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# ONE POT MULTI COMPONENT SYNTHESIS OF NOVEL 14-SUBSTITUTED-14H-DIBENZOXANTHENE DERIVATIVES

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***Abstract*: The one pot synthesis of 14-substituted-14H-dibenzoxanthenes 1a-e in good yields were synthesized by the reaction of different aryl hetero cyclic aldehydes 2a-e with 2- naphthol 3 in presence of boric acid under solvent free conditions. The structures of the synthesized compounds were established based on spectral data ($^1$H-NMR, $^{13}$C-NMR, IR and MS).**

***Keywords***— *Dibenzoxanthenes, 2-naphthol, Hetero cyclic aryl aldehydes, Boric acid: Solvent Free Synthesis.*

## I. INTRODUCTION

Xanthenes and benzoxanthenes are having multiple biological and pharmaceutical applications. They are cited as active oxygen heterocyclic compounds possessing biological applications such as anti-bacterial [2], anti-inflammatory [1], anti-viral [3]. In addition to pharmaceutical applications, these compounds have been also used in the laser technologies [4], $P^H$ sensitive fluorescent material [5], photodynamic therapy [6] and antagonists for paralyzing action of zoxazolamine [7].
A number of synthetic methods have been developed for the synthesis of xanthenes and benzoxanthenes [8-12]. These synthetic methods includes the reaction of alkyl or aryl aldehydes with 2-naphthol in the presence of boric acid under solvent free condition. Here the author synthesized 14-sudstituted-14H-dibenzoxanthenes by using heterocyclic aryl aldehydes because of the great biological importance of heterocyclic aldehydes in both medicinally and industrially. Heterocyclic compounds have received considerable attention due to their broad range of biological activities and many of the pharmaceuticals and agrochemicals. For the preparation of 14-sudstituted-14H-dibenzoxanthenes derivatives some catalysts have been reported such as acetic acid and sulphuric acid [13], acetic acid and hydrochloric acid [14], $H_3PO_4$ or $HClO_4$ [15], bismuth nitrate, P-Toluene sulfonic acid [16], sulfamic acid [17], $I_2$[18], Oxalic acid [19], silica sulphuric acid [20], and $NH_4H_2PO_4$ [21]. However these methods experience many draw backs like low yields, prolonged reaction time, use of toxic organic solvents, the requirement of special apparatus and harsh reaction conditions. Because of these practical limitations, an efficient catalyst boric acid has been introduced. The use of boric acid catalyst improves the yield and rate of the reaction. Catalytic action of boric acid with different catalysts, oxalic acid, bismuth nitrate and perchloric acid has been shown in Table-1, Table-2, and Table-3.

**Catalytic action of boric acid in comparison with oxalic acid for the preparation of 14-substituted-14H-dibenzo xanthene derivatives**:
Table-1:

| Entry | Product | With boric acid | | With oxalic acid | |
|---|---|---|---|---|---|
| | | Time in hrs | Yield | Time in hr | Yield |
| 1 | 1a | 0.5 | 90% | 1.5 | 65% |
| 2 | 1b | 1.5 | 80% | 2.5 | 55% |
| 3 | 1c | 1 | 85% | 2 | 60% |
| 4 | 1d | 1 | 90% | 2 | 63% |
| 5 | 1e | 1.5 | 87% | 3.5 | 52% |

**Catalytic action of boric acid in comparison with bismuth nitrate for the preparation of 14-substituted-14H-dibenzo xanthenes derivatives**:
Table-2:

| Entry | Product | With boric acid | | With Bi(NO)$_3$ | |
|---|---|---|---|---|---|
| | | Time in hrs | Yield | Time in hr | Yield |
| 1 | 1a | 0.5 | 90% | 1 | 60% |
| 2 | 1b | 1.5 | 80% | 2 | 65% |
| 3 | 1c | 1 | 85% | 1.5 | 58% |
| 4 | 1d | 1 | 90% | 1.5 | 62% |

| | | | | | |
|---|---|---|---|---|---|
| 5 | 1e | 1.5 | 87% | 2.5 | 50% |

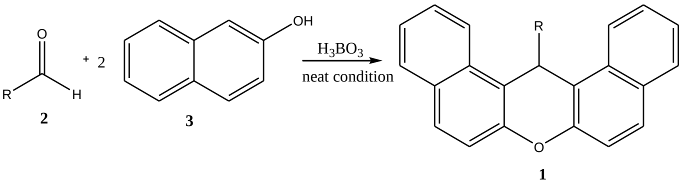
**Catalytic action of boric acid in comparison with Perchloric acid for the preparation of 14-substituted-14H-dibenzo xanthenes derivatives**:
Table-3**:**

