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Synthesis of 9, 10-dihydrophenanthrene, Phenanthrene, Mono- and Dialkyl Phenanthrene and Octa-hydro Phenanthrene by Palladium-catalyzed Heck Reactions¥

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Abstract

A new approach of reaction mechanism to synthesize 9, 10-dihydro phenanthrene, alkyl phenanthrene, and octa-hydro phenanthrene has been developed via a palladium-catalyzed Heck reaction followed by Reverse Diels-Alder reaction of formaldehyde elimination. The procedure is useful for the synthesis of homologous compounds with a suitable starting material. This will be a new approach to synthesize the phenanthrene derivatives to originate a huge variety of natural and synthetic products for industrial and biomedical applications.

Keywords: Palladium-catalyzed; Heck reaction; Phenanthrene; Revers Diels-Alder reactions; Oxo-palladium complex.
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Article type: Research article.

1. Introduction:

Palladium-catalyzed C-C bond-forming[1-3] reaction is significant for the synthesis of carbocyclic compounds.[4] Polycyclic aromatic hydrocarbons (PAHs), more simply known as polyarenes, constitute extraordinarily large and various classes of organic molecules.[5,6] Phenanthrene is an important core structure of PAH. The phenanthrene moiety can not only be found in several natural products, of which many exhibit interesting biological activity,[7] but more recently phenanthrenes plays an important role as interesting ligands for novel catalyst systems.[8] Previously it was thought that PAHs are direct-acting carcinogens,[9] but it is now accepted that PAHs require metabolic activation to express tumorogenic reactivity.[10,11] Due to its highly lipophilic behavior, phenanthrenes and PAHs are soluble in most organic solvents, and they manifest various functions such as light sensitivity, heat resistance, conductivity; emit ability, corrosion resistance, and physiological action.[12] Numerous synthetic efforts for the preparation of PAHs have been reported by different groups, of which most common is oxidative photocyclization of stilbene derivatives.[13] Among other methods intramolecular Diels–Alder reaction,[14] flash vacuum pyrolysis,[15-18] olefin metathesis,[19] Friedel-Crafts type cyclization,[20] dimerization or trimerization of acetylenes and arynes,[21,22] transition metal-catalyzed cycloisomerization,[23,24] etc. are the key step for making of a benzene ring for the synthesis of PAHs. Palladium-catalyzed C-H bond activation has been used widely in numerous organic syntheses since this reaction gives a solution for the construction of carbo- and heterocycles from the corresponding halides and triflates.[25-31] Some groups have developed a novel Palladium-catalyzed 1,4 migration/C-H activation for the synthesis of complex fused polycycles.[32-34] The major sources of PAHs are crude oil, coal, oil shale. Methylphenanthrene belongs to an important group of alkyl-aromatic hydrocarbons which are present in natural environments. There is a great variety of methods which are available for the synthesis of phenanthrene and its derivatives. Perhaps the most extensive method is the classical Haworth synthesis. The importance of these PAH compounds attracted researchers to synthesize new phenanthrene derivatives and also to try alternate reaction mechanisms. In recent times (2019), Juan *et al.* has derived a variety of phenanthrene derivatives by palladium-catalyzed controlled Suzuki–Miyaura coupling reaction followed by C–H activation.[35] Scientists also reported efficient approaches for synthesizing functionalized phenanthrenes with a broad substrate range and good functional groups.[36] Recently Jin *et.al.* reported a series of azole-fused phenanthrenes which are found to be important

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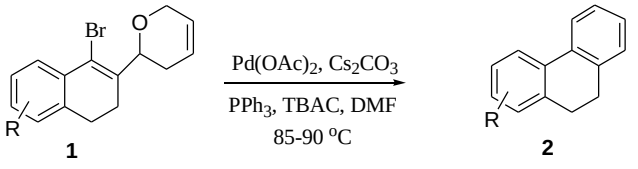
¥ *All authors want to dedicate this article to Prof. J. K. Ray on his 72nd Birthday (06- January 1950) celebration.*

redox-active organic functional materials.[37] Being inspired by the importance of phenanthrene derivatives and also to find out some new reaction pathways, we have tried a new mechanism to synthesize a series of phenanthrene derivatives which are reported in this article.



