant for the development and application of several organic materials, such as organic light-emitting diodes (OLEDs) [5], organic field-effect transistors (OFETs) [6], polymeric materials [7], and other kinds of materials.

Despite some difficulties and limitations, a number of synthetic methods for preparing anthracene derivatives has been reported over the years. The most familiar methods to obtain substituted anthracenes include Friedel–Crafts reactions [17], Elbs reaction [18], aromatic cyclodehydration [19,20], Bradsher-type reactions from diarylmethanes [21-23], and,

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more recently, metal-catalyzed reactions with alkynes [24,25]. Numerous synthetic routes have also been reported for the synthesis of anthraquinones [26-29]. On the other hand, preparative methods for dibenzoanthracene derivatives are less common, mainly relying on the cyclization of the corresponding lactone derivative followed by sequential modifications [30], photocyclization of divinylterphenyl derivatives [31], tandem radical cyclization of (Z,Z)-1,4-bis(2-iodostyryl)benzene derivatives [32], and ring-closing olefin metathesis of tetravinylterphenyls [33] as the best-known synthetic routes.

Anthracene is a widely studied polycyclic aromatic hydrocarbon composed of three fused benzene rings. It has significant applications in various industries, including the production of dyes, plastics, and organic semiconductors. One of the key methods for synthesizing anthracene involves the reduction of 9,10-anthraquinone, a derivative of anthracene with carbonyl groups attached to the 9 and 10 positions of the aromatic ring system.

Herein, we have classified the synthetic methods into three general categories: synthesis of substituted anthracene frameworks, synthesis of benzanthracene and dibenzanthracene derivatives, and synthesis of anthraquinone derivatives. We will focus on the construction of the anthracene and anthraquinone frameworks published in the last twelve years (2008–2020); methods for simple modifications of the anthracene and anthraquinone rings will be excluded. To the best of our knowledge, this is the first review involving the synthesis of anthracene derivatives spanning this period.
Anthracene is a polycyclic aromatic hydrocarbon consisting of three fused benzene rings, and it is widely used in the synthesis of dyes, pharmaceuticals, and in the production of organic semiconductors. 9,10-Anthraquinone, a carbonyl-containing aromatic compound, serves as an important precursor in various organic transformations, including the synthesis of anthracene.

The conversion of 9,10-anthraquinone to anthracene is a crucial step in organic synthesis, primarily achieved through reduction reactions. The reduction of 9,10-anthraquinone involves the transformation of its carbonyl groups into alkyl groups, resulting in the formation of anthracene. This reaction typically requires the use of reducing agents, which are capable of donating electrons to the quinone structure, thereby facilitating the reduction process.

Various methods can be employed to reduce 9,10-anthraquinone to anthracene, including catalytic hydrogenation, metal-based reductions (such as using zinc or iron), or chemical reducing agents like sodium dithionite. These methods are chosen based on factors such as the efficiency of the reduction, the ease of product isolation, and the minimization of side reactions.

This reaction not only provides a synthetic route to anthracene but also highlights the importance of controlling reaction conditions to achieve high yields and purity. The preparation of anthracene from 9,10-anthraquinone has been studied extensively, and the reaction mechanisms, along with various optimization strategies, are of great interest in organic chemistry.

The reduction of 9,10-anthraquinone to anthracene is an important reaction in organic chemistry, primarily aimed at eliminating the carbonyl groups from the quinone structure. This transformation typically involves the use of reducing agents such as zinc, iron, or hydrogen, which donate electrons to the carbonyl groups, converting them into methylene (-CH2) groups, thus yielding anthracene.

This process is significant not only for its ability to produce anthracene but also for its role in demonstrating important concepts in organic reaction mechanisms, such as reduction and electron transfer. In this context, the reaction conditions—such as solvent choice, temperature, and reaction time—are crucial for optimizing yield and ensuring the

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purity of the final product. Overall, the reduction of 9,10-anthraquinone to anthracene serves as a practical example of synthetic organic chemistry, with applications in both industrial settings and academic research.

