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A Review on the Synthesis of Polycyclic Aromatic Hydrocarbons through Substituted Phenanthrenes

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Abstract : The synthesis of substituted phenanthrenes has witnessed significant progress, transitioning from conventional cyclization and Friedel–Crafts strategies to advanced catalytic methodologies. Recent innovations such as transition-metal-catalyzed coupling reactions, C–H activation, and photocatalytic approaches have greatly improved efficiency, selectivity, and environmental sustainability in phenanthrene construction. The evolution of synthetic methodologies has reinforced the importance of phenanthrenes as a versatile class of compounds, while also paving the way for future innovations in their derivatization and application. This growing scope highlights the relevance of sustainable, selective, and efficient synthetic strategies in advancing both fundamental research and practical applications of substituted phenanthrenes.

***IndexTerms* – Poly Aromatic Hydrocarbon, Phenanthrenes, C–H activation, Alkylation, Oxidative Cyclization.**

I. INTRODUCTION

Polycyclic aromatic hydrocarbons (PHCs) have received attention, especially in material science, due to their utility in photovoltaic devices such as field-effect transistor, light emitting diodes and solar cells. Most of polycyclic aromatic hydrocarbon composed of phenanthrene molecule (Fig. 1).

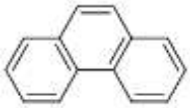


Fig.1

Substituted phenanthrenes represent an important class of polycyclic aromatic compounds with significant relevance in medicinal chemistry, material science, and natural product synthesis. Numerous synthetic strategies have been developed for their preparation, employing both classical and modern organic transformations. Traditional methods often rely on oxidative cyclization, Friedel–Crafts alkylation/acylation, or transition-metal-catalyzed coupling reactions to construct the phenanthrene core.

In recent years, metal-catalyzed C–H activation, photocatalysis, and cascade cyclization techniques have gained prominence due to their improved efficiency, regioselectivity, and environmental compatibility.

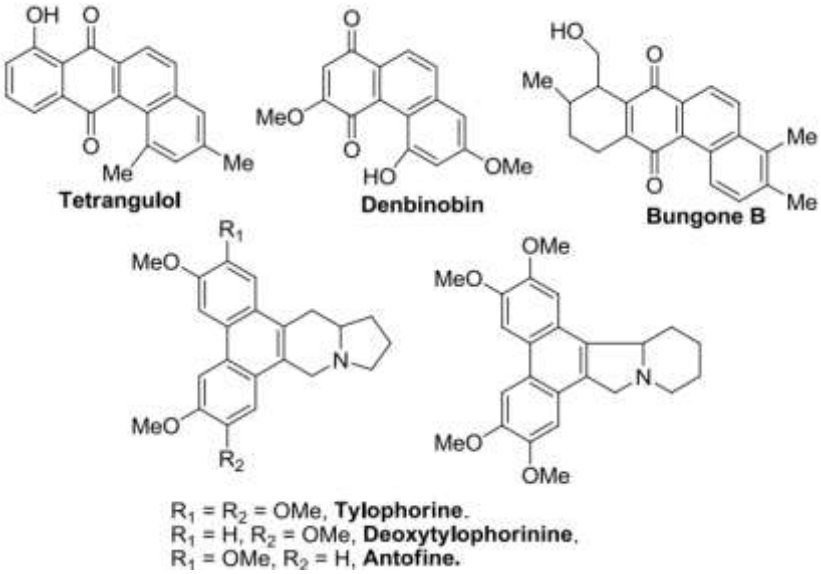


Fig.2

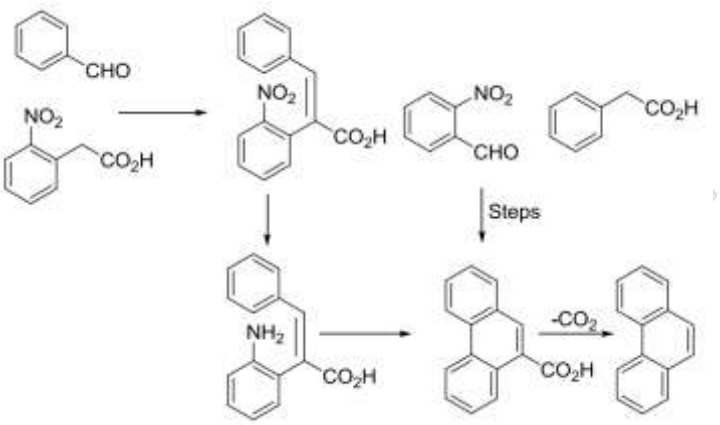
Additionally, biomimetic approaches have been explored to replicate phenanthrene frameworks found in natural products. The selection of a suitable method typically depends on the desired substitution pattern, functional group tolerance, and reaction scalability.