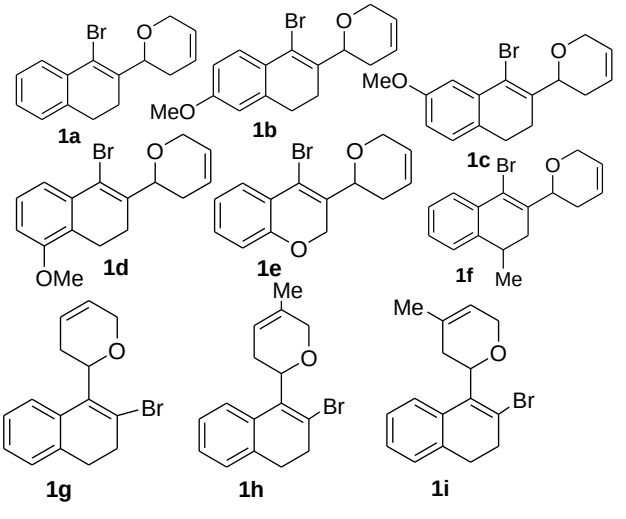
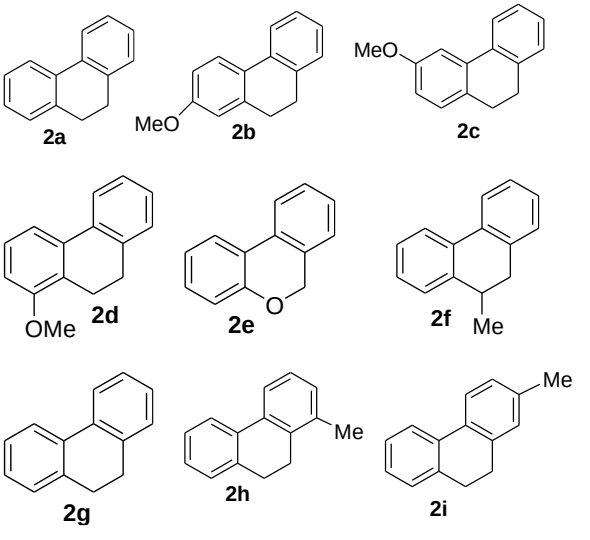
Scheme 1. Synthesis of 9,10-dihydro phenanthrene compounds by palladium-catalyzed 6π electrocyclic reaction.

2. Result and discussion

From our laboratory,[38] we first reported a novel and rapid synthesis of substituted 9, 10-dihydrophenanthrene compounds by a palladium-catalyzed 6 π electrocyclic reaction (Scheme 1).

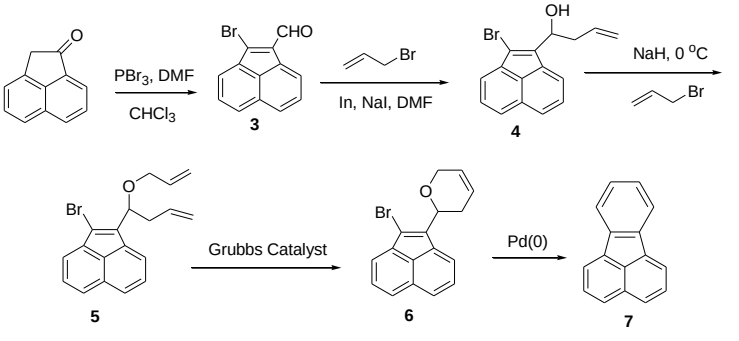
When 1 was treated with $Pd(OAc)_2$ (10 mol%), Cs_2CO_3 (2 Eqv.), PPh_3 (0.5 Eqv.), TBAC (1 Eqv.), in DMF solvent, heated at 85 °C -90 °C for 1.5 h – 2 h, the unexpected products 2 was obtained in good yield. A fused aromatic ring resulted from cleavage of the pyran ring present in the substrate followed by ring closer and aromatization. We examined the scope of the reaction with different substituted 2-(1-bromo-3,4-dihydro-naphthene-2-yl)-3,6-dihydro-2H-pyran, 2-(2-bromo-3,4-dihydro-naphthene-1-yl)-3,6-dihydro-2H-pyran, 4–bromo-3-(3,6-dihydro-2H-pyran-2-yl)-2H-chromene and 2-(2-bromo-acenaphthylen-yl)-3,6-dihydro-2H-pyran (1a-i) as shown in (Table 1) with satisfactory yields were obtained in this reaction. This reaction represents a unique method for the preparation of 9,10-dihydrophenanethrene and its analogs from vinyl bromoaldehydes.

Table1. Reactents and products of scheme-1.

Reactants	Products
	

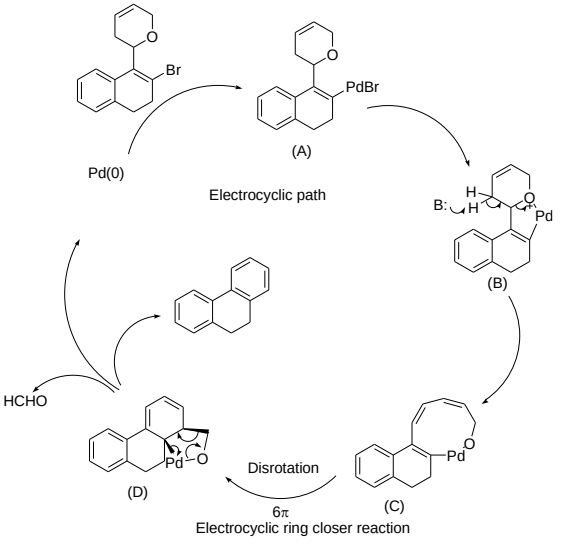
Reagent and condition: 1 (a-i) (1 equiv), $Pd(OAc)_2$ (10 mol %), PPh_3 (0.5 equiv), Cs_2CO_3 (2 equiv), n-Bu_4NCl (1 equiv) DMF (6-7 mL), heated at 85-90 °C.