II. METHODOLOGY

The methodology for the reduction of 9,10-anthraquinone to anthracene typically involves selecting an appropriate reducing agent, optimizing reaction conditions (temperature, solvent, and concentration), and ensuring proper isolation and purification of the final product. Below is a general outline of the typical experimental procedure for the reduction of 9,10-anthraquinone to anthracene using metal-driven reductions, catalytic hydrogenation, or sodium dithionite.

1. Catalytic Hydrogenation Method

Materials:

9,10-Anthraquinone (reagent-grade), Hydrogen gas (H□), Catalyst: Palladium (Pd) on carbon (10% Pd), Solvent: Ethanol or toluene, High-pressure reaction vessel (hydrogenation apparatus)

Procedure:

Preparation of Reaction Mixture:

Weigh a specific amount of 9,10-anthraquinone (typically 1.0–2.0 g) and dissolve it in a suitable solvent (e.g., ethanol or toluene) in the reaction vessel. Add an appropriate amount of palladium on carbon (10% Pd) catalyst to the reaction mixture (usually 0.1–0.2 g of catalyst for every gram of 9,10-anthraquinone). The reaction vessel is sealed, and hydrogen gas (H□) is introduced into the system at a constant pressure, typically between 1–5 atm. The reaction is conducted at a moderate temperature (50–150°C) for several hours (usually 4–8 hours), depending on the progress of the reaction. During the reaction, hydrogen molecules adsorb onto the surface of the palladium catalyst and react with the carbonyl groups of 9,10-anthraquinone to reduce them to methylene (-CH□) groups. The progress of the reduction can be monitored by thin-layer chromatography (TLC) or by checking for changes in the appearance of the reaction mixture (from yellow/brown to colorless or pale yellow). Once the reaction is complete, the catalyst is filtered off from the mixture, and the solvent is evaporated under reduced pressure using a rotary evaporator. The crude product is purified by recrystallization from a solvent such as ethanol or a mixture of hexane and dichloromethane.

2. Reduction with Zinc (Zn) or Iron (Fe)

Materials:

9,10-Anthraquinone, Zinc powder or iron filings, Hydrochloric acid (HCl) (10–15% concentration), Solvent: Ethanol or acetic acid

Procedure:

Preparation of Reaction Mixture:

Dissolve 9,10-anthraquinone (1.0–2.0 g) in an appropriate solvent such as ethanol or acetic acid (15–25 mL). Add zinc powder (2.0–3.0 g) or iron filings (2.0–3.0 g) to the mixture, ensuring an excess of metal is present to drive the reduction. Add a few drops of concentrated hydrochloric acid (HCl) to the reaction mixture to promote the reduction process. Heat the mixture under reflux for 4–6 hours, allowing the zinc or iron to donate electrons to the carbonyl groups of 9,10-anthraquinone. The reduction process typically produces anthracene and a by-product such as zinc chloride (ZnCl□) or iron(III) chloride (FeCl□). The progress of the reaction can be checked periodically using TLC or by observing the appearance of the reaction mixture. After the reaction is complete, cool the reaction mixture to room temperature and filter off the zinc or iron residues. The solvent is evaporated under reduced pressure to yield the crude product. The crude product is purified by recrystallization, typically from ethanol or hexane, to isolate pure anthracene.

3. Reduction Using Sodium Dithionite (Na□S□O□

Materials:

9,10-Anthraquinone, Sodium dithionite (Na□S□O□), Water or ethanol

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Preparation of Reaction Mixture:

Dissolve 9,10-anthraquinone (1.0–2.0 g) in a solvent such as ethanol or water (20–30 mL). Add sodium dithionite (2.0–3.0 g) to the reaction mixture under an inert atmosphere, ensuring complete dissolution of the dithionite. Stir the reaction mixture at room temperature for 1–3 hours. The sodium dithionite will donate electrons to the 9,10-anthraquinone, reducing the carbonyl groups and converting the compound into anthracene. The reduction progress can be followed by TLC or observing the color change in the reaction mixture. Once the reaction is complete, the solvent is evaporated to concentrate the product. The crude product is purified by recrystallization from ethanol or a mixture of ethanol and water. Purity and identity of the anthracene product can be confirmed by methods such as NMR spectroscopy, UV-Vis spectroscopy, and mass spectrometry.

