



## SYNTHESIS OF BENZOXANTHENES AND 1-AMIDOALKYL-2-NAPHTHOLS USING SOLID SUPPORTED PbCl<sub>2</sub> UNDER SOLVENT FREE CONDITION

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### Abstract

Silica supported anhydrous PbCl<sub>2</sub> was prepared using simple method. The prepared catalyst was found to be thermally stable up to 220<sup>o</sup>C. It was found to be heterogeneous and recyclable catalyst for the synthesis of Benzoxanthenes and 1-amidoalkyl-2-naphthols under solvent free condition. In both cases, good to excellent yields were obtained.

**Key-words:** Silica supported; thermally stable; recyclable; solvent free condition; excellent yields.

### Introduction

Multicomponent reactions are useful to build complex organic molecules in single step from easily available starting materials with minimum requirements in terms of reaction time, labor and overall cost. The reduction in number of steps involved, minimum waste production, overall flexibility makes Multicomponent reactions to satisfy the principles of Green Chemistry.<sup>i</sup> In recent years, solid supported catalysts have attracted attention of researchers due to their several advantages.<sup>ii, iii</sup> An attachment of catalyst on suitable solid support provides many advantages of homogeneous catalytic system such as high reactivity, overall selectivity along with those of heterogeneous catalysts like increase in number of active sites, easy handling, easy separation, thermal stability, and reusability. So using solid supported catalysts, organic transformations can be carried out in economical and environment friendly way.<sup>iv-ix</sup>

Xanthenes and Benzoxanthenes have attracted considerable interest because of their biological activities such as anti-inflammatory, antibacterial, and antiviral activities.<sup>x-xiii</sup> Different catalytic systems such as ionic liquid,<sup>xiv, xv</sup> hetero polyacid,<sup>xvi</sup> Amberlyst-15,<sup>xvii</sup> Montmorillonite K10,<sup>xviii</sup> sulfuric acid,<sup>xix</sup> I<sub>2</sub>,<sup>xx</sup> sulfamic acid<sup>xxi</sup> have been used to synthesize Xanthenes and Benzoxanthenes.

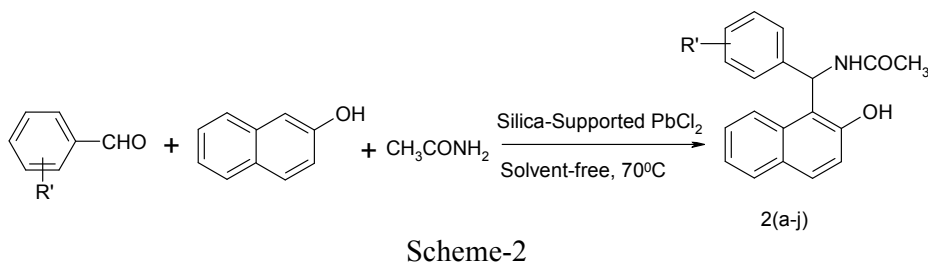
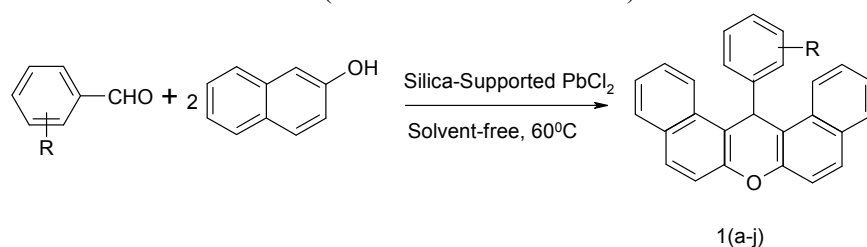
Similarly, Amidoalkyl naphthols are important organic compounds from biological activity point of view.<sup>xxii</sup> Amidoalkyl naphthols can be hydrolyzed to 1-aminomethyl-2-naphthol derivatives which exhibits important biological activities such as hypotensive and bradycardiac effects.<sup>xxiii, xxiv</sup> These compounds have been synthesized under various reactions

such as  $K_5CoW_{12}O_{40} \cdot 3H_2O$ ,<sup>xxv</sup>  $Yb(OTf)_3$  in ionic liquid,<sup>xxvi</sup>  $TMSCl/NaI$ ,<sup>xxvii</sup>  $Al_2O_3 \cdot HClO_4$ ,<sup>xxviii</sup>  $Sr(OTf)_2$ ,<sup>xxix</sup>  $H_2NSO_3H$ ,<sup>xxx</sup>