By using this methodology, we have synthesized Fluoranthene in the following way (Scheme 2):



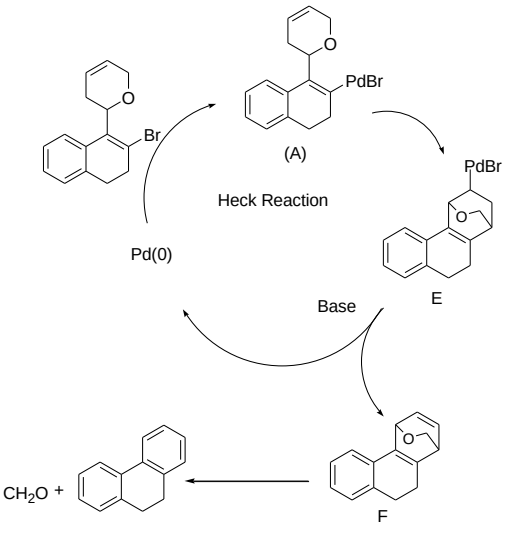
Scheme 2. Synthesis of fluoranthene from vinyl bromoaldehydes.

Initially, we thought that the reaction mechanism (Scheme 3) occurs through a 6π electrocyclic ring closer reaction followed by formaldehyde elimination. Our proposed catalytic cycle involves initial oxidation of the Pd(0) to palladium (II) intermediate (A) via oxidative addition of the Pd(0) to the substrate, which then co-ordinates with oxygen to generate the intermediate (B).



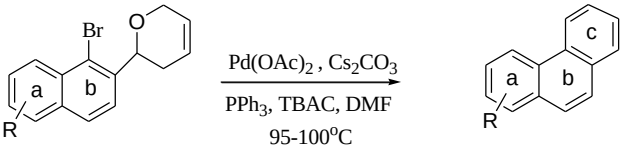
Scheme 3. 6π electrocyclic ring closer reaction mechanism.

Then the proton abstraction is followed by rearrangement to generate cyclic O-Pd complex (C). Then the 6π electrocyclic ring closer disrotation reaction forming complex (D), followed by formaldehyde elimination to afford 9,10-dihydrophenanthrene. Lan *et al.* proposed an alternative mechanism of our reaction that occurs through an intramolecular Heck reaction. Using theoretical calculations they investigated both the mechanisms and find that the intramolecular Heck mechanism (Scheme 4)[39] is lower in energy than the electrocyclic pathway (Scheme 3).