The construction of phenanthrene skeleton has much demanded due to their occurrence in numerous natural products.[1] Moreover, compounds having this structural motif exhibit interesting biological activities such as antimalarial,[2] anticancer,[3] and emetic activity.[4] They are also very useful in material science due to their photoconductivity, photochemical and electroluminescent properties.[5]

In addition, there are many phenanthrene based alkaloids such as phenanthroindolizidine, phenanthroquinolizidine etc. for which the synthesis of phenanthrene is the key step in the preparation of these alkaloid molecules.[6] There are many natural products such as Tetrangulol, Denbinobin and Bungone B etc contain an oxidized phenanthrene subunit (Fig. 2).

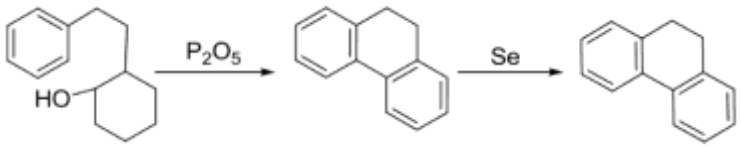
II. RESULT AND DISCUSSION:

In 1896, Pschorr[7] has developed a classical method on phenanthrene synthesis starting from 2-nitrophenyl acetic acid derivatives and benzaldehyde derivatives. The reaction was followed the steps sequentially, the reduction of nitro to the amine, diazotization and ring closure, and decarboxylation of the phenanthrene-9-carboxylic acid formed (Scheme 1). Although these reaction sequences were still widely used, there were certain limitations. Since then the *o*nitrobenzaldehydes and substituted phenylacetic acids were often difficult to obtain so that theα-aryl-*trans*-cinnamic acids were inaccessible. Overall yields in the five-stage synthesis were also low.



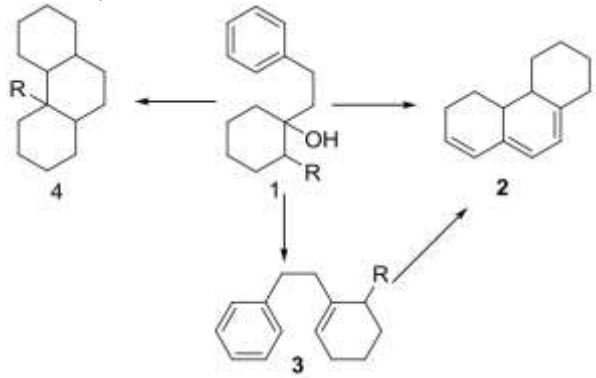
Scheme 1

In 1932, a classical phenanthrene synthesis was developed by the Bardhan-Sengupta groups[8]. In this reaction the first step was an electrophilic aromatic substitution reaction, which was allowed the diphosphorous pentoxide which made the alcohol a better leaving group. However, no alkenes outside of the initial aromatic ring were created. In the second step of this reaction 9,10-dihydrophenanthrene was oxidized with elemental selenium. The aromatization of sixmembered rings by selenium was not clearly understood, but it produced H_2Se (Scheme 2).



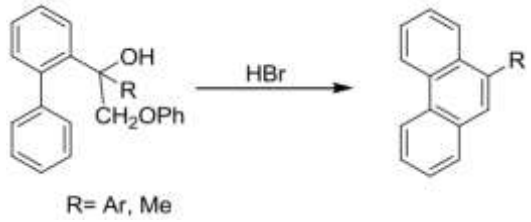
Scheme 2

In 1933, the Bogart-Cook *et al*.[9] have reported a synthesis of octahydrophenanthrene **2** from phenethylcyclohexan-l-ol **1** (R = H) which can be cyclodehydrated to the **2** (R = H) by concentrated sulfuric acid. Alternatively, they also showed that the milder dehydrating agents (e.g., iodine, potassium hydrogen sulfate, 50% sulfuric acid) also converted **1** (R = H) into the olefin **3** (R = H), which further could be ring-closed to **2** (R = H) with stannic chloride. The olefin **3** (R = H) had also been photocyclized to **2** (R = H) in good yield. Now the required alcohol **1** (R = H) is usually prepared by the interaction of the Grignard reagent derived from a 6-phenethyl bromide and a cyclohexanone. If a substituted cyclohexanone is used, the alcohol, e.g., **1** (R = alkyl) may be cyclized to either **2** (R = alkyl) or **4** (R = alkyl) (Scheme 3).