| **Entry** | **Product** | **With boric acid** | | **With perchloric acid** | |
|---|---|---|---|---|---|
| | | **Time in hrs** | **Yield** | **Time in hr** | **Yield** |
| 1 | 1a | 0.5 | 90% | 2.5 | 52% |
| 2 | 1b | 1.5 | 80% | 3.5 | 65% |
| 3 | 1c | 1 | 85% | 3 | 55% |
| 4 | 1d | 1 | 90% | 2.5 | 66% |
| 5 | 1e | 1.5 | 87% | 3.5 | 50% |

Boric acid is a useful and environmentally benign catalyst and it is used in numerous reactions [22-27] because it is readily available and inexpensive reagent and can conveniently be handled and easily removed from the reaction mixture. So boric acid is the suitable catalyst for the synthesis of 14-substituted-14H-dibenzoxanthenes. This method includes various advantages like simplicity of handling of reaction, solvent free conditions, low cost and this is eco-friendly method. The use of boric acid improves the yield and rate of the reaction.

## II. RESULTS AND DISCUSSION

The development of catalytic systems for various environmentally friendly procedures for several organic transformations involving cyclo condensations is an active on-going research area, thus there is a scope for further improvement by changing substituents which will give better biological activities. So, we have utilized various heterocyclic aryl aldehydes as efficient substituents for synthesizing 14-substituted -14H-dibenzo xanthenes in our laboratory.
In an initial experiment using traditional conditions, benzaldehyde, 2-naphthol, boric acid taken in the ratio's of 1:2:0.2,the reaction mass was stirred at 120$^0$ C, after completion of the reaction the reaction mixture was cooled to 25$^0$ C, and water was added, further the reaction mass was stirred for 10 min. The obtained solid 14-substituted-14H-dibenzoxanthenes derivatives was collected by filtration and purified by recrystallization from ethanol. By using heterocyclic aryl aldehydes with 2-naphthol, surprisingly, a significant improvement was observed and the biological activity of the product was increased. Due to these anti-viral, anti-fungal, anti-inflammatory, anti-tumour, anti-biotic, antidepressant, anti-malarial, anti HIV, anti-microbial, anti-bacterial activities, the newly synthesized 14-substituted-14H-dibenzoxanthene derivatives using heterocyclic aryl aldehydes have great significant importance in pharmaceutical industries.

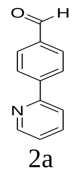
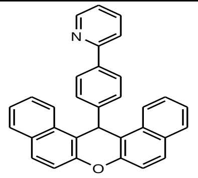
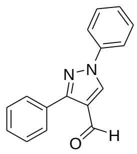
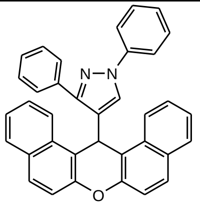
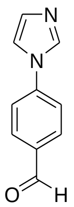
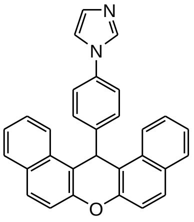


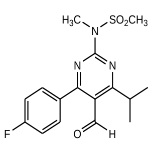
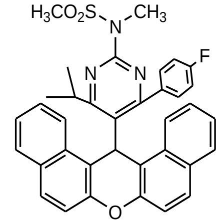
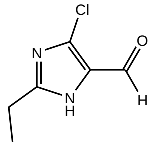
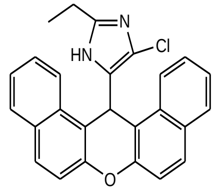
**Scheme-1: Reported synthetic scheme for 14-substituted14H-dibenzoxanthenes:**

Encouraged by this success, we attempted the reaction of 2-naphthol with different hetero atom containing aryl aldehydes with boric acid producing the respective 14-substituted-14H-dibenzoxanthene derivatives which are shown in Table-4. It is noteworthy to mention that the structural variation of the aldehydes and substituent on the aromatic ring show obvious effect on this conversion because the desired product were obtained with high biological activities.

