# FeCl<sub>3</sub>-Catalyzed Condensation of 2-Naphthol and Aldehydes under Solvent-Free Reaction Conditions: An Efficient and Green Alternative for the Synthesis of 14-Aryl(Alkyl)-14-*H*-dibenzo[*a*, *j*]xanthenes

Wang, Bo<sup>a</sup>(王波) LI, Pinhua<sup>\*,a</sup>(李品华) Zhang, Yicheng<sup>a</sup>(张义成) Wang, Lei<sup>\*,a,b</sup>(王磊)

<sup>a</sup> Department of Chemistry, Huaibei Normal University, Huaibei, Anhui 235000, China <sup>b</sup> State Key Laboratory of Organometallic Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, Shanghai 200032, China

An iron-catalyzed condensation of 2-naphthol with aldehydes has been described. In the presence of a catalytic amount of iron(III) chloride (5 mol%), the condensation reaction of 2-naphthol and aldehydes underwent smoothly to generate the corresponding products in good to excellent yields under base-, acid-, ligand-, additive-, and solvent-free reaction conditions.

Keywords 2-naphthol, aldehydes, iron(III) chloride, solvent-free, benzoxanthenes

# Introduction

Xanthenes and benzoxanthenes are important substructures found in numerous natural sources.<sup>1</sup> They possess various biological activities and pharmacological properties such as anti-bacterial,<sup>2</sup> anti-inflammatory,3 and antiviral properties.4 These compounds have received an extensive attention because some of them have also been used as leuco-dyes in laser technology,<sup>5</sup> as pH-sensitive fluorescent materials for the visualization of biomolecular assemblies,<sup>6</sup> and antagonists of the paralyzing action of zoxazalamine.<sup>7</sup> Therefore, the synthesis of 14-aryl- and 14-alkyl-14-H-dibenzo[a,j]xanthenes has attracted considerable and continuing interest in organic synthesis and pharmaceutical science. The recommended synthetic routes, including the reaction of aryloxymagnesium halides with triethylorthoformate,<sup>8</sup> cyclization of polycyclic aryltriflate esters<sup>9</sup> and cyclocondensation between 2-hydroxy aromatic aldehydes and 2-tetralone<sup>10</sup> have been reported. In addition, other reactions through the condensation of 2-naphthol with aldehydes in the presence of an effective catalyst have been developed.<sup>11</sup> Nevertheless, these methods have several disadvantages, such as toxic organic solvent, excess of catalysts, and poor yields. Hence, simpler, more convenient and efficient approaches for the preparation of benzoxanthenes are still highly desirable.

In contrast, the catalysts derived from heavy or rare metals have several drawbacks for large-scale applications due to their toxicity and prohibitive price. Iron is one of the most abundant, and consequently one of the most inexpensive and environmentally friendly metals on the earth. Iron salts as effective, alternative and promising transition-metal catalysts have received much attention because of their unique properties.<sup>12</sup> Over the past few years, iron-catalyzed oxidation,<sup>13</sup> hydrogenation,<sup>14</sup> hydrosilylation,<sup>15</sup> rearrangement,<sup>16</sup> Michael addi-tion,<sup>17</sup> and Mannich reaction<sup>18</sup> have been intensively investigated. Recently, iron-catalyzed C-S,<sup>19</sup> C-N,<sup>20</sup>  $C-O^{2T}$  and C-C bond formation reactions (such as Sonogashira, Heck, Negishi, Kumada and Suzuki reactions) have been developed.<sup>22</sup> However, it is still desirable to expand the application scope of iron salts in organic transformations due to their unique properties and significant advantages for both the academic and the industrial community. Recently, our group has reported the wide use of iron as an efficient catalyst in organic synthesis.<sup>23</sup> As a result of our ongoing interest in iron-catalyzed organic reactions, we wish to report the preparation of benzoxanthenes by the FeCl3-catalyzed (5 mol%) condensation reaction of 2-naphthol with aldehydes under base-, acid-, ligand-, additive-, and solvent-free reaction conditions (Scheme 1).