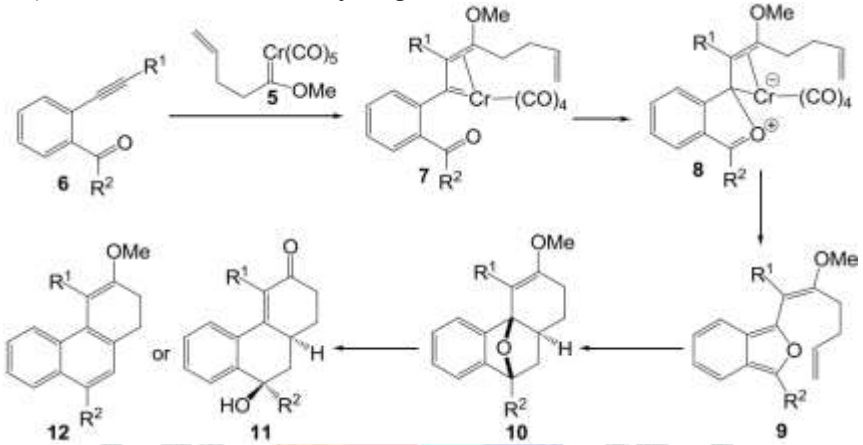
Scheme 3

In 1939, Charles K. Bradsher *et al.*[10] have demonstrated that 1-aryl-1-(2-biphenyl)-2- phenoxyethanols (R = aryl) readily undergo cyclization to give 9-arylphenanthrenes in presence of HBr. They also examined that the crude carbinol (R = CH_3), synthesized from 2- biphenylmagnesium iodide and phenoxyacetone, underwent cyclization by refluxing in a mixture of hydrobromic and acetic acids to obtained 9-methylphenanthrene (Scheme 4).



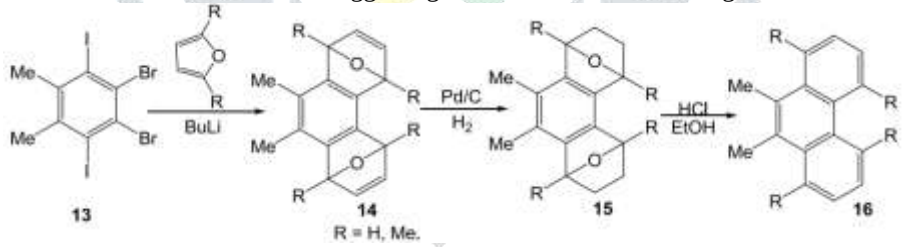
Scheme 4

James W. Herndon *et al.*[11] have synthesized a phenanthrene derivatives from 2-alkynylbenzoyl derivatives with reaction of prenylated carbene complexes. In this reaction, an isobenzofuran was formed, which underwent an *exo* selective intramolecular Diels–Alder reaction followed by ring opening to afford hydrophenanthrenone derivatives in an overall net [5+5]-cycloaddition reaction. They have also observed that reaction showed a remarkable degree of versatility with respect to the R^1 and R^2 substituents, however, the yield of the hydrophenanthrene products was considerably lower when the dienophile alkene was hindered. But product yields became considerably higher using bis(prenylated) species owing to an increase in the effective molarity of dienophilic entities (Scheme 5). Under these conditions alkyl migration reactions occur to form the observed products.



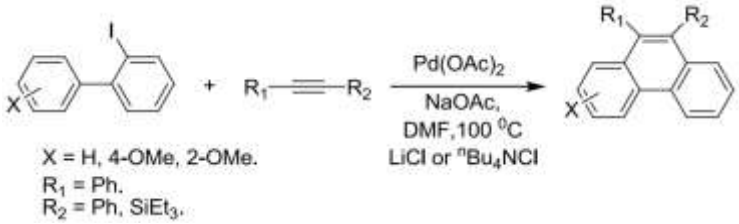
Scheme 5

Harold Hart *et al.*[12] have illustrated that the ortho bis(aryne) equivalent (tetra haloarenes) can be converted to phenanthrenes in two (or three) steps which can be used to synthesize hindered phenanthrenes such as deca-methyl phenanthrene. Here ortho bis(aryne) equivalent 4,5-dibromo-3,6-diiodo-*o*-xylene reacts with *n*-butyllithium in the presence of furans or N-substituted pyrroles to give di-adducts followed by removal of the oxygen or nitrogen bridges gives phenanthrenes (Scheme 6). They also found a sharp melting point of adduct **14** at 260-262 ^{0}C suggesting that in formation of **14** single isomer was obtained.



Scheme 6

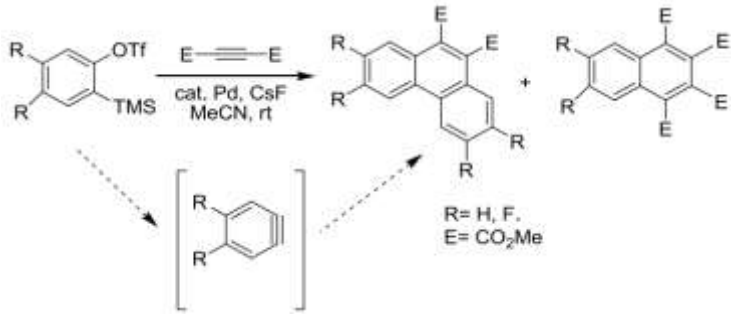
Richard C. Larock *et al.*[13] have reported synthesis of polycyclic aromatic hydrocarbons by the Pd-catalyzed annulation of alkynes. They have showed that this methodology provided an exceptionally efficient route to a wide variety of substituted polycyclic aromatic hydrocarbons from readily available starting materials, affording the products in moderate to excellent yields (Scheme 7). On optimization with a variety of internal alkynes, they obtained two general procedures which affect this annulation process, **procedure A**: 1 equiv of the organic iodide or triflate, 1.1 or 2.0 equiv of the alkyne, 5 mol % of $Pd(OAc)_2$, 2 equiv of NaOAc, 1 equiv of LiCl in DMF at 100 ^{0}C in which alkyl-substituted acetylenes provided good yield of products whereas in