**Table: 4**

**Synthesis of 14-substituted-14H dibenzoxanthene derivatives by using different heterocyclic aryl aldehydes through cyclo condensation using boric acid as a catalyst:**

| **Entry** | **Aldehyde (R)** | **Product** | **Time** | **Yield** |
|---|---|---|---|---|
| **1** |  <br>2a |  <br>1a | 0.5 hrs | 90% |
| **2** |  <br>2b |  <br>1b | 1.5 hrs | 80% |
| **3** |  <br>2c |  <br>1c | 1hr | 85% |

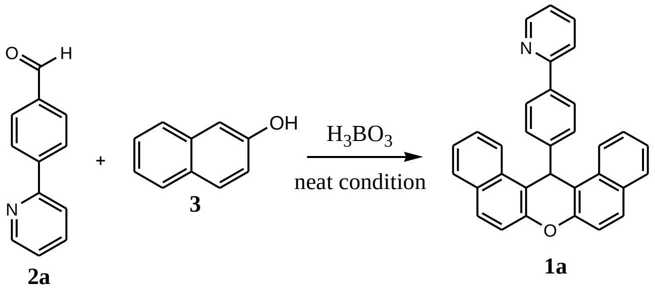
| | | | | |
|---|---|---|---|---|
| **4** |  2d |  1d | 1hr | 90% |
| **5** |  2e |  1e | 1.5hr | 87% |

In entry (1), the 4-(2-pyridyl)benzaldehyde is a six membered heterocyclic compound containing one nitrogen atom which is called as pyridine has considerable interest in different areas such as medicinal, pesticide, polymer and material science. In entry (2), the 1, 3-diphenyl pyrazole-4-carbaldehyde contains pyrazole nucleus which has great significance in the field of pharmacy as well as to develop newer anti-cancer agent. In entry (3), the 4-imidazol-1-yl benzaldehyde contains a five membered heterocyclic ring containing two nitrogen atoms which is called as imidazole and are an important class of heterocyclics and include many substances of both biological and chemical interest. They are part of a large number of highly significant bio molecules such as the essential amino acid histidine and related compounds, biotin, and the imidazole alkaloids. In entry (4), the N-[4-(4-fluorophenyl)-5-formyl-6-isopropyl-pyrimidin-2-yl]-N-methyl-methane sulphonamide contains a six membered heterocyclic ring containing two nitrogen atoms which is called as pyrimidine and heterocyclics containing pyrimidine moiety are of great interest because they constitute an important class of natural and non-natural products, many of which exhibit useful biological activities and clinical applications [28, 29]. Substituted pyrimidines occur widely in living organisms and were some of the first compounds studied by the organic chemists [30] and are an important class of heterocyclics and include many substances of both biological and chemical interest. They are part of a large number of highly significant biomolecules such as the essential amino acid histidine and related compounds, biotin, and the imidazole alkaloids. In entry (5), the 4-chloro-2-ethyl-1H-imidazole-5-carbaldehyde is a fused hetero aromatic system which has been the subject of substantial attention by synthetic and medicinal chemists because of the role of this hetero aromatic ring in many biological systems.

## III. EXPERIMENTAL SECTION

Solvents and reagents were obtained from commercial source and used without purification. The progress of the reaction was monitored by Thin Layer Chromatography (TLC) using aluminium sheets coated with silica gel F $_{254}$ (Merck) with yielding under short wavelength UV lamp detection. The IR spectra (ϑmax, cm$^{-1}$) were recorded in solid state KBr dispersion using Perkin Elmer FT-IR spectrometer. The $^1$H-NMR and $^{13}$C-NMR spectra were recorded on Bruker-Avance 300 & 500 MHz spectrometers using DMSO-d$_6$ or CDCl$_3$ as the solvent and TMS as an internal standard. The chemical shifts were reported in δ/ ppm relative to TMS. The mass spectra were recorded on API 2000 Perkin Elmer PE-Sciex mass spectrometer. Melting points were determined on polman melting point apparatus (Model No MP96) by open capillary method and are uncorrected.

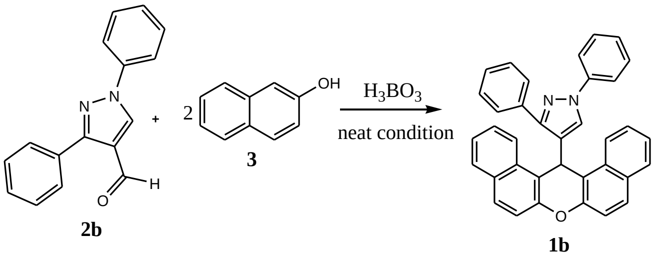
### Synthesis of 2-[4-(14H-dibenzoxanthene-14-yl) phenyl] pyridine (compound-1a)