# **Results and discussion**

As a model reaction of this protocol, the reaction of



 <sup>\*</sup> E-mail: leiwang@chnu.edu.cn; Tel.: 0086-0561-3802069; Fax: 0086-561-03090518 Received January 15, 2010; revised June 20, 2010; accepted July 10, 2010.
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Scheme 1

#### **Table 1** Optimization of the reaction conditions<sup>a</sup>



2-naphthol with benzaldehyde was chosen as model substrates for the optimization of the reaction conditions. The results are summarized in Table 1. It can be seen from Table 1, this reaction was strongly influenced by solvents. No desired product was isolated when the reaction was carried out in DMF, or CH<sub>3</sub>CN (Table 1, Entries 8 and 9). Meanwhile, only trace amount of the products were obtained when the reaction was carried out in THF, or NMP (Table 1, Entries 6 and 7). When the reaction was performed in 1,2-dibromoethane, 1,2-dichloroethane, CHCl<sub>3</sub>, toluene and 1,4-dioxane in the presence of a catalytic amount of  $FeCl_3$  (10 mol%), 56%, 45%, 42%, 16%, and 10% yields of the corresponding products were obtained, respectively (Table 1, Entries 1-5). To our delight, 91% of product was isolated when the reaction was carried out in the absence of any solvent at 100 °C for 10 h (Table 1, Entry 10). Among a variety of catalyst species tested, FeCl<sub>3</sub> was found to be the most effective one (Table 1, Entries 10-14), which is better than FeBr<sub>3</sub>, and much better than FeCl<sub>2</sub>, FeBr<sub>2</sub>, and ZnCl<sub>2</sub> (Table 1, Entries 11-14). With respect to the catalyst loading, when 1-3 mol% of FeCl<sub>3</sub> was used, the reaction did not go to completion, but a higher loading (5-10 mol%) of the catalyst gave very good results (Table 1, Entries 1, and 15-17). However, with an increased loading of the catalyst up to 15 mol%, there was no increase in the isolated yield of the product (Table 1, Entry 18). It is important to note that under solvent-free reaction conditions and at a relative low temperature (80, 60 °C), 65% and 32% yields of the expected products were isolated, respectively (Table 1, Entries 19 and 20). Therefore, 5 mol% of FeCl<sub>3</sub> was considered as an efficient catalyst in the absence of any solvent or additive at 100 °C for the reaction.

Under the optimized reaction conditions, a series of benzaldehydes were examined to probe the generality of this reaction. The results are shown in Table 2. In the presence of FeCl<sub>3</sub> (5 mol%) and in the absence of any solvent at 100 °C, without any acid, base, ligand or additive, reactions of a variety of substituted benzaldehydes containing electron-withdrawing or electron-donating groups on the benzene rings with 2-naphthol underwent smoothly, and the corresponding products were obtained in good to excellent yields (Table 2, Entries 1—14). When hindered benzaldehydes were



Entry	Catalyst/mol%	Solvent/Temp. (°C)	Yield <sup>b</sup> /%
1	FeCl <sub>3</sub> (10)	1,2-Dibromoethane/100	56
2	FeCl <sub>3</sub> (10)	1,2-Dichloroethane/80	45
3	FeCl <sub>3</sub> (10)	CHCl <sub>3</sub> /62	42
4	FeCl <sub>3</sub> (10)	Toluene/100	16
5	FeCl <sub>3</sub> (10)	1,4-Dioxane/100	10
6	FeCl <sub>3</sub> (10)	THF/68	<5
7	FeCl <sub>3</sub> (10)	NMP/100	<5
8	FeCl <sub>3</sub> (10)	DMF/100	NR
9	FeCl <sub>3</sub> (10)	CH <sub>3</sub> CN/80	NR
10	FeCl <sub>3</sub> (10)	Solvent-free/100	91
11	FeCl <sub>2</sub> (10)	Solvent-free/100	<5
12	FeBr <sub>3</sub> (10)	Solvent-free/100	68
13	FeBr <sub>2</sub> (10)	Solvent-free/100	12
14	ZnCl <sub>2</sub> (10)	Solvent-free/100	25
15	$\operatorname{FeCl}_{3}(5)$	Solvent-free/100	91
16	$\operatorname{FeCl}_{3}(3)$	Solvent-free/100	80
17	$\operatorname{FeCl}_{3}(1)$	Solvent-free/100	43
18	FeCl <sub>3</sub> (15)	Solvent-free/100	91
19	$\operatorname{FeCl}_{3}(5)$	Solvent-free/80	65
20	$\operatorname{FeCl}_{3}(5)$	Solvent-free/60	32