Scheme 7

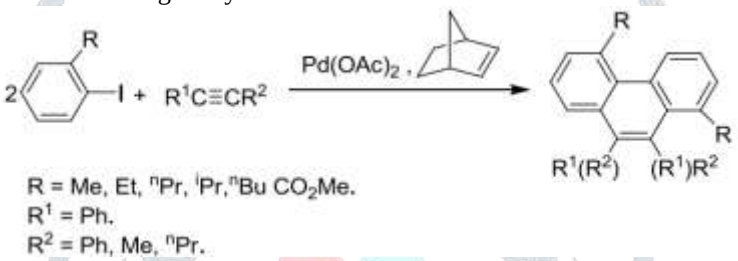
procedure B (1 equiv of the organic iodide or triflate, 1.1 or 2.0 equiv of the alkyne, 5 mol % of $Pd(OAc)_2$, 2 equiv of NaOAc, 3 equiv of nBu_4NCl in DMF at 100 ^{0}C) diaryl acetylenes and silyl acetylenes provided the product with better yield. Hence, the use of readily available silylsubstituted alkynes produced silyl aromatics which could be readily functionalized further.

In 1999, Dolores Perez and his group[14] have described that arynes can undergo palladium catalyzed co-cyclization with alkynes to afford phenanthrenes. They also investigated that mixture of phenanthrene and naphthalene were obtained in this reaction, but the selective formation of these products could also be achieved by varying the number of alkynes and changing the catalyst. Best yield of phenanthrene was obtained using 1.4 equiv. of alkynes in presence of $Pd(PPh_3)_4$ catalyst whereas $Pd_2(dba)_3$ catalyst gave naphthalene derivatives in better yield (Scheme 8).



Scheme 8

Marta Catellani and coworkers[15] have developed a new synthesis of disubstituted phenanthrenes by taking advantage of the effect of *o*-substituents on the reactivity of norbornene derived palladacycle intermediates (Scheme 9). They have examined that the yield was strongly influenced by the *ortho* substituent R. They found that aryl iodides with both primary and secondary *ortho* alkyl groups gave good to excellent results, while the presence of a *tert*-butyl substituent prevented the formation of the corresponding phenanthrene derivative. When methoxy or methoxy methylene or CO_2Me groups were employed, poor yield was obtained. However, the reaction appeared to be general for diphenylacetylenes and alkylphenylacetylenes and no significant result has been obtained with disubstituted acetylenes containing bulky substituents.

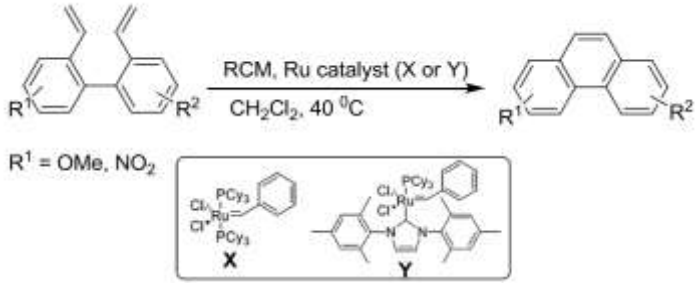


Scheme 9

George A. Kraus *et al.*[16] have synthesized phenanthrenes derivatives by condensation of formyl benzoquinone with a substituted toluene followed by *O*-methylation and cyclization using the phosphazine base (P_4-tBu). Here formyl benzoquinone reacted with a substituted toluene near ambient temperature and the resulting hydroquinone was *O*-methylated and then converted into the phenanthrene using the hindered phosphazine base P_4-tBu. They have shown that deprotonation, cyclization and dehydration occurred in a one-pot reaction to generate the phenanthrene structure (Scheme 10).

Scheme 10

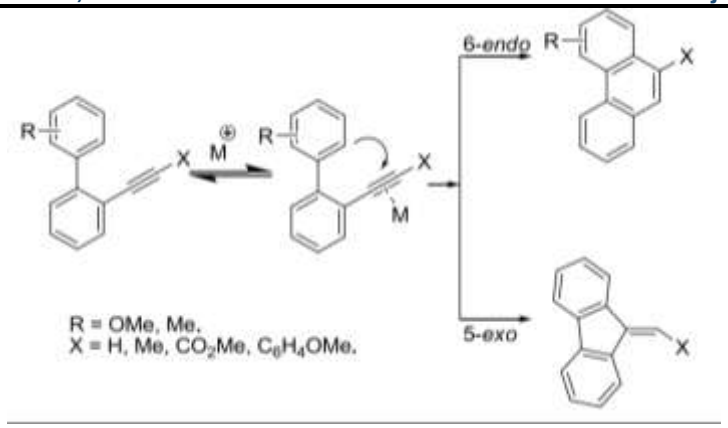
In 2004, Anna Iuliano *et al.*[17] emphasized on a general method for obtaining high yields of substituted phenanthrenes through RCM of 2,2′-divinylbiphenyls using the ruthenium-carbene complex as catalyst. Depending on the conformational rigidity of the