**2a** **3** **1a**

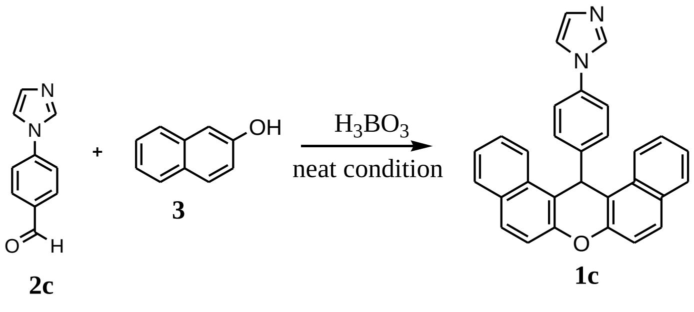
To a solution of 4-(2-pyridyl) benzaldehyde (10g, 54.6mmol), 2-naphthol (15.737g, 109.15mmol) and boric acid (0.467g, 7.553mmol) was added at room temperature in the ratio's of 1:2:0.2, the reaction mass was stirred at 120$^0$ C for 0.5hr, after completion of the reaction the reaction mixture was cooled to 25$^0$ C, and water was added, further the reaction mass was stirred for 10 min. After evaporation of the solvent, the obtained solid was collected by filtration and purified by recrystallization from ethanol to give white coloured solid product 9.2052g (90%) mp: 302-304°C. Mass for $C_{32}H_{21}NO$ (M + H) $^+$Calcd: 436.5152, found: 436. $^1$H-NMR (500 MHz, DMSO-d$_6$) δ (ppm): 8.562 (d, J = 8 Hz, 1H, Ar), 7.872 (d, J = 7.5 Hz, 2H, Ar), 7.638 (d, J = 8 Hz, 2H, Ar), 7.603 (d, J = 7.5 Hz, 2H, Ar), 7.543 (d, J = 7.5Hz, 1H, Ar), 7.476 (dd, J = 8Hz, 1H, Ar), 7.295 (d, J = 8 Hz, 2H, Ar), 7.187 (dd, J = 8Hz, 2H, Ar), 6.982 (dd, J = 7.5Hz, 1H, Ar), 6.843 (d, J = 8Hz, 2H, Ar), 5.341 (s, 1H, -CH-). $^{13}$C-NMR (125 MHz, DMSO) δ (ppm): 154.713, 153.72, 149.301, 142.021, 137.209, 133.812, 133.521, 128.865, 128.572, 128.321, 128.301, 128.121, 124.292, 120.817, 118.912, 115.423, and 45.63. IR (KBr pellet):3020, 2010, 1600, 1580, 1490, 1450, 1150, 1100, 830 Cm$^{-1}$.

### Synthesis of 1, 3-diphenyl-4-(14H-dibenzoxanthene-14-yl) pyrazole (Compound-1b)



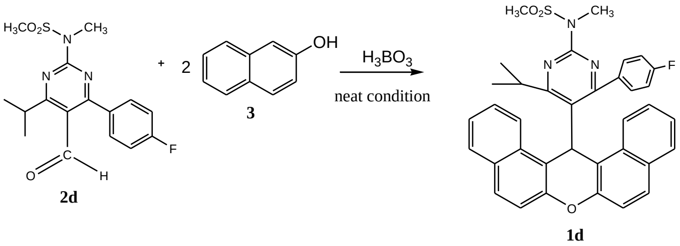
To a solution of 3-aryl-1-phenyl-1H-pyrazole-4-carbaldehyde (10g, 40.3mmol) 2-naphthol (15.737g, 109.15mmol) and boric acid (0.467g, 7.553mmol) was added at room temperature in the ratio's of 1:2:0.2, the reaction mass was stirred at $120^0$ C for 1.5hr, after completion of the reaction the reaction mixture was cooled to $25^0$ C, and water was added, further the reaction mass was stirred for 10 min. After evaporation of the solvent ,the obtained solid was collected by filtration and purified by recrystallization from ethanol to give white coloured solid product 8.2304g (80%)**,**mp: 316-318 °C. Mass for $C_{36}H_{24}N_2O$ (M + H) $^+$Calcd: 501.5885, found: 501. $^1$H-NMR (500 MHz, DMSO-$d_6$) δ (ppm): 7.653 (s, 1H, -C=$\underline{CH}$-N-), 7.637 (d, J = 7.5Hz, 2H, Ar), 7.612 (d, J = 7.5 Hz, 2H, Ar), 7.482 (d, J = 8 Hz, 2H, Ar), 7.321 (dd, J = 8 Hz, 2H, Ar), 7.301 (m, 5H, Ar), 7.291 (dd, J = 7.5Hz, 2H, Ar), 7.221 (dd, J = 8Hz, 1H, Ar), 7.181 (dd, J = 8 Hz, 2H, Ar), 6.843 (dd, J = 8Hz, 2H, Ar),6.463 (d, J = 8 Hz, 2H, Ar), 5.342 (s, 1H, -$\underline{CH}$-), $^{13}$C-NMR (125 MHz, DMSO-$d_6$) δ (ppm): 153.786, 139.741, 133.525, 129.312, 129.428, 128.872, 128.812, 128.302, 128.301, 127.576, 126.312, 123.289, 123.171, 122.542, 120.271, 118.918, 117.212, 115.413, 44.023. IR (KBr pellet):3030, 2100, 1602, 1580, 1500, 1450, 1140, 1110, 750, 700 Cm$^{-1}$.