<sup>*a*</sup> Reaction conditions: 2-naphthol (1.0 mmol), benzaldehyde (0.5 mmol), solvent (1.0 mL) or without solvent for 10 h. <sup>*b*</sup> Isolated yield.

used as substrates for the reactions, slightly lower reactivity was found (Table 2, Entries 7, 8 and 13). It is important to note that aliphatic aldehydes were also successfully reacted with 2-naphthol to give the desired products in good yields (Table 2, Entries 15 and 16). In addition, we also investigated the reactivity of 2-pyridinecarboxaldehyde, and the reaction provided the corresponding product in good yield (Table 2, Entry 14).

Based on the Lewis acid catalyzed condensation protocol, a tentative mechanism for the formation of benzoxanthenes was proposed (Scheme 2). A carbocation was initially formed and aryl-methanebisnaphthol was then formed in the second step, which then underwent dehydration to give the final product, benzoxanthene.

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#### Table 2 Reactions of aldehydes with 2-naphthol<sup>a</sup>



Entry	Aldehyde	Product	Yield <sup>b</sup> /%
1	СНО	<b>3</b> a	91
2	Н <sub>3</sub> С-СНО	3b	91
3	F-CHO	3c	92
4	СІ—	3d	96
5	Br — CHO	3e	95
6	O <sub>2</sub> N-CHO	3f	97
7	СНО	3g	87
8	CHO NO <sub>2</sub>	3h	81
9	СІ СНО	3i	97
10	Вr CHO	3ј	93
11	МеО	3k	93
12	CI-CHO O <sub>2</sub> N	31	94
13	CI CHO O <sub>2</sub> N	3m	86

			Continued
Entry	Aldehyde	Product	Yield <sup>b</sup> /%
14	СНО	3n	84
15	СНО	30	81
16	СНО	3p	83

<sup>*a*</sup> Reaction conditions: 2-naphthol (1 mmol), aldehyde (0.5 mmol), FeCl<sub>3</sub> (5 mol%), at 100  $^{\circ}$ C for 10 h under solvent- and additive-free reaction conditions. <sup>*b*</sup> Isolated yield.

### Scheme 2



# Conclusion

In summary, we have developed an efficient protocol for the synthesis of 14-aryl(alkyl)-14-*H*-dibenzo-[a,j]xanthenes. This protocol involves using FeCl<sub>3</sub> (5 mol%) as an eco-friendly, inexpensive, and efficient catalyst without any acid, base, ligand or additive under thermal, solvent-free reaction conditions. The reactions of 2-naphthol with both of aliphatic and aromatic aldehydes underwent smoothly to generate the corresponding products in good to excellent yields. Simple, neutral, green and safe experimental conditions are the characteristics of this protocol.

### Experimental

## Physical measurements and materials

All <sup>1</sup>H NMR and <sup>13</sup>C NMR spectra were recorded on

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a 400 MHz Bruker FT-NMR spectrometers. All chemical shifts ( $\delta$ ) are given with reference to tetramethylsilane (TMS) as an internal standard. Products were purified by flash chromatography on 230—400 mesh silica gel.

The chemicals and solvents were purchased from commercial suppliers either from Aldrich, Fluka, USA or Shanghai Chemical Company, China and were used without purification prior to use.

## General procedure for synthesis of benzoxanthenes

A mixture of an aldehyde (0.5 mmol), 2-naphthol (1.0 mmol) and FeCl<sub>3</sub> (4 mg, 5 mol%) was heated at 100 °C for 10 h. The progress of the reaction was monitored by TLC. After completion of the reaction, water (3 mL) was added and the product was extracted with ethyl acetate (3 mL $\times$ 3). The organic layer was dried (MgSO<sub>4</sub>) and evaporated, and the crude product was purified by flash chromatography (ethyl acetate/petroleum ether as eluent) to provide the pure product.

