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# Facile and Efficient Method for the Synthesis of 14-Substituted-14-Hdibenzo[a,j]xanthenes Catalyzed by Ruthenium Chloride Hydrate as a Homogeneous Catalyst

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### FACILE AND EFFICIENT METHOD FOR THE SYNTHESIS OF 14-SUBSTITUTED-14-*H*-DIBENZO[*a,j*]XANTHENES CATALYZED BY RUTHENIUM CHLORIDE HYDRATE AS A HOMOGENEOUS CATALYST

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#### **GRAPHICAL ABSTRACT**



**Abstract** Synthesis of dibenzoxanthenes through condensation of  $\beta$ -naphthol with various aromatic and aliphatic aldehydes in ethanol as an ecofriendly solvent using  $Ru^{III}$  as catalyst is reported.

Keywords Aldehyde; homogeneous catalyst; β-naphthol; ruthenium; xanthene

#### INTRODUCTION

Xanthene framework is featured in a wide variety of compounds that show pharmacological and biological activities such as antibacterials, antivirals, and anti-inflammatory agents.<sup>[1]</sup> Xanthene-based compounds are also being developed to act as new clinical agents in cancer therapy.<sup>[2]</sup> It is also noteworthy that upon oxidation, xanthene derivatives can be converted to corresponding xanthylium salts, which are candidates as dyes and fluorescent materials.<sup>[3]</sup> Xanthene dyes are extracted from natural sources.<sup>[4]</sup> Therefore, significant effort has been directed toward synthesis of xanthene derivatives. Synthetic 14-*H*-dibenzo[*a,j*]xanthenes have been prepared by different methods such as the reaction of 2-naphthol with 2-naphthol-1-methanol,<sup>[5]</sup> aldehyde acetals,<sup>[6]</sup> and carbon monoxide.<sup>[7]</sup> However, these methods suffer from long reaction times, unsatisfactory yields, tedious experimental procedures, and excess reagents. Recently, the synthesis of xanthenes has

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Scheme 1.  $RuCl_3 \cdot nH_2O$ -catalyzed synthesis of 14-substituted-14-H-dibenzo[a,j]xanthenes.

been improved by mixing  $\beta$ -naphthol with aldehydes in the presence of a catalyst such as *para*-toluenesulfonic acid (*p*-TSA),<sup>[8]</sup> sulfamic acid,<sup>[9]</sup> AcOH/H<sub>2</sub>SO<sub>4</sub>,<sup>[10]</sup> amberlyst 15,<sup>[11]</sup> LiBr,<sup>[12]</sup> I<sub>2</sub>,<sup>[13]</sup> cyanuric chloride,<sup>[14]</sup> K<sub>5</sub>CoW<sub>12</sub>O<sub>40</sub> · 3H<sub>2</sub>O,<sup>[15]</sup> HClO<sub>4</sub>-SiO<sub>2</sub>,<sup>[16]</sup> silica sulfuric acid,<sup>[17]</sup> Yb(OTf)<sub>3</sub>,<sup>[18]</sup> KAl(SO<sub>4</sub>)<sub>2</sub> · 12H<sub>2</sub>O (alum),<sup>[19]</sup> tetrabutylammonium bromide,<sup>[20]</sup> H<sub>14</sub>[NaP<sub>5</sub>W<sub>30</sub>O<sub>110</sub>],<sup>[21]</sup> and Dowex-50w.<sup>[22]</sup>. In spite of their potential utility, some of these methods have their own drawbacks such as use of toxic solvents<sup>[8]</sup> and catalysts,<sup>[13]</sup> cumbersome product isolation procedures, strong acidic conditions,<sup>[10]</sup> long reaction times,<sup>[8,9]</sup> use of expensive reagents, poor yields of products,<sup>[8]</sup> special apparatus, requirement of a large or stoichiometric amount of catalysts,<sup>[19]</sup> and drastic conditions for catalyst preparation.<sup>[16]</sup> Thus, development of new benign protocols using simple operating conditions is still required.

Recently, we have been involved in the study of the catalytic activity of ruthenium in organic reactions such as condensation of indoles and aldehydes,<sup>[23]</sup> oxidation of aromatic and heteroaromatic compounds,<sup>[24]</sup> double-conjugate 1,4-addition to enones,<sup>[25]</sup> nucleophilic addition to epoxides,<sup>[26]</sup> oxidative trimerization of indoles,<sup>[27]</sup> and cross-aldol reactions.<sup>[28]</sup> As a matter of fact, many organic transformations, which involve ruthenium species as catalyst, are known and well documented.<sup>[29–31]</sup>

We found that ruthenium chloride hydrate smoothly acts as a homogeneous catalyst in the double addition of  $\beta$ -naphthol to aldehydes, providing the desired xanthene derivatives in excellent yields under mild reaction conditions and using a relatively easy workup (Scheme 1).