III. LITERATURE REVIEW

The conversion of 9,10-anthraquinone to anthracene has been an area of extensive research due to its significance in both academic and industrial chemistry. Several methods have been developed to achieve the reduction of 9,10-anthraquinone to anthracene, each with different reducing agents, reaction conditions, and mechanistic pathways.

OLEDs fabricated with 9,10-diphenylanthracene derivatives 1 and 2 are blue light emitters [11,12], the 2,2'-bianthracene derivative 3 provides a green and fluorescent OLED [13], 2,2'- bianthracenyl (4) has been employed as an organic semiconductor in an OFET device [14], and di- n-alkoxyanthracenes have gelling properties with diverse solvents, mainly alkanes and alcohols [4]. Furthermore, anthracene derivatives display useful biological activities; for instance, the anthraquinone derivatives 5 and 6 exert antimicrobial and anti-inflammatory activity, respectively (Figure 1) [15,16].

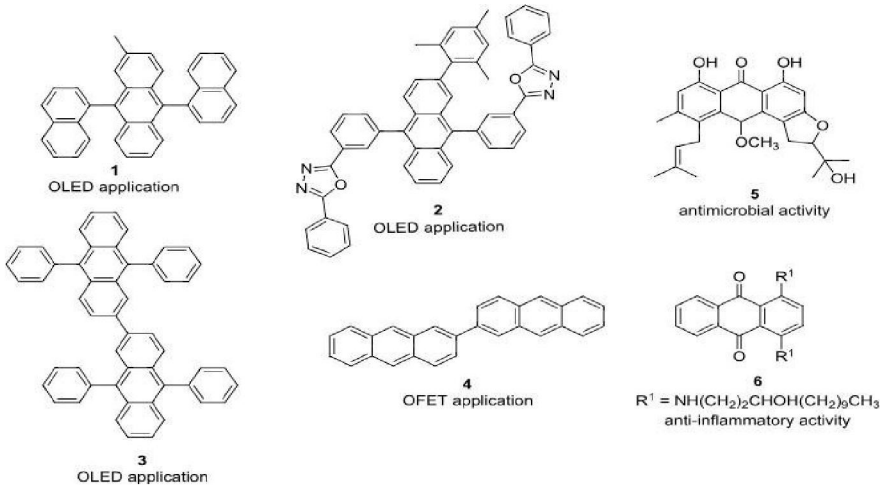

Fig No. 1 Examples of anthracene derivatives and their applications

Synthesis of substituted anthracene frameworks Metal-catalyzed reactions with alkynes. Metal-catalyzed reactions with alkynes have gained attention in the last years and have provided new methodologies to prepare anthracene derivatives. In 2009, Miura and co-workers were the first to obtain substituted anthracenes selectively by homologations with monofunctionalized naphthyl substrates. These authors demonstrated that the rhodium-catalyzed oxidative 1:2 coupling reactions of arylboronic acids 7 with alkyne 8 occurred in the presence of a copper–air oxidant, to give the corresponding 1,2,3,4-tetrasubtituted anthracene derivatives 9a and 9b (Scheme 1) [34]. Although the scope of the reaction was broader for 1,2,3,4-substituted naphthalenes, the authors developed a potentially applicable methodology to synthesize substituted anthracenes and other polysubstituted fused aromatic compounds