**14-Phenyl-14-***H***-dibenzo[***a,j***]xanthene (3a)<sup>24</sup> <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) \delta: 8.37 (s, 1H), 8.35 (s, 1H), 7.80 (s, 1H), 7.78 (s, 1H), 7.76 (s, 1H), 7.74 (s, 1H), 7.56—7.49 (m, 4H), 7.47 (s, 1H), 7.44 (s, 1H), 7.39— 7.35 (m, 2H), 7.11 (t,** *J***=8.07 Hz, 2H), 6.98—6.93 (m, 1H), 6.45 (s, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) \delta: 148.69, 144.94, 131.41, 131.01, 128.79, 125.74, 128.42, 128.20, 126.73, 126.32, 124.18, 122.64, 117.96, 117.28, 37.97.** 

**14-**(*p*-**Methylphenyl**)-**14**-*H*-**dibenzo**[*a,j*]**xanthene** (**3b**)<sup>11b</sup> <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$ : 8.34 (d, *J*= 8.38 Hz, 2H), 7.77—7.71 (m, 4H), 7.52 (t, *J*=7.03 Hz, 2H), 7.44—7.33 (m, 6 H), 6.90 (d, *J*=8.02 Hz, 2H), 6.39 (s, 1H), 2.07 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$ : 148.60, 142.06, 135.81, 131.39, 131.01, 129.10, 128.71, 128.68, 128.03, 126.67, 124.13, 122.65, 117.92, 117.38, 37.54, 20.81.

**14-**(*p*-Fluorophenyl)-14-*H*-dibenzo[*a*,*j*]xanthene (3c)<sup>11a</sup> <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$ : 8.29 (d, J= 8.61 Hz, 2H), 7.80—7.74 (m, 4H), 7.56—7.52 (m, 2H), 7.45—7.36 (m, 6 H), 6.78 (t, J=8.87 Hz, 2H), 6.42 (s, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$ : 148.64, 140.73, 131.23, 131.01, 129.61, 129.53, 128.93, 128.84, 126.81, 124.27, 122.43, 117.96, 117.02, 115.37, 115.16, 37.13.

**14-(***p***-Chlorophenyl)-14-***H***-dibenzo[***a,j***]xanthene (<b>3d**)<sup>11b</sup> <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$ : 8.29 (d, J= 8.46 Hz, 2H), 7.83—7.77 (m, 4H), 7.58—7.54 (m, 2H), 7.47 (s, 1H), 7.45—7.38 (m, 5H), 7.10—7.07 (m, 2H), 6.44 (s, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$ : 148.66, 143.43, 132.05, 131.22, 131.02, 129.45, 129.05, 128.87, 128.60, 126.87, 124.33, 122.37, 117.97, 116.72, 37.38.

**14-**(*p*-Bromophenyl)-14-*H*-dibenzo[*a,j*]xanthene (**3e**)<sup>11b</sup> <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$ : 8.30 (s, 1H), 8.28 (s, 1H), 7.83 (s, 1H), 7.81 (s, 1H), 7.79 (s, 1H), 7.77 (s, 1H), 7.56 (t, *J*=6.96 Hz, 2H), 7.47 (s, 1H), 7.45 (s, 1H), 7.42 (s, 1H), 7.40 (s, 1H), 7.38 (s, 1H), 7.36 (s, 1H), 7.24 (d, *J*=2.93 Hz, 2H), 6.43 (s, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$ : 148.66, 143.95, 131.55, 131.22,

131.03, 129.83, 129.07, 128.88, 126.88, 124.34, 122.36, 120.18, 117.97, 116.63, 37.41.

**14**-(*p*-Nitrophenyl)-14-*H*-dibenzo[*a,j*]xanthene (**3f**)<sup>11b</sup> <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$ : 8.28 (s, 1H), 8.26 (s, 1H), 8.00—7.96 (m, 2H), 7.85 (s, 1H), 7.83 (s, 2H),7.81 (s, 1H), 7.68—7.64 (m, 2H), 7.61—7.57 (m, 2H), 7.51 (s, 1H), 7.48 (s, 1H), 7.45—7.41 (m, 2H), 6.58 (s, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$ : 151.97, 148.76, 131.06, 131.05, 129.56, 129.03, 128.93, 127.16, 124.55, 123.83, 122.01, 118.03, 115.74, 37.84.