### Synthesis of 1-[4-(14H-dibenzoxanthene-14-yl) phenyl] imidazole (Compound-1c)



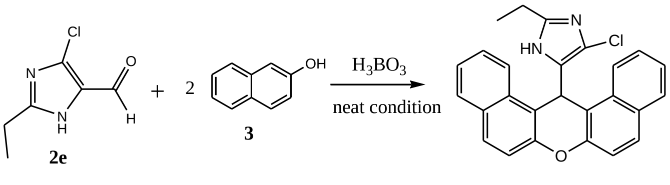
To a solution of 4-(1H-imidazol-1-yl) benzaldehyde (10g, 58.1mmol), 2-naphthol (15.737g, 109.15mmol) and boric acid (0.467g, 7.553mmol) was added at room temperature in the ratio's of 1:2:0.2, the reaction mass was stirred at $120^0$ C for 1hr, after completion of the reaction the reaction mixture was cooled to $25^0$ C, and water was added, further the reaction mass was stirred for 10 min. After evaporation of the solvent, the obtained solid was collected by filtration and purified by recrystallization from ethanol to give white coloured solid product 8.6938g (85%) mp: 296-298 °C. Mass for $C_{30}H_{20}N_2O$ (M + H) $^+$Calcd: 425.4926, found: 425. $^1$H-NMR (500 MHz, DMSO-$d_6$) δ (ppm): 8.031 (s, 1H, -N=$\underline{CH}$-N-), 7.631 (d, J = 8 Hz, 2H, Ar), 7.613 (d, J = 7.5 Hz, 2H, Ar), 7.468 (d, j = 7.5Hz, 2H, Ar), 7.465 (d, J = 6.5Hz, 1H, -N-CH=$\underline{CH}$-N-), 7.295 (dd, J = 7.5 Hz, 2H, Ar), 7.272 (d, J = 6.5 Hz, 1H, -N-$\underline{CH}$=CH-N-), 7.189 (dd, J = 8Hz, 2H, Ar), 7.112 (m, 4H, Ar), 6.843 (d, J= 8Hz, 2H, Ar), 5.346 (s, 1H, -CH-). $^{13}$C-NMR (125 MHz, DMSO-$d_6$) δ (ppm): 183.35, 153.787, 142.95, 135.786, 134.912, 133.512, 130.213, 129.223, 128.801, 128.323, 126.346, 123.247, 122.902, 118.912, 117.721, 115.401, 45.72.IR (KBr pellet):3025, 2010, 2090, 1610, 1585, 1490, 1460, 1150, 1100, 834 Cm$^{-1}$.

### Synthesis of N-[4-(4-fluorophenyl)-6-isopropyl-5-(14H-dibenzoxanthene-14-yl) pyrimidin-2-yl]-N-methyl-methane sulfonamide (Compound---1d)