#### **RESULTS AND DISCUSSION**

Typical results of the ruthenium-catalyzed condensation of  $\beta$ -naphthol and aldehydes are shown in Table 1. In procedure optimized with respect to the catalyst concentration, choice of solvent, temperature, and time, the treatment of benzal-dehyde (1 mmol) with  $\beta$ -naphthol (2 mmol) in the presence of RuCl<sub>3</sub> · *n*H<sub>2</sub>O catalyst (10 mol%) in ethanol (5 mL) at 100 °C for 2 h gave the corresponding dibenzox-anthene as a solid precipitate, which was easily purified (product **3a**, 90% yield).

As shown in Table 1, the present protocol works with a wide range of aromatic aldehydes bearing electron-withdrawing and electron-donating groups. All reactions were completed within 1–4 h, as indicated. Although much effort has been made to exclude the reaction solvent or to use environmentally friendly solvents in the reaction step, less attention has been paid to the high amounts of organic solvent waste in the purification step in the reported methods.<sup>[13,16,22]</sup> The unique feature of the reaction is that most of the corresponding dibenzoxanthene derivatives are insoluble in the reaction solvent, and this leads to an easy workup along with excellent yields (80–94%).

Entry	Aldehyde	Time (h)	Product	Yield (%) <sup>a</sup>	Mp (°C)	
					Found	Reported
1	СНО	2	3a	90 <sup>b</sup>	180–182	181–183
2	O <sub>2</sub> N-CHO	1	3b	92 <sup>c</sup>	326–328	324
3	СІ—	2	3c	92 <sup><i>b</i></sup>	308–310	300-302
4	Br	1.5	3d	94 <sup><i>d</i></sup>	298–300	297–299
5	СІ	2	3e	86 <sup>e</sup>	218–220	214–216
6	СІ-СНО	1.5	3f	90 <sup>b</sup>	255–258	253–255
7	О <sub>2</sub> N СНО	1.5	3g	86 <sup>c</sup>	212–214	212–220
8	Н <sub>3</sub> С-СНО	2.5	3h	87 <sup>f</sup>	230–232	229
9	Н <sub>3</sub> СО-СНО	3	3i	90 <sup>b</sup>	215–218	213–215
10	но-Д-Сно	2.5	3j	85 <sup>f</sup>	140–142	140
11	H <sub>3</sub> CO H <sub>3</sub> CO	3	3k	89	208–210	_
12	ССНО	4	31	85	260–265	_
13	CH <sub>3</sub> CH <sub>2</sub> CHO	3.5	3m	$82^g$	150-152	152

Table 1. RuCl<sub>3</sub> · nH<sub>2</sub>O-catalyzed synthesis of 14-substituted-14-H-dibenzo[a,j]xanthenes

(Continued)

	Aldehyde	Time (h)	Product	Yield (%) <sup>a</sup>	Mp (°C)	
Entry					Found	Reported
14	CH <sub>3</sub> CH <sub>2</sub> CH <sub>2</sub> CHO	4	3n	85 <sup>e</sup>	153–154	152–154
15	(CH <sub>3</sub> ) <sub>2</sub> CHCHO	3	30	$80^{h}$	154-156	155–157
16	(CH <sub>3</sub> ) <sub>2</sub> CHCH <sub>2</sub> CHO	4	3p	$84^i$	110-112	112-113

Table 1. Continued

*Note.* All products were characterized by <sup>1</sup>H NMR, <sup>13</sup>C NMR, and IR data. <sup>*a*</sup>Isolated yields.

<sup>b</sup>Identified by comparison with authentic samples.<sup>[20]</sup>

<sup>c</sup>Identified by comparison with authentic samples.<sup>[21]</sup>

<sup>d</sup>Identified by comparison with authentic samples.<sup>[22]</sup>

<sup>e</sup>Identified by comparison with authentic samples.<sup>[16]</sup>

<sup>f</sup>Identified by comparison with authentic samples.<sup>[17]</sup>

gIdentified by comparison with authentic samples.<sup>[8]</sup>

<sup>h</sup>Identified by comparison with authentic samples.<sup>[14]</sup>

<sup>*i*</sup>Identified by comparison with authentic samples.<sup>[19]</sup>



Scheme 2. Plausible mechanism for the synthesis of xanthene in the presence of  $RuCl_3 \cdot nH_2O$  as catalyst.

In many cases, simple filtration of the reaction mixture and washing with cold ethanol provided spectroscopically pure products. It was also observed that the electronic properties of the aromatic ring have an effect on the rate of this nucleophilic addition reaction. The rate is accelerated if an electron-withdrawing group is present on the aromatic ring. With regard to the aliphatic aldehydes, however, the present protocol showed longer reaction times and lower yields (Table 1, entries 13–16).

A plausible mechanism for the reaction under ruthenium catalysis is outlined in Scheme 2. As it is shown, prior activation of the carbonyl group of aldehyde by  $Ru^{III}$  to give intermediate 1, followed by a nucleophilic attack from  $C_1$  of  $\beta$ -naphthol, provides intermediate 2, which serves as an electrophile, ready for attack by a second molecule of  $\beta$ -naphthol. After loss of water, bis(naphtholyl)methane species (3) undergo dehydration to afford the desired product (4).

#### CONCLUSION

In summary, we have developed a simple, convenient, and efficient synthetic protocol for the double addition of  $\beta$ -naphthol to aldehydes. To the