All of these protocols for the synthesis of xanthenes and 1-amidoalkyl-2-naphthols give good to excellent yields of desired compounds. But due to one or other disadvantages such as long reaction time, use of costly materials and techniques, need of harsh reaction conditions, difficult work-up procedures, there is still need to develop mild and efficient methodology which also comply with Green Chemistry principles.

The literature survey shows that lead salts and compounds exhibits catalytic activity which is utilized in synthesis of various biologically active and hence commercially important organic compounds.<sup>xxxi, xxxii, xxxiii</sup> Moreover, Lead compounds are easy to handle due to their stability towards moisture.<sup>xxxiv</sup>

The present work deals with synthesis of Benzoxanthenes and 1-amidoalkyl-2-naphthols using Multicomponent reaction pathway in presence of silica supported anhydrous  $PbCl_2$  as catalyst under solvent free condition (Scheme 1 and Scheme 2)



## Results and Discussion

### Selection of catalyst

When synthesis of both Benzoxanthenes and 1-amidoalkyl-2-naphthols were performed with  $SiO_2$  as catalyst, no reaction was observed. Even anhydrous  $PbCl_2$  failed to give any results. So  $SiO_2$  supported  $PbCl_2$  was tried for the synthesis of Benzoxanthenes and 1-amidoalkyl-2-naphthols.

### Synthesis of Benzoxanthenes

A reaction between benzaldehyde (1 mmol, 0.101ml) and  $\beta$ -naphthol (2 mmol, 0.288 g) to form 14-phenyl-14H-dibenzo[a,j]xanthene was selected as a model reaction for the Scheme 1. Optimum reaction conditions were established for selected model reaction (Table 1).

Table 1. Optimization of reaction conditions for the synthesis of 14-phenyl-14H-benzo[a,j]xanthene.

Entry	Catalyst	Amount (gm)	Temperature ( $^{\circ}C$ )	Solvent	Time (Min.)	Yield <sup>a</sup> (%)
1	$SiO_2$ - $PbCl_2$	0.02	30	Solvent free	90	30
2	$SiO_2$ - $PbCl_2$	0.03	40	Solvent free	85	40

3	SiO <sub>2</sub> - PbCl <sub>2</sub>	0.04	50	Solvent free	55	60
4	SiO <sub>2</sub> - PbCl <sub>2</sub>	<b>0.06</b>	<b>60</b>	<b>Solvent free</b>	<b>20</b>	<b>92</b>
5	SiO <sub>2</sub> - PbCl <sub>2</sub>	0.08	70	Solvent free	20	92
6	SiO <sub>2</sub> - PbCl <sub>2</sub>	0.06	60	CH <sub>2</sub> Cl <sub>2</sub>	>180	35
7	SiO <sub>2</sub> - PbCl <sub>2</sub>	0.06	60	CHCl <sub>3</sub>	>180	40
8	SiO <sub>2</sub> - PbCl <sub>2</sub>	0.06	60	CH <sub>3</sub> CN	>180	55
9	SiO <sub>2</sub> - PbCl <sub>2</sub>	0.06	60	C <sub>2</sub> H <sub>5</sub> OH	>180	45

<sup>a</sup>Isolated Yields

Thus, the reactions for the synthesis of Benzoxanthenes were to be performed in presence of 0.06 gm of SiO<sub>2</sub> supported PbCl<sub>2</sub> as catalyst at 60<sup>o</sup>C. By using optimized reaction conditions, the general applicability of the Scheme 1 was examined by using different aromatic aldehydes (Table 2)

Table 2 Synthesis of Benzoxanthenes in presence of SiO<sub>2</sub> supported PbCl<sub>2</sub> as catalyst at 60<sup>o</sup>C under solvent free condition