Scheme 11

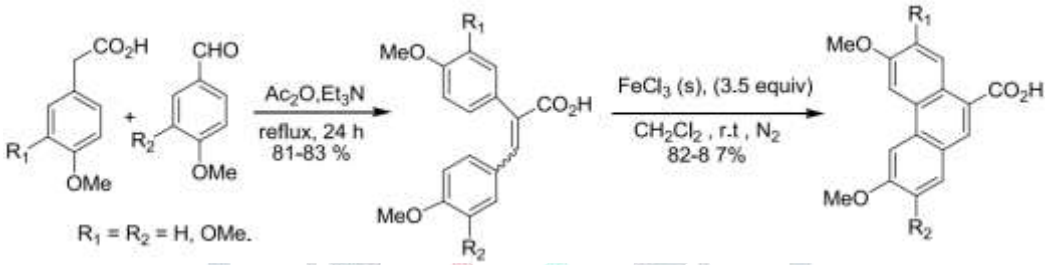
biphenyl moiety as well as the electronic properties of the substituents the product was obtained with good to excellent yield but the using of a second-generation ruthenium-carbene complex **Y** as catalyst afforded quantitative yields of the phenanthrene derivative with independent of the structure of the starting biphenyl (Scheme 11).

Alois Förstner *et al.*[18] have described that a readily available biphenyl derivatives containing an alkyne unit at one of their *ortho*-positions were converted into substituted phenanthrenes on exposure to catalytic amounts of either $PtCl_2$, AuCl, $AuCl_3$, $GaCl_3$ or $InCl_3$ in toluene. They proposed that this 6-*endo-dig* cyclization likely proceeded through initial π-complexation of the alkyne unit followed by interception of the resulting η^2-metal species by the adjacent arene ring (Scheme 12).



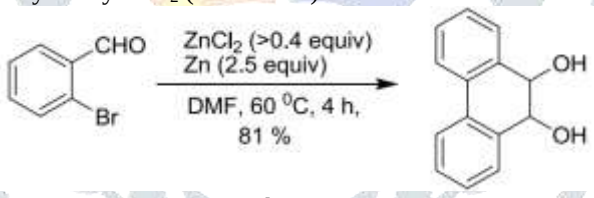
Scheme 12

Qing-Min Wang *et al.*[19] have described a preparation of polymethoxy-substituted phenanthrene-9-carboxylic acid *via* intramolecular oxidative coupling at room temperature in excellent yields (Scheme 13). Mild reaction conditions and the use of environmentally friendly $FeCl_3$ provided a novel practical route for the synthesis of the important phenanthrene ring. Using readily available pyrrole, this protocol was further applied for total synthesis of tylophorine, deoxytylophorinine, and antofine in 48%, 44%, and 46% overall yields, respectively. Here the experimental procedure was simple under mild conditions, atom economy was very high without any protecting-group and starting materials were cheap or easily prepared. Hence this short and practical method was applicable to large-scale production.



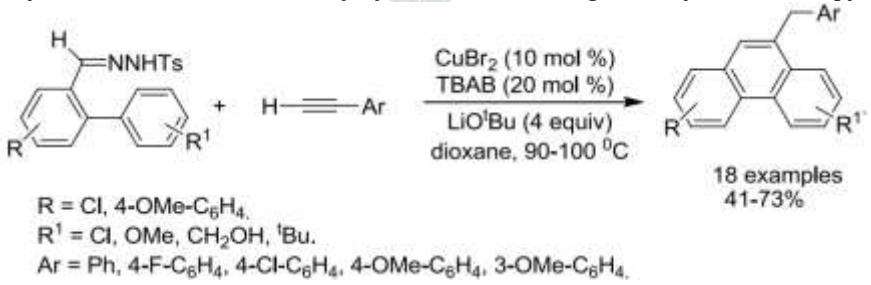
Scheme 13

Tian-pa You *et al.*[20] have developed a tandem $(Ph_3P)_2NiCl_2$-catalyzed Ullmann reaction/ZnX_2- mediated pinacol coupling. In the presence of catalyst $(Ph_3P)_2NiCl_2$ and reductant Zn, first the Ullmann reactions of *ortho*-halo aryl aldehydes generated biaryl-dialdehydes and zinc halides in situ and then the intramolecular pinacol coupling reaction of biaryl-dialdehydes occured to form 9,10-dihydrophenanthrene-9,10-diols catalyzed by ZnX_2 (Scheme 14).