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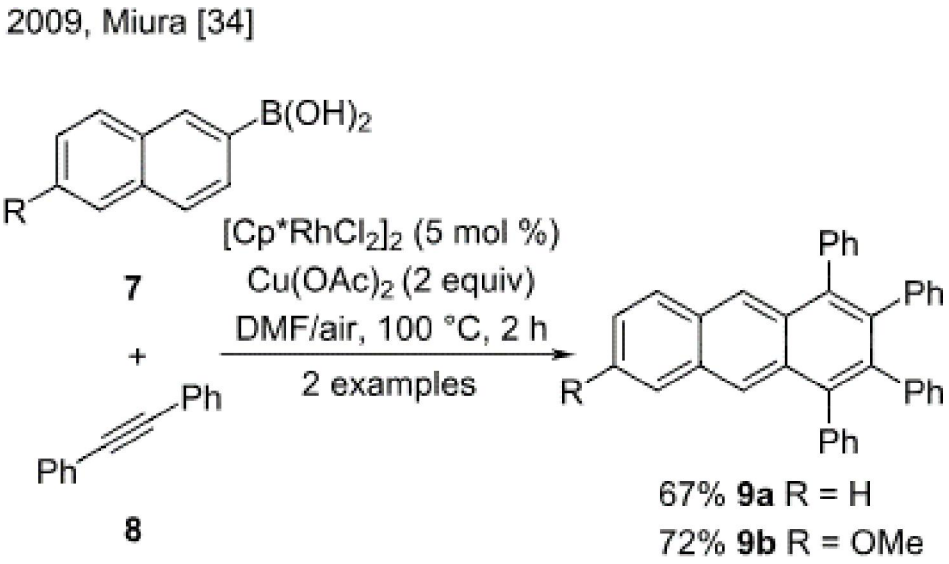

Fig No. 2 Rhodium-catalyzed oxidative coupling reactions of arylboronic acids with internal alkynes

Synthesis of substituted anthracenes from anthraquinones

An easy and common method to obtain anthracenes is to reduce anthraquinones by using several reagents. An important advantage of this method is that the reactive positions 9 and 10 of anthracene are protected, directing substitution to one of the other rings. By using this kind of methodology, in 2009, Han, Nedeltchev, and Bhowmik reported the facile synthesis of 9,10-diacetoxyanthracenes 56 and 2,6-dialkoxyanthracenes 58 from the corresponding anthraquinones 55 and 57 via a single- step reduction with either zinc/pyridine or zinc/NaOH (Scheme 12). The scope of their work consisted of six examples, and they obtained anthracene derivatives in moderate to good yields (50– 87%)

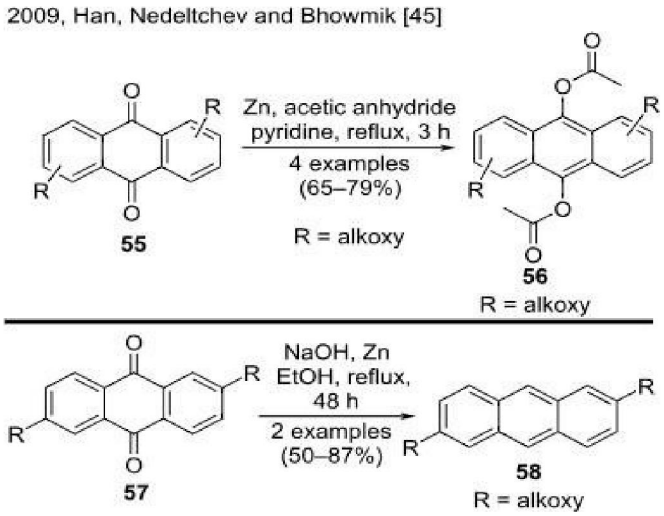

Fig No. 3 Reduction of anthraquinones by using Zn/pyridine or Zn/NaOH reductive methods. [45].