Entry	R <sup>a</sup>	Product <sup>b</sup>	Time (Min.)	Yield <sup>c</sup> (%)	M.P.( <sup>o</sup> C) (Lit. Value)
1	C <sub>6</sub> H <sub>5</sub> -	1a	20	92	181-183(183) <sup>xxxvi</sup>
2	4-OCH <sub>3</sub>	1b	20	93	204-205(205-206) <sup>xxxvii</sup>
3	2-OCH <sub>3</sub>	1c	25	91	256-258(258) <sup>xxi</sup>
4	4-Cl	1d	18	90	287-288 (287) <sup>xxxviii</sup>
5	2-Cl	1e	22	88	214-216 (215) <sup>xxi</sup>
6	4-CH <sub>3</sub>	1f	15	93	227-229 (228) <sup>xxxvi</sup>
7	4-NO <sub>2</sub>	1g	15	92	313-314 (312) <sup>xxi</sup>
8	3-NO <sub>2</sub>	1h	18	91	212-214 (213) <sup>xxi</sup>
9	2-NO <sub>2</sub>	1i	20	89	292-293 (293) <sup>xxi</sup>
10	4-OH	1j	15	91	110-111 (111) <sup>xxxix</sup>

<sup>a</sup>An aromatic aldehyde (1 mmol) was reacted with β-naphthol (2 mmol) in presence of 0.06 gm of SiO<sub>2</sub> supported PbCl<sub>2</sub> as catalyst at 60<sup>o</sup>C under solvent free condition.

<sup>b</sup>All the products were confirmed by comparing melting point and spectral data with data available in Literature.

<sup>c</sup>Isolated Yields

### Synthesis of 1-amidoalkyl-2-naphthols

The efficiency of SiO<sub>2</sub> supported PbCl<sub>2</sub> as catalyst was further tested by using it in synthesis of 1-amidoalkyl-2-naphthols. A model reaction involving benzaldehyde (1mmol, 0.101ml), β-naphthol (1 mmol, 0.144gm) and acetamide (1.5 mmol, 0.885 gm) with silica supported PbCl<sub>2</sub> as catalyst was used and optimum reaction conditions were established (Table 3).

Table 3 Optimization of reaction conditions for the synthesis of N-[(2-hydroxynaphthalen-1-yl)-phenyl-methyl] acetamide

Entry	Catalyst	Amount (gm)	Temperature ( <sup>o</sup> C)	Solvent	Time (Min.)	Yield <sup>a</sup> (%)
1	SiO <sub>2</sub> - PbCl <sub>2</sub>	0.02	45	Solvent free	120	Nil
2	SiO <sub>2</sub> - PbCl <sub>2</sub>	0.04	55	Solvent free	75	30
3	SiO <sub>2</sub> - PbCl <sub>2</sub>	0.06	65	Solvent free	45	65
4	SiO <sub>2</sub> - PbCl <sub>2</sub>	<b>0.08</b>	<b>70</b>	<b>Solvent free</b>	<b>15</b>	<b>93</b>

5	SiO <sub>2</sub> - PbCl <sub>2</sub>	0.1	85	Solvent free	15	93
6	SiO <sub>2</sub> - PbCl <sub>2</sub>	0.08	70	CH <sub>2</sub> Cl <sub>2</sub>	>180	25
7	SiO <sub>2</sub> - PbCl <sub>2</sub>	0.08	70	CHCl <sub>3</sub>	>180	45
8	SiO <sub>2</sub> - PbCl <sub>2</sub>	0.08	70	CH <sub>3</sub> CN	>180	50
9	SiO <sub>2</sub> - PbCl <sub>2</sub>	0.08	70	C <sub>2</sub> H <sub>5</sub> OH	>180	40

<sup>a</sup>Isolated Yields

The required amount of silica supported PbCl<sub>2</sub> to be used as catalyst in reaction medium was found to be 0.08 gm at 70°C under solvent free condition as per data presented in Table 4. The scope of Scheme 2 was checked by performing it using various aromatic aldehydes under the set experimental conditions (Table 4).

Table 4 Synthesis of 1-amidoalkyl-2-naphthols using catalytic amount of silica supported PbCl<sub>2</sub> at 70°C under solvent free condition.