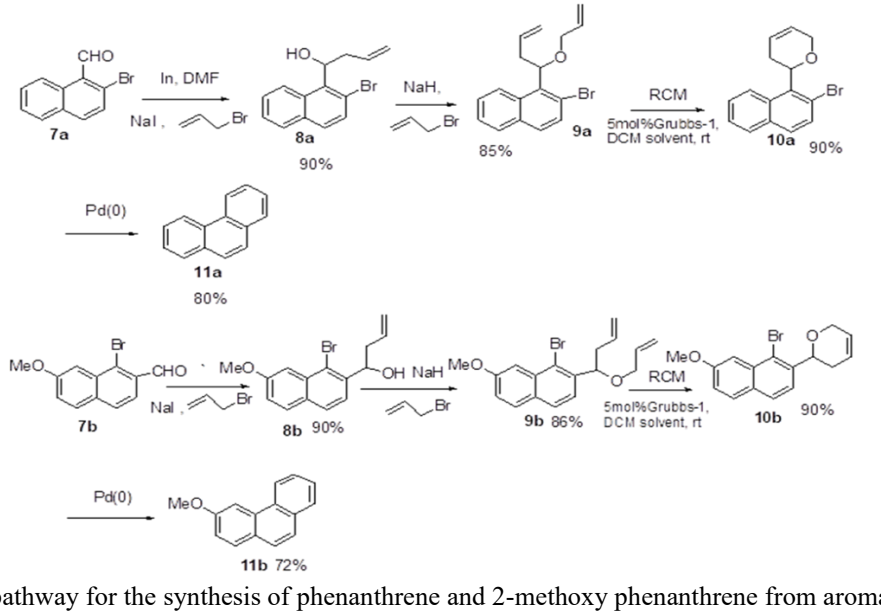


Scheme 4. Intramolecular Heck reaction mechanism[39] proposed by Lan *et al.*

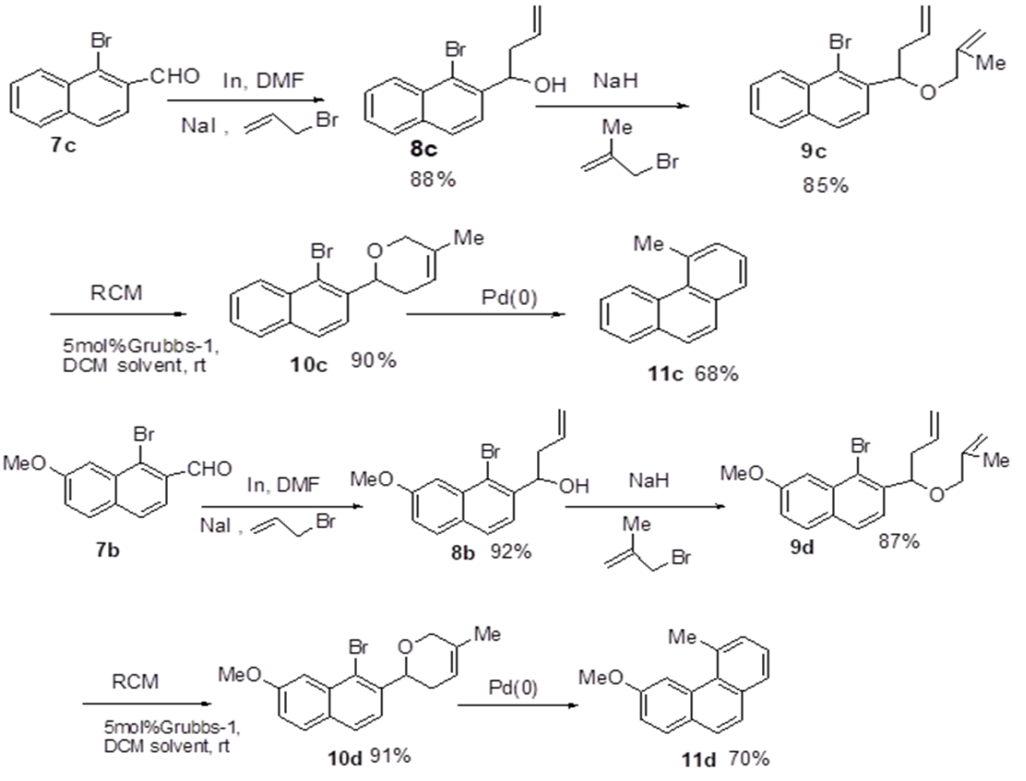
We have prepared 9,10-dihydrophenanthrenes by palladium-catalyzed reaction first from vinyl bromoaldehydes. Then we have utilized our methodology for the aromatic bromoaldehydes system, and we got a successful result to synthesize phenanthrene and alkyl phenanthrene[40] in moderate to good yield. The significance of this method is that we can introduce one or two alkyl groups at newly formed benzene ring (c) of phenanthrene (Scheme 5).



Scheme 5. Synthesize phenanthrene and alkyl phenanthrene from aromatic bromoaldehydes.



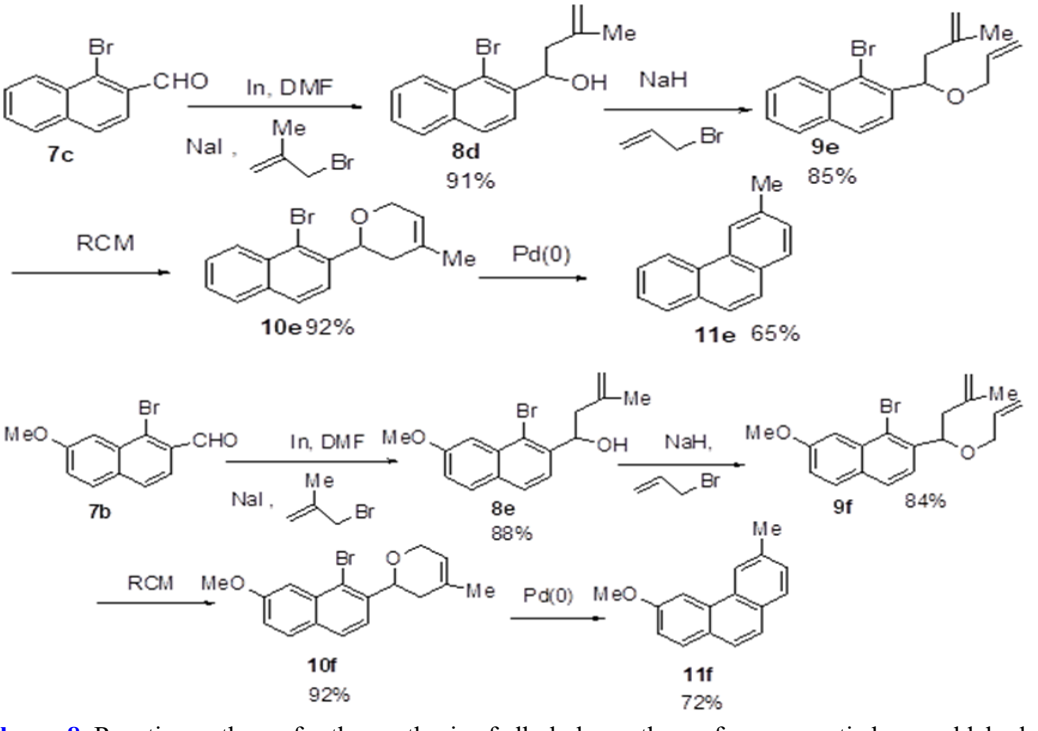
Scheme 6. Reaction pathway for the synthesis of phenanthrene and 2-methoxy phenanthrene from aromatic bromoaldehydes.