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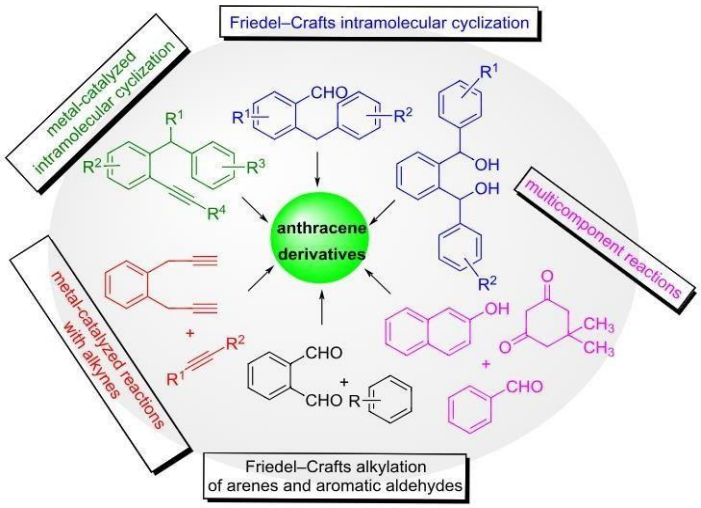

update of the recent strategies employed to prepare anthracene derivatives

Fig No. 4 Reduction of anthracene derivatives .

IV. FUTURE SCOPE

The conversion of 9,10-anthraquinone to anthracene remains an important topic in organic chemistry, with ongoing research focused on improving the efficiency, sustainability, and applicability of the reduction process. Several areas of future research and development could further enhance this reaction

The future scope of reducing 9,10-anthraquinone to anthracene holds promising opportunities for advancements in sustainable chemistry, improved catalyst design, and broader applications in materials science and electronics. As research continues to focus on environmental impact reduction, efficiency, and scalability, the methods for anthracene production could be revolutionized, leading to more cost-effective, green, and industrially applicable processes.

As environmental concerns continue to rise, there is a strong push towards finding greener, more sustainable methods for synthesizing anthracene. The use of non-toxic and renewable reducing agents, such as bio-based reagents or recyclable catalysts, can significantly reduce the environmental impact of the reduction process. Exploring solvent-free reactions or using water as a solvent could also lower the ecological footprint of the reaction. The development of catalytic systems that require fewer precious metals (e.g., palladium and platinum) is another avenue for improving the sustainability of this reaction.

The reduction of 9,10-anthraquinone to anthracene is not only a key reaction in organic synthesis but also opens up opportunities in fields like materials science, electronics, renewable energy, and medicine. The future scope involves expanding the use of anthracene-based materials in advanced technologies, enhancing its role in green chemistry, and continuing to explore its biological activities. The increasing demand for more sustainable and efficient materials means the future potential for anthracene and its derivatives will continue to grow.

V. CONCLUSION

In this review, we have highlighted the most recent preparative methods for anthracene derivatives. Among the many synthetic strategies reported in the last twelve years, metal- catalyzed/promoted reactions, especially those with internal alkynes, have been the most used, possibly because they provide a simple and straightforward ring extension method to construct polycyclic aromatic compounds, especially anthracenes with substituents in the 2-, 3-, 6-, and 7-positions.

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Also, considering the difficulties and limitations of direct syntheses of anthracene derivatives, the considerable number of methodologies reported in recent years is truly surprising. Due to the wide applicability of anthracene and anthraquinone derivatives in important fields of science, the development of new synthetic methods is likely to increase. We hope that this review can serve to guide and to inspire future advances in synthetic organic chemistry for this kind of polycyclic compounds.

The reduction of *9,10-anthraquinone to anthracene* represents an important synthetic route with significant implications across various industries. Anthracene, a valuable organic compound, has broad applications in *organic electronics, **polymer chemistry, **pharmaceuticals, and **renewable energy technologies. Its potential in **OLEDs, **organic solar cells, and **drug development* highlights its versatility. As the demand for sustainable materials and advanced technologies increases, the continued exploration of anthracene and its derivatives in *materials science, **nanotechnology, and **catalysis* will likely unlock new innovations and applications. Therefore, the future scope of anthracene extends beyond simple reduction reactions, paving the way for advancements in multiple fields

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