To a solution of N-[4-(4-fluorophenyl)-5-formyl-6-isopropyl-pyrimidin-2-yl]-N-methyl-methane sulphonamide (10g, 28.5mmol), 2-naphthol (15.737g, 109.15mmol) and Boric acid (0.467g, 7.553mmol) was added at room temperature. The reaction mass was stirred at $120^0$C for 1hr, until the reaction was completed, after completion of the reaction the reaction mixture was cooled to $25^0$C and water was added and the reaction mixture was stirred for 10min. after evaporation of the solvent , the product was recrystallized from ethanol to give white coloured solid product 9.2052g (90%) mp: 322-324°C. Mass for $C_{36}H_{30}N_3O_3SF$ (M + H)$^+$Calcd: 604.7017, found:604. $^1$H-NMR (500 MHz, DMSO-$d_6$) δ (ppm): 7.632 (d, J = 8Hz, 2H, Ar), 7.607 (d, J = 7.5 Hz, 2H, Ar), 7.468 (d, J = 7.5 Hz, 2H, Ar), 7.465 (d, J = 7.5 Hz, 2H, Ar), 7.298 (dd, J = 8Hz, 2H, Ar), 7.189 (dd, J = 7.5Hz, 2H, Ar), 7.039 (d, J = 8 Hz, 2H, Ar), 6.841 (d, J = 7.5Hz, 2H, Ar), 5.347 (s, 1H, -CH-), 3.123 (m, 1H, -$\underline{CH}(CH_3)_2$), 2.842 (s, 3H, $\underline{CH_3}SO_2$), 2.473 (s, 3H, $\underline{CH_3}$-N-), 1.293 (d, J = 8Hz, 6H, - $CH(\underline{CH_3})_2$).$^{13}$C-NMR (125 MHz, DMSO-$d_6$) δ (ppm): 166.521, 162.912, 153.751, 133.525, 129.182, 128.812, 128.316, 128.314, 126.342, 126.327, 123.287, 122.501, 118.916, 116.827, 116.104, 115.42, 39.101, 33.274, 31.123, 23.745, 23.712. IR (KBr pellet):3040, 2960, 2890, 2872, 2085, 1610, 1590, 1485, 1460, 1450, 1385, 1370, 1365, 1200, 1140, 1100, 830 $Cm^{-1}$.

### Synthesis of 4-chloro-2-ethyl-5-(14H-dibenzoxanthene-14-yl)-1H-imidizole (Compound-1e)



2e 3 1e

To a solution of 2H-ethyl-4-chloro-1H-imidizole-5-carboldehyde (10g, 63mmol), 2-naphthol (15.737g, 109.15mmol) and boric acid (0.467g, 7.553mmol) was added at room temperature in the ratios of 1:2:0.2,the reaction mass was stirred at $120^0$ C for 1.5hr, after completion of the reaction the reaction mixture was cooled to $25^0$ C, and water was added, further the reaction mass was stirred for 10 min. After evaporation of the solvent ,the obtained solid was collected by filtration and purified by recrystallization from ethanol to give white colored solid product 8.8983g (87%), mp: 308-310 °C. Mass for $C_{26}H_{19}N_2OCl$ $(M + H)^+$Calcd: 411.8940, found:411. $^1$H-NMR (500 MHz, DMSO-$d_6$) δ (ppm): 13.4 ( s, 1H, -NH-) 7.632 (d, J = 7.5 Hz, 2H, Ar), 7.602 (d, J = 7.5 Hz, 2H, Ar), 7.465 (d, J = 8 Hz, 2H, Ar), 7.298 (dd, J = 8Hz, 2H, Ar), 7.187 (dd, J = 8Hz, 2H, Ar), 6.845 (d, J = 7.5 Hz, 2H, Ar), 5.341 (s, 1H, -CH-), 2.592 (q, J = 6.5Hz, 2H, $CH_3$-$CH_2$), 1.231 (t, J = 7 Hz, 3H, $CH_2CH_3$). $^{13}$C-NMR (125 MHz, DMSO-$d_6$) δ(ppm): 153.742, 148.19, 135.762, 133.511, 128.823, 128.323, 128.312, 126.312, 123.298, 122.502, 118.921, 115.456, 28.902, 26.743, 14.312.IR (KBr pellet):3450, 3010, 2962, 2910, 2870, 2852, 1990, 1610, 1570, 1500, 1465, 1451, 1373, 1300, 1140 $Cm^{-1}$.

## IV. CONCLUSION

In conclusion, aryl heterocyclic aldehydes **2a-e** were used as starting materials for the synthesis of new biologically active heterocyclic 14-substituted-14H-dibenzoxanthene derivatives **1a-e** by using 2-naphthol **3** and boric acid .The newly synthesized compounds are characterized by spectral data. The outcome of this experiment has resulted in excellent yield generation within a short span of time. This method has inherent advantages like simplicity of performance, solvent free condition, low cost and eco- friendly, and the catalyst is readily available and inexpensive, conveniently be handled and easily removed from the reaction mixture. The use of heterocyclic aryl aldehydes in this process has enhanced the biological activity of desired product, 14-substituted-14H-dibenzoxanthene derivatives.

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