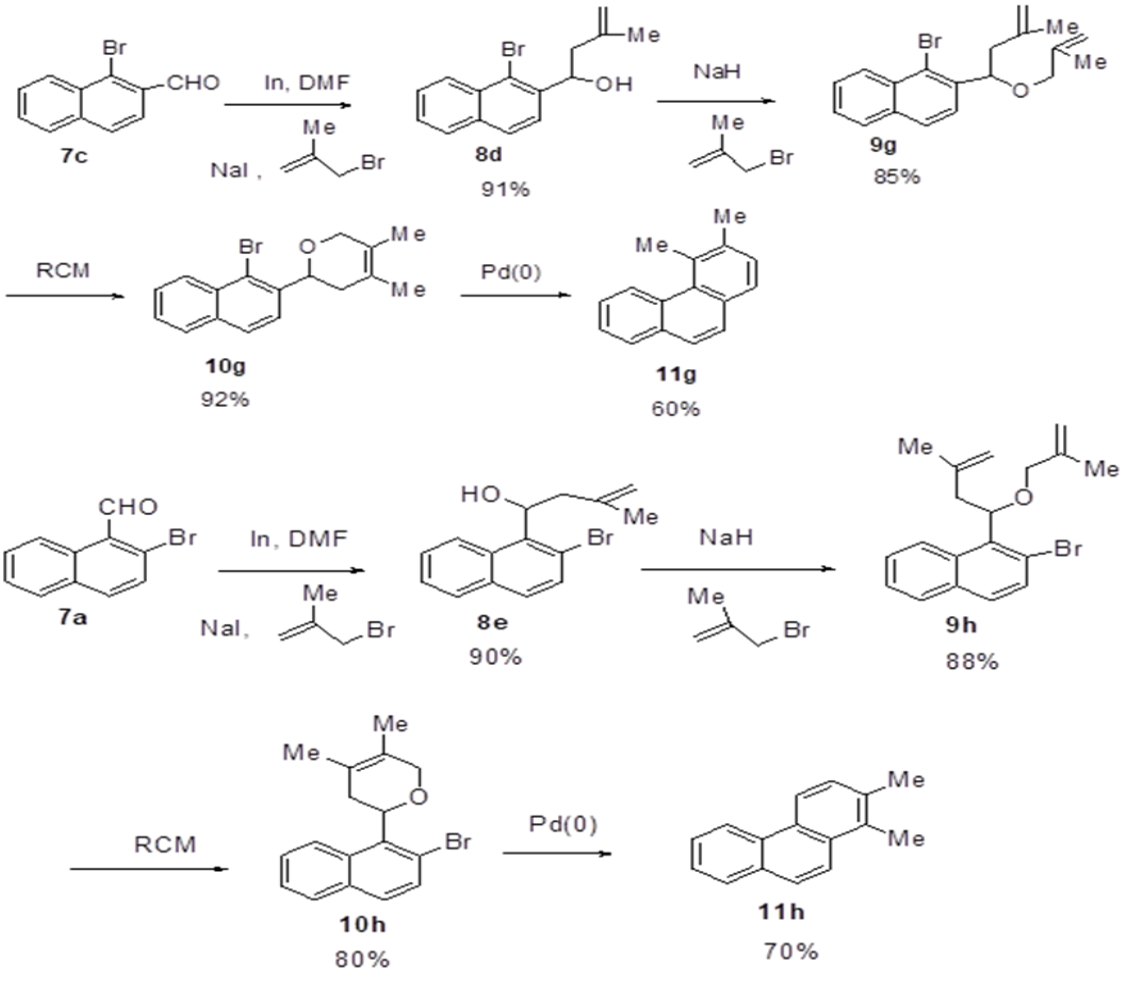
Scheme 7. Reaction pathway for the synthesis of alkyl phenanthrene from aromatic bromoaldehydes.