**14-(o-Chlorophenyl)-14-***H*-**dibenzo**[*a,j*]**xanthene** (**3g**)<sup>11b</sup> <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$ : 8.69 (d, *J*= 8.53 Hz, 2H), 7.78—7.73 (m, 4H), 7.58 (t, *J*=7.38 Hz, 2H), 7.45—7.33 (m, 5H), 7.22 (s, 1H), 6.89—6.81 (m, 2H), 6.75 (s, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$ : 148.89, 143.52, 131.75, 131.71, 130.84, 130.09, 129.53, 129.01, 128.58, 127.87, 127.79, 126.86, 124.37, 123.40, 34.58.

**14-(***o***-Nitrophenyl)-14-***H***-dibenzo**[*a*,*j*]**xanthene** (**3h**)<sup>25</sup> <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$ : 8.51 (d, *J*=8.09 Hz, 2H), 7.81—7.78 (m, 5H), 7.61—7.57 (m, 2H), 7.54 (s, 1H), 7.50—7.46 (m, 3H), 7.43—7.39 (m, 2H), 7.22—7.18 (m, 1H), 7.06—7.01 (m, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$ : 149.36, 146.99, 140.83, 134.08, 132.21, 131.68, 130.94, 129.42, 128.68, 127.54, 127.36, 124.87, 124.63, 122.54, 117.98, 117.53, 32.48.

**14-(m-Chlorophenyl)-14-***H*-**dibenzo**[*a,j*]**xanthene** (**3i**)<sup>11b</sup> <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$ : 8.31 (s, 1H), 8.29 (s, 1H), 7.82 (s, 1H), 7.80 (s, 1H), 7.79 (s, 1H), 7.77 (s, 1H), 7.57 (t, *J*=7.42 Hz, 2H), 7.48—7.38 (m, 6H), 7.05 (t, *J*=8.05 Hz, 1H), 6.96—6.93 (m, 1H), 6.43 (s, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$ : 148.74, 146.84, 134.38, 131.22, 131.02, 129.56, 129.12, 128.86, 128.29, 126.92, 126.71, 126.36, 124.34, 122.36, 118.04, 116.53, 37.72.

**14-(***m***-Bromophenyl)-14-***H***-dibenzo[***a,j***]xanthene (<b>3j**)<sup>11a</sup> <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$ : 8.25 (d, J= 8.40 Hz, 2H), 7.78—7.72 (m, 4H), 7.58—7.52 (m, 3H), 7.46—7.42 (m, 3H), 7.38—7.34 (m, 2H), 7.08—7.05 (m, 1H), 6.95 (t, J=7.60 Hz, 1H), 6.36 (s, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$ : 148.66, 147.10, 131.17, 131.09, 130.97, 129.85, 129.59, 129.10, 128.83, 126.88, 126.82, 124.31, 122.69, 122.31, 117.99, 116.45, 37.66.

**14-**(*m*-**Methoxyphenyl**)-**14-***H*-**dibenzo**[*a,j*]**xanthene** (**3k**)<sup>26</sup> <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$ : 8.35 (s, 1H), 8.33 (s, 1H), 7.77 (s, 1H), 7.75 (s, 1H), 7.73 (s, 1H), 7.71 (s, 1H), 7.53 (t, *J*=7.75 Hz, 2H), 7.44 (s, 1H), 7.42 (s, 1H), 7.35 (t, *J*=7.36 Hz, 2H), 7.12 (d, *J*=7.55 Hz, 1H), 7.03—7.00 (m, 2H), 6.47 (dd, *J*=1.90, 1.86 Hz, 1H), 6.40 (s, 1H), 3.56 (s, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$ : 159.54, 148.67, 146.44, 131.40, 130.99, 129.21, 128.78, 128.70, 126.70, 124.16, 122.66, 120.72, 117.93, 117.11, 114.90, 110.88, 54.95, 37.89.