Entry	R <sup>a</sup>	Product <sup>b</sup>	Time (Min.)	Yield <sup>c</sup> (%)	M.P.(°C) Lit. Value
1	C <sub>6</sub> H <sub>5</sub> -	2a	15	93	240-242 (241-243) <sup>xi</sup>
2	4-CH <sub>3</sub>	2b	10	94	218-219 (218-220) <sup>xliv</sup>
3	2-CH <sub>3</sub>	2c	15	90	201-203 (200-202) <sup>xliii</sup>
4	4-OCH <sub>3</sub>	2d	10	93	185-187 (184-186) <sup>xi</sup>
5	4-Cl	2e	20	91	237-239 (237-238) <sup>xliv</sup>
6	3-Cl	2f	22	90	236-237 (237-238) <sup>xliii</sup>
7	4-NO <sub>2</sub>	2g	12	92	249-251 (248-250) <sup>xliv</sup>
8	3-NO <sub>2</sub>	2h	15	89	254-255 (255-256) <sup>xxix</sup>
9	2-NO <sub>2</sub>	2i	25	88	181-183 (180-182) <sup>xi</sup>
10	4-OH	2j	18	90	207-208 (206-208) <sup>xli</sup>

<sup>a</sup>An aromatic aldehyde (1 mmol) was reacted with β-naphthol (1 mmol) and acetamide (1.5 mmol) in presence of 0.08 gm of SiO<sub>2</sub> supported PbCl<sub>2</sub> as catalyst at 70°C under solvent free condition.

<sup>b</sup>All the products were confirmed by comparing melting point and spectral data with Literature.

<sup>c</sup>Isolated Yields

In both Scheme 1 and Scheme 2, it was observed that all aromatic aldehydes bearing either electron donating or withdrawing substituent reacted smoothly under the established reaction conditions giving good to excellent yields. The yield and reaction time were found to be almost independent of nature of substitution pattern in aromatic aldehydes. The efficiency of SiO<sub>2</sub> supported PbCl<sub>2</sub> as a catalyst must be a reason for these observations. In case of ortho substitution, decrease in yield was observed (Table 2, Entries 3, 5, 9 and Table 4, Entries 3, 9) which must be due to steric crowding near to reaction centre.

#### Study of reusability of SiO<sub>2</sub> supported PbCl<sub>2</sub>

The catalyst being heterogeneous in nature was easily separable from reaction mixture. It was carefully separated from reaction mixture and washed thoroughly with Ethyl acetate. After air drying, it was used for three more sets of same reaction. Its catalytic activity was found to be almost sustainable in every run (Table 5)

Table 5 Study of recyclability of SiO<sub>2</sub> supported PbCl<sub>2</sub>

Entry	Run	Synthesis of 14-phenyl-14H-dibenzo[a,j]xanthenes (1a)		Synthesis of N-[(2-hydroxynaphthalen-1-yl)-phenyl-methyl] acetamide (2a)	
		Time (Min.)	Yield <sup>b</sup> (%)	Time (Min.)	Yield <sup>b</sup> (%)
1	1	20	92	15	93
2	2	15	89	20	90
3	3	30	85	28	86
4	4	38	83	35	82

<sup>bb</sup> Isolated Yields**Checking of sample of product for presence of Lead content**

The product was dissolved in Dichloromethane and it was analyzed with using Atomic Absorption Spectrometer (AAS, Model-Shimadzu, AA-7000 series) at wavelength-217 nm with air-acetylene flame. This experiment indicated **NO** Lead concentration in sample of product.

**Experimental**

The required chemicals and solvents were obtained from Merck, Sigma-Aldrich and used without any further purification. The values of melting points obtained using open capillary method were used without correction. The crude products were purified using column chromatography (silica gel of 60-120 mesh size and mixture of pet ether and ethyl acetate in suitable proportions as system). All <sup>1</sup>H NMR were recorded on 300 MHz Avance FT-NMR spectrometer. Tetramethylsilane (TMS,  $\delta=0$ ) was used as internal standard and all other chemical shifts were expressed with respect to  $\delta$ -value of it. IR spectra were recorded on Perkin-Elmer FTIR spectrometer as KBr pellets. The selected Benzoxanthenes and 1-amidoalkyl-2-naphthols were further analyzed by IR and <sup>1</sup>H NMR.