First homo allyl alcohols 8a and 8b were obtained from aromatic bromoaldehydes 7a, 7b by the reaction with allyl bromide, sodium iodide in presence of indium metal. These alcohols were converted to diallylated compounds 9a and 9b in presence of sodium hydride and allyl bromide in THF medium at 0 °C temperature which was subjected to ring-closing metathesis (RCM) reaction by Grubbs catalyst to obtain desired cyclic precursor 10a and 10b respectively. These cyclic precursors were finally treated with $Pd(OAc)_2$, PPh_3, Cs_2CO_3 in DMF solvent at 85-90 °C to obtain phenanthrene 11a and 2-methoxy phenanthrene 11b in good yield which is shown in Scheme 6.

To introduce alkyl groups, homo allyl alcohols (8b and 8c) were obtained first from aromatic bromoaldehydes 7b and 7c by the reaction with allyl bromide, sodium iodide in presence of indium metal. These alcohols were converted to diallylated



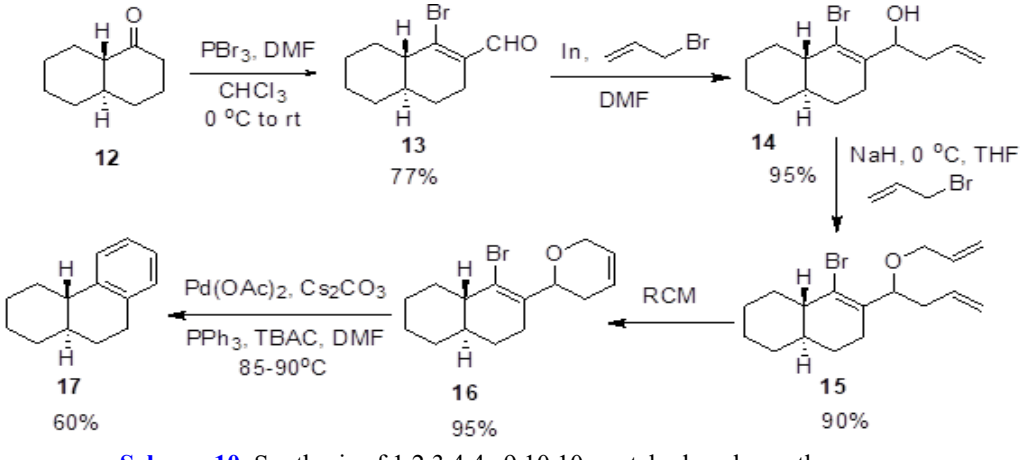
Scheme 8. Reaction pathway for the synthesis of alkyl phenanthrene from aromatic bromoaldehydes.



Scheme 9. Reaction pathway for the synthesis of dialkyl phenanthrene from aromatic bromoaldehydes.

compounds 9c and 9d in presence of sodium hydride and allyl bromide in THF medium at 0 °C temp., which were subjected to metathesis reaction by Grubbs catalyst to obtained desired cyclic precursor 10c and 10d respectively. These cyclic precursors were finally treated with $Pd(OAc)_2$, PPh_3, Cs_2CO_3 in DMF solvent at 85-90 °C to obtained alkyl phenanthrenes 11c, 11d respectively in good yield which is shown in (Scheme 7).

To alternating the position of methyl group, the same aromatic bromoaldehydes 7b and 7c were treated with methyallylbromide, sodium iodide in presence of indium metal to obtained compounds 8d, 8e. These alcohols were then converted to diallylated compounds 9e, 9f in presence of sodium hydride and allyl bromide in THF medium at 0 °C which were contingent upon Grubbs catalyzed metathesis reaction to obtain desired cyclic precursor 10e, 10f respectively. These cyclic precursors were finally treated with $Pd(OAc)_2$, PPh_3, Cs_2CO_3 in DMF solvent at 85-90 °C to obtained alkyl phenanthrenes 11e, 11f respectively in good yield which is shown in (Scheme 8).



Scheme 10. Synthesis of 1,2,3,4,4a,9,10,10a-octahydro-phenanthrene.