**14-(***p***-Chloro-***m***-nitrophenyl)-14-***H***-dibenzo[***a,j***]xanthene (31)<sup>27</sup> <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) \delta: 8.21 (d,** *J***=8.44 Hz, 2H), 8.01 (d,** *J***=1.92 Hz, 1H), 7.85 (s, 1H), 7.83 (s, 2H),7.81 (s, 1H), 7.61 (t,** *J***=7.42 Hz, 3H), 7.49—7.42 (m, 4H), 7.26 (d,** *J***=8.27 Hz, 1H), 6.25 (s,**  1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ: 148.80, 145.21, 132.68, 131.98, 131.05, 130.88, 129.70, 129.12, 127.32, 125.12, 124.80, 124.65, 121.78, 118.04, 115.37, 37.05.

**14-(2-Chloro-5-nitrophenyl)-14-***H***-dibenzo**[*a*,*j*]-**xanthene** (**3m**)<sup>27</sup> <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$ : 8.57 (d, *J*=8.47 Hz, 2H), 8.22 (d, *J*=2.55 Hz, 1H), 7.84—7.80 (m, 4H), 7.73 (dd, *J*=2.59, 6.26 Hz, 1H), 7.63—7.58 (m, 2H), 7.54 (s, 1H), 7.51 (s, 1H), 7.45—7.40 (m, 3H), 6.84 (s, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$ : 149.13, 147.56, 145.48, 136.31, 131.37, 130.93, 130.59, 129.70, 128.91, 127.17, 126.96, 124.65, 122.71, 122.56, 118.38, 116.57, 34.89.

**14**-(*o*-Pyridyl)-14-*H*-dibenzo[*a,j*]xanthene (3n)<sup>11a</sup> <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$ : 8.67 (d, *J*=8.62 Hz, 2H), 8.50 (d, *J*=4.52 Hz, 1H), 7.78 (s, 2H), 7.76 (s, 2H), 7.55 (t, *J*=7.69 Hz, 2H), 7.45 (d, *J*=8.88 Hz, 2H), 7.37 (t, *J*=7.79 Hz, 2H), 7.28 (t, *J*=7.41 Hz, 1H), 7.12 (d, *J*=7.85 Hz, 1H), 6.68 (t, *J*=5.45 Hz, 1H), 6.73 (s, 1H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$ : 164.46, 148.15, 137.25, 131.84, 130.85, 129.18, 128.34, 126.92, 126.34, 124.36, 123.91, 121.26, 117.84, 115.87, 41.67.

**14-**(*n***-Propyl)-14-***H***-dibenzo**[*a,j*]**xanthene** (**3o**)<sup>11b</sup> <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$ : 8.20 (s, 1H), 8.18 (s, 1H), 7.80 (d, *J*=8.11 Hz, 2H), 7.70 (d, *J*=8.90 Hz, 2H), 7.57—7.53 (m, 2H), 7.40—7.36 (m, 2H), 7.33 (s, 1H), 7.31 (s, 1H), 5.47 (t, *J*=4.45 Hz, 1H), 1.99—1.94 (m, 2H), 1.03—0.95 (m, 2H), 0.55 (t, *J*=7.27 Hz, 3H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$ : 149.89, 131.40, 130.97, 128.78, 128.09, 126.49, 123.99, 122.40, 117.49, 116.64, 38.16, 30.98, 18.09, 14.03.

**14**-(*iso*-Butyl)-14-*H*-dibenzo[*a,j*]xanthene (**3p**)<sup>28</sup> <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz)  $\delta$ : 8.22 (s, 1H), 8.19 (s, 1H), 7.80 (s, 1H), 7.78 (s, 1H), 7.70 (s, 1H), 7.68 (s, 1H), 7.58—7.53 (m, 2H), 7.40—7.35 (m, 4H), 5.42 (t, *J*= 5.72 Hz, 1H), 1.80 (t, *J*=5.80 Hz, 2H), 1.50—1.40 (m, 1H), 0.70 (d, *J*=6.56 Hz, 6H); <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz)  $\delta$ : 150.14, 131.56, 130.92, 128.75, 127.99, 126.46, 124.01, 122.23, 118.63, 117.69, 46.79, 29.56, 25.02, 23.57.

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