**General procedure for synthesis of Benzoxanthenes (Scheme 1)**

A reaction mixture of an aromatic aldehyde (1 mmol),  $\beta$ -naphthol (2 mmol, 0.288 gm) and silica supported PbCl<sub>2</sub> (0.06 gm) as catalyst was heated with stirring at 60<sup>0</sup>C under solvent free condition. After the completion of reaction as indicated by TLC, the crude product was extracted in Ethyl acetate (3x10 ml) and catalyst was carefully separated. All the collected extracts were dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> to get crude product. The obtained crude product was purified using Column Chromatography (Using Silica gel of 60-120 mesh size and mixture of Pet ether and Ethyl acetate in suitable proportions as system). The separated catalyst was washed with Ethyl acetate and used again.

**Spectral data for selected Benzoxanthenes**

Entry-1 (1a) 14-phenyl-14H-dibenzo[a,j]xanthenes

<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)  $\delta$ = 8.41(2H,d, J=8.6Hz), 7.85(2H,d,J=8.0Hz), 7.11(2H,d,J=8.5Hz), 7.56 (2H,t,J=7.5Hz), 7.52 (2H, d, J=7.8 Hz), 7.47 (2H,d,J=8.10Hz), 7.44(2H, t, J=7.6Hz), 7.18 (2H, t, J=7.6Hz),7.05(1H, t, J=7.6 Hz), 6.48(1H, s).

IR (KBr): 3077, 3022, 2889, 1625, 1594, 1515, 1491, 1460, 1405, 1255, 1080, 1025, 966, 830, 745, 702 cm<sup>-1</sup>

**General procedure for synthesis of 1-amidoalkyl-2-naphthols (Scheme 2)**

The reaction mixture prepared by mixing an aromatic aldehyde (1mmol),  $\beta$ -naphthol (1 mmol, 0.144 gm) and acetamide (1.5 mmol, 0.885 gm) with silica supported PbCl<sub>2</sub> (0.08 gm) as catalyst was heated at 70<sup>0</sup>C under solvent free condition for appropriate time. When

reaction was completed as indicated by TLC, the reaction mixture was dissolved in Ethyl acetate (3x10 ml) and insoluble catalyst was carefully separated. All the extracts were dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> which was followed by purification of crude product using Column Chromatography (Using Silica gel of 60-120 mesh size and mixture of Pet ether and Ethyl acetate in suitable proportions as system). The separated catalyst was washed repeatedly with Ethyl acetate and after its air drying, it was re-used.

#### Spectral data for selected 1-amidoalkyl-2-naphthols

Entry-1 (2a) N-[(2-hydroxynaphthalen-1-yl)-phenyl-methyl] acetamide

<sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>) δ= 1.95 (s, 3 H), 7.14-7.19 (m, 4 H), 7.23-7.29 (m, 4 H), 7.30-7.35 (m, 1 H), 7.72-7.80 (m, 3 H), 8.46 (d, *J*= 8.0 Hz, 1 H), 9.91(s, 1 H).

IR (KBr) cm<sup>-1</sup>: 3402, 3250, 1640, 1518, 1440, 1336, 1280, 810, 746.

The formation of Benzoxanthenes and 1-amidoalkyl-2-naphthols was confirmed by comparing their melting point data with data reported in literature.

#### Conclusion

By using simple method, useful SiO<sub>2</sub> supported PbCl<sub>2</sub> catalyst was successfully prepared. It was easy to store and handle due to its stability towards moisture. The characterization using TGA indicated its stability up to 220 °C giving scope to use it at elevated temperature. Its analysis using standard B.E.T. technique proved diffusion of PbCl<sub>2</sub> in silica support forming –Si-O-PbCl linkages. These linkages must be responsible for activation of Pb as Lewis acid leading to increased catalytic activity. It was found to be an efficient catalyst for synthesis of Benzoxanthenes and 1-amidoalkyl-2-naphthols. Good to excellent yields in short reaction time were obtained for both Benzoxanthenes and 1-amidoalkyl-2-naphthols. Moreover, it was found to be recyclable retaining its catalytic activity for at least three additional turns. This factor made this protocol cost effective.

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