We can introduce two alkyl groups in phenanthrene ring at the same time. First homo allyl alcohols 8d and 8e were obtained from aromatic bromoaldehydes 7a, 7c by the reaction with allyl bromide, sodium iodide in presence of indium metal. These alcohols were converted to diallylated compound 9g and 9h in presence of sodium hydride and allyl bromide in THF medium at 0 °C which were subjected to metathesis reaction by Grubbs catalyst to Obtained desired cyclic precursor 10 g and 10h respectively. These cyclic precursor were finally treated with $Pd(OAc)_2$, PPh_3, Cs_2CO_3 in DMF solvent at 85-90 °C to obtained dialkyl phenanthrenes 11g and 11h in good yield which is shown in (Scheme 9).

By using above methodology, we can synthesized 1,2,3,4,4a,9,10,10a-octahydro-phenanthrene (Scheme 10) which is a core structure of various natural product like totarol, *cis*-4*a*-methyloctahydrophenanthrene, podocarpa-8,11,13-triene.

3. Conclusion

In this article, we have reported a series of different phenanthrene derivatives like 9, 10-dihydrophenanthrene, phenanthrene, mono- and di-alkylated phenanthrene, and octahydrophenanthrene derivatives with this new reaction pathway. This method also useful for the synthesis of higher homologous polynuclear aromatic hydrocarbons provided an efficient route to the synthesis of suitable starting material. This will be a new approach to synthesize the phenanthrene derivatives to originate a wide variety of natural and synthetic products with industrial and biomedical applications.

4. Experimental

The general procedures for the synthesis of compounds are reported in supporting information file (Text S1) along with all spectral graphs (Fig. S1). Some selected analytical data are reported here for the reference of synthesized compounds.

4.1 Some selected data:

4.1.1 1-(2-Bromo-naphthalen-1-yl)-3-methyl-but-3-en-1-ol (8e)

Liquid, ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 1.93 (s, 3H), 2.52 (m, 2H), 2.93 (m, 1H), 4.96 (s, 1H), 4.99 (s, 1H), 5.90 (dd, 1H, J = 3.6Hz, J = 10.4Hz), 7.41-7.56 (m, 2H), 7.58 (s, 2H), 7.80 (dd, 1H, J = 6.4Hz, J = 8Hz), 8.83 (d, 1H, J = 8Hz). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 22.29, 44.75, 73.35, 114.027, 121.11, 125.90(2C), 126.16, 128.70, 129.48, 130.16, 132.25, 133.67, 136.81, 142.38. Anal. Calcd for $C_{15}H_{15}BrO$: C, 61.87; H, 5.19 Found: C, 61.95; H, 5.13.

4.1.2 1-(1-Bromo-7-methoxy-naphthalen-2-yl)-but-3-en-1-ol (8b)

Liquid, ^{1}H NMR ($CDCl_3$, 200 MHz) δ: 1.89 (s, 3H), 1.98 (brs), 2.23-2.33 (m,1H), 2.56-2.62 (m, 1H), 3.93 (s, 3H), 4.92 (m, 2H), 5.42 (d, 1H, J = 3Hz), 5.47 (d, 1H, J = 3Hz), 7.10 (d, 1H, J = 2.4Hz), 7.17-7.23 (m, 1H), 7.66 (m, 2H), 8.20 (d, 1H, J = 9Hz). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 21.10, 47.73, 55.42, 72.89, 107.05, 115.20, 119.91, 121.5, 124.72, 127.91, 127, 128.90, 136.27, 138.51, 143.57, 159.07. Anal. Calcd for $C_{16}H_{17}BrO_2$: C, 59.83; H, 5.33 Found: C, 59.95; H, 5.21.

4.1.3 1-Bromo-2-[1-(2-methyl-allyloxy)-but-3-enyl]-naphthalene (9c)

Liquid, ^{1}H NMR ($CDCl_3$, 200 MHz) δ: 1.83(s, 3H), 2.57 (t, 2H, J = 6.4Hz), 3.73(d, 1H, J = 12.6Hz), 3.84 (d, 1H, J = 12.6Hz), 4.91-5.18 (m, 5H), 5.85-6.02 (m, 1H), 7.49-7.65 (m, 3H), 7.84 (d, 2H, J = 8Hz), 8.34 (d, 1H, J = 8Hz). ^{13}C NMR ($CDCl_3$, 50 MHz) δ: 19.76, 41.23, 72.89, 79.98, 112.44, 117.18, 123.02, 124.81, 126.54, 127.47, 128.09, 128.17, 132.19, 134.20, 134.61, 139.40, 142.09.