Scheme 14

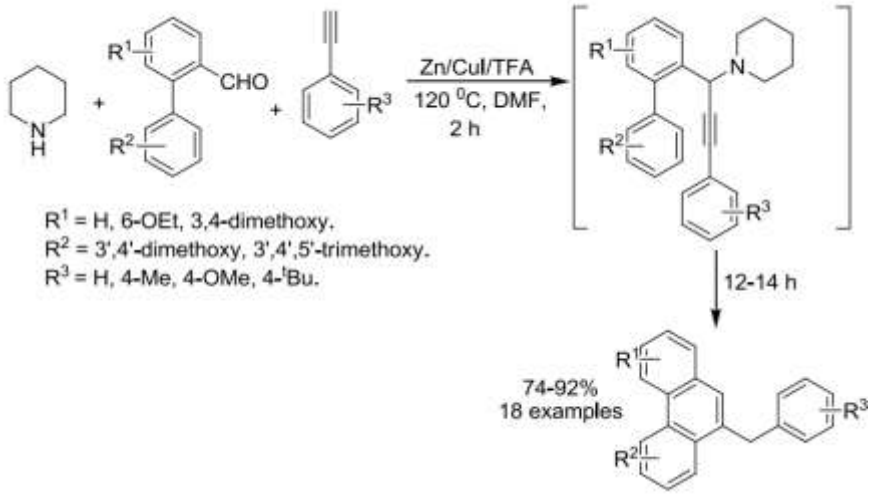
Jianbo Wang *et al.*[21] have developed a new method for the synthesis of phenanthrene derivatives from N-tosylhydrazones derivatives and terminal alkynes as substrates *via* ligand free $CuBr_2$ catalyst. The reaction involved a sequence of $CuBr_2$-catalyzed coupling/allenylation/6π electron cycloaddition/aromatization. Studying the scope of Ntosylhydrazone they have described that various substituted N-tosylhydrazones reacted with aryl terminal alkynes to afford the corresponding phenanthrenes smoothly in moderate to good yields. They also observed that the N-tosylhydrazones containing heterocycles, such as pyridine and thiophene,



Scheme 15

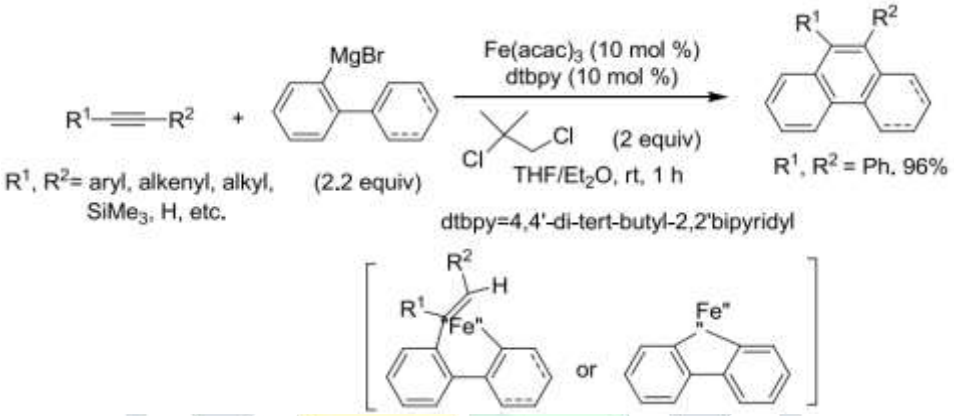
also underwent a cyclization process with the allene moiety to afford the corresponding phenanthrene products. They have also found that the free hydroxyl group could tolerate the reaction condition to afford the phenanthrene product in comparable yield (Scheme 15).

Bijoy Kundu *et al.*[22] have discovered a one-pot protocol involving Zn/CuI/TFA-catalyzed domino three-component and subsequent carbocyclization reactions. The reaction proceeded via formation of propargyl amines from biphenyl-2-carbaldehydes/terminal alkynes/piperidine followed by the elimination of piperidine and ring closure to furnish phenanthrene derivatives in good yields. This strategy involved C(sp)-H activation, CH functionalization with imine, alkyne activation, 1,5-hydride shift, β-elimination of piperidine, allene formation, 6π-cycloaddition and isomerisation domino sequence (Scheme 16).



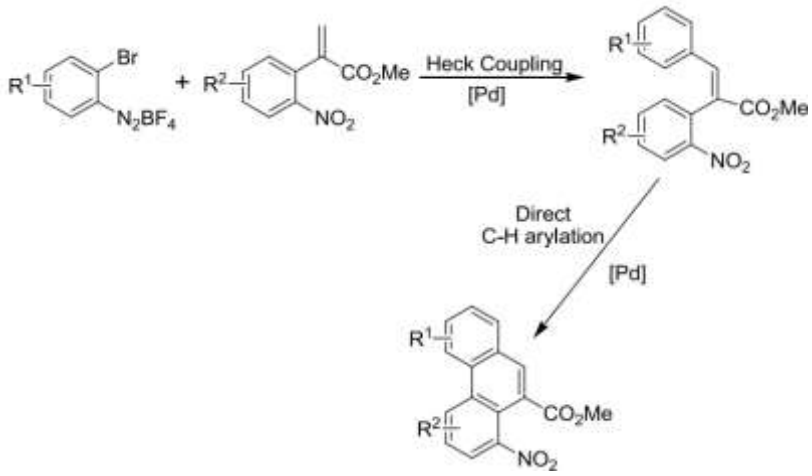
Scheme 16

Eiichi Nakamura *et al.*[23] have developed a new [4+2] benzannulation method that allowed the coupling of a variety of alkynes with diaryl and related Grignard reagents. This reaction took place under mild conditions catalyzed by iron salt and enabled the construction of sterically congested systems. These oxidative coupling conditions allowed the reaction to take place with remarkable chemoselectivity, such as the tolerance of bromide, chloride, trimethylsilyl, trifluoromethyl, and olefinic groups (Scheme 17).



Scheme 17

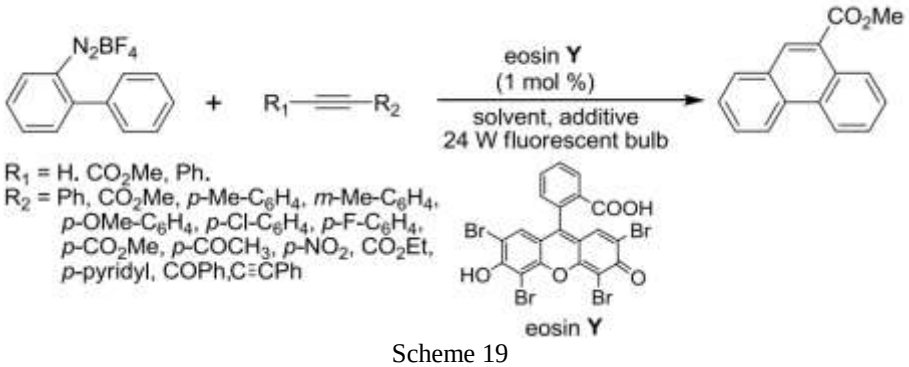
François-Xavier Felpin group[24] have reported a intramolecular direct C–H arylation leading to the formation of the phenanthrene. This process involved a highly efficient Heck coupling of aryl diazonium salts with phenyl acrylates, giving the



Scheme 18

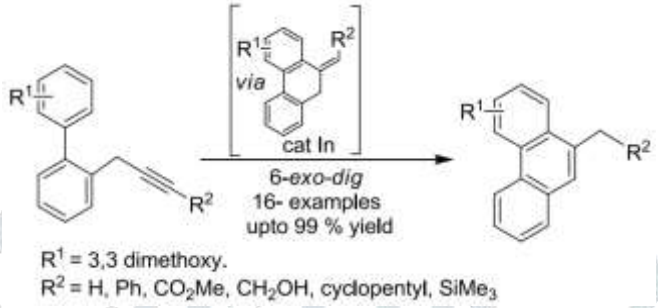
corresponding *cis*-stilbenes. Then on cyclization of stilbenes gave phenanthrenes through a direct intramolecular C–H arylation (Scheme 18).

Lei Zhou *et al.*[25] have reported metal-free, visible light-induced [4+2] benzannulation of biaryldiazonium salts with alkynes. A wide range of functional groups could be tolerated in the reaction conditions catalyzed by eosin **Y** as photoredox catalyst. They also found that various 9- substituted and 9,10-disubstituted phenanthrenes were assembled via cascade radical addition and cyclization. Finally they have developed new approach towards the phenanthrene synthesis based on visible light-induced sequential radical reactions (Scheme 19).



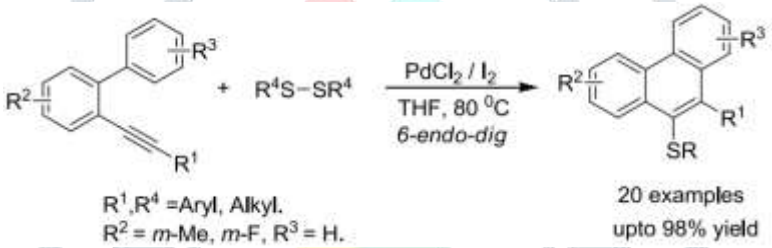
Scheme 19

In addition this, Sanghee Kim group[26] have depicted the first example of In(III)-catalyzed selective *6-exo-dig* hydroarylation of *o*-propargyl biaryls and their subsequent double bond migration to obtain functionalized phenanthrenes. They also showed that electron-rich biaryl substrates underwent hydroarylation more effectively and the substrates with various types of substituents on the alkyne could also be smoothly and selectively converted to phenanthrenes (Scheme 20).



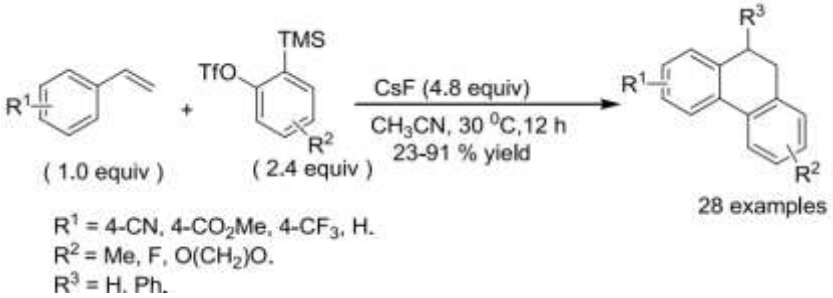
Scheme 20

A palladium-catalyzed, iodine-mediated electrophilic annulation between 2-(1 alkynyl)biphenyl and disulfide have been developed by Xing-Guo Zhang *et al.*[27] Here the combine catalyst of $PdCl_2$ and I_2 underwent electrophilic annulations with various disulfides successfully from a variety of 2-(1-alkynyl)biphenyls to afford the corresponding 9-sulfenyl phenanthrenes in moderate to excellent yields (Scheme 21).



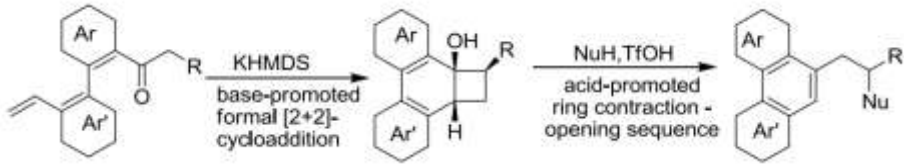
Scheme 21

In 2014, Biju groups[28] have reported a mild, general, and transition-metal-free protocol for the synthesis of 9,10-dihydrophenanthrenes. They explained that the aryne generated by the fluoride-induced 1,2-elimination of 2-(trimethylsilyl)aryl triflates, underwent an efficient cascade reaction with the differently substituted styrenes leading to the formation of 9-aryl-9,10-dihydrophenanthrene derivatives in moderate to good yields (Scheme 22).



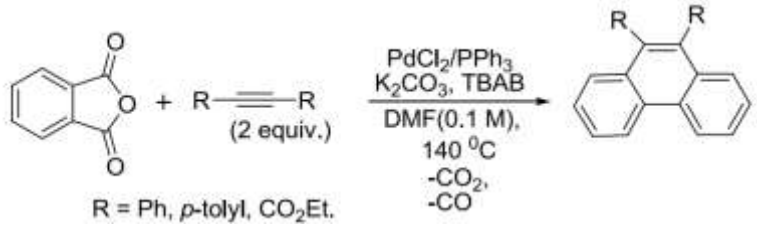
Scheme 22

In 2014 again Kiyosei Takasu *et al.*[29] have reported a base-promoted formal [2+2]-cycloaddition biaryls to provide polycyclic cyclobutanols as a step toward the synthesis of substituted polycyclic aromatic hydrocarbons and their heterocyclic analogues (Scheme 23).



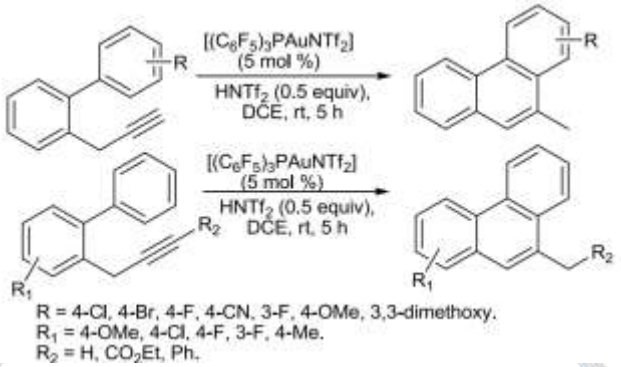
Scheme 23

Farnaz Jafarpour *et al.*[30] have developed a new method for decarboxylative and decarbonylative addition of cyclic anhydrides to alkynes. The protocol allowed for the waste minimized construction of sterically congested phenanthrenes employing a palladium catalyst (Scheme 24).



Scheme 24

A novel gold-catalyzed *6-exo-dig* cycloisomerization of *o*-propargylbiaryls has been developed by Long-Wu Ye *et al.*[31] that provides ready access to functionalized phenanthrenes in good to excellent yields. Notable features of this method were readily available starting materials, mild reaction conditions, and broad substrate scope (Scheme 25).



Scheme 25

CONCLUSION:

The synthesis of substituted phenanthrenes has evolved considerably, moving from conventional cyclization and Friedel–Crafts strategies to modern, efficient, and selective catalytic methods. Innovations such as transition-metal-catalyzed coupling, C–H activation, and photocatalytic approaches have not only broadened structural diversity but also enhanced sustainability in synthesis. These advances provide chemists with versatile tools to design and access phenanthrene derivatives with tailored properties, reinforcing their significance in pharmaceuticals, materials science, and natural product research. Overall, advancements in synthetic methodologies continue to expand the scope and applicability of substituted phenanthrenes, paving the way for the development of novel derivatives with diverse biological and functional properties.

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