4.1.4 1-Bromo-2-[3-methyl-1-(2-methyl-allyloxy)-but-3-enyl]-naphthalene (9g)

Liquid , ^{1}H NMR ($CDCl_3$, 200 MHz) δ: 1.75 (s, 3H), 1.90 (s, 3H), 2.49 (m, 2H), 3.68 (d, 1H, J = 12.4Hz), 3.83 (d, 1H, J = 12.4Hz), 4.83-4.99 (m, 4H), 5.25 (dd, 1H, J = 5.4Hz, J = 7.6Hz), 7.49-7.68 (m, 3H), 7.84 (dd, 2H, J = 2.4Hz, J = 8.6Hz), 8.36 (dd, 1H, J = 8.2Hz, J = 0.8Hz). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 19.88, 23.07, 45.41, 73.22, 79.57, 112.50, 113.21, 123.07, 124.93, 126.66, 127.55(2C), 128.32(2C), 132.41, 134.39, 140.14, 142.36, 142.58. Anal. Calcd for $C_{19}H_{21}BrO$: C, 66.09; H, 6.13 Found: C, 59.95; H, 6.25.

4.1.5 2-(1-bromo-naphthalen-2-yl)-5-methyl-3,6-dihydro-2H-pyran (10c)

Liquid, ^{1}H NMR ($CDCl_3$, 200 MHz) δ: 1.70 (s, 3H), 2.04-2.22 (m, 1H), 2.42-2.55 (m, 1H), 4.19-4.40 (m, 2H), 5.15 (dd, 1H, J = 3.4Hz, J=10.4Hz), 5.64 (m, 1H), 7.46-7.63 (m, 2H), 7.71 (m,1H), 7.79-7.87 (m,2H), 8.32 (d, 1H, J = 8.2Hz). ^{13}C NMR ($CDCl_3$, 50 MHz) δ: 18.67, 31.86, 69.99, 75.91, 118.94, 121.36, 124.36, 126.44, 127.41, 127.45, 128.18, 128.29, 132.07, 133.10, 134.06, 140.10.

4.1.6 2-(1-bromo-naphthalen-2-yl)-4,5-dimethyl-3,6-dihydro-2H-pyran (10g)

Liquid, ^{1}H NMR ($CDCl_3$, 200 MHz) δ: 1.66 (s, 3H), 1.77 (s, 3H), 2.10 (m, 2H), 4.13-4.38 (m, 2H), 5.23 (dd, 1H, J = 3.6Hz, J = 10.2Hz), 7.47-7.64 (m, 2H), 7.70-7.89 (m, 3H), 8.35 (dd, 1H, J = 0.8Hz, J = 8.2Hz). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 14.03, 18.46, 36.95, 70.50, 76.52, 121.35, 124.12, 124.49, 124.61, 126.48, 127.45, 127.53, 128.25, 128.37, 132.16, 134.14, 140.28. Anal. Calcd for $C_{17}H_{17}BrO$: C, 64.37; H, 5.40 Found: C, 64.52; H, 5.25.

4.1.7 4-methyl-phenanthrene (11c)

Solid, mp 52-54 °C, ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 3.17 (s, 3H), 7.50 (m, 2H), 7.59-7.67 (m, 2H), 7.71 (s, 2H), 7.79 (m, 1H), 7.94 (dd, 1H, J = 2Hz, J = 8.4Hz), 8.94 (d, 1H, J = 8Hz). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 27.37, 125.52, 125.74, 125.83, 127.04, 127.41, 127.47, 127.96, 128.66, 130.03, 131.18, 131.62, 133.44, 133.69, 135.49.

4.1.8 1,2-dimethyl-phenanthrene (11h)[41]

Solid, mp 141-143 °C, ^{1}H NMR ($CDCl_3$, 200 MHz) δ : 2.54 (s, 3H), 2.66 (s, 3H), 7.47 (dd,1H, J = 8.4Hz), 7.54-7.64 (m, 2H), 7.76 (d, 1H, J = 9.2Hz), 7.87 (d,1H, J = 8.4Hz), 8.02 (d,1H, J = 9.2Hz), 8.49 (d, 1H, J = 8.4Hz), 8.67 (d, 1H, 8Hz).

4.1.9 1,2,3,4,4a,9,10,10a-octahydro-phenanthrene (17)

Liquid, ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 1.12-1.51 (m, 6H), 1.77 (m, 3H), 1.90 (m, 1H), 2.25 (m 1H), 2.44 (dd, 1H, J = 3.2H, J = 12.8Hz), 2.78-2.96 (m, 2H), 7.06-7.15 (m, 3H), 7.29 (d, 1H, J = 7.6Hz). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: Anal. Calcd for $C_{14}H_{18}O$: C, 90.26; H, 9.74 Found: C, 90.57; H, 9.49. HRMS: calcd for $C_{14}H_{19}$ $[M+H]^+$ 187.1489 found 187.1484.

Abbreviation

PAH = Polycyclic aromatic hydrocarbons, DMF = Dimethylformamide, TBAC = Tetrabutylammonium chloride, THF = Tetrahydrofuran, RCM = Ring-closing metathesis.

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Supporting Information

Applicable, Selective ^{1}HNMR and ^{13}CNMR of synthesized compounds are available as Supplementary Information.

Conflict of Interest

There is no conflict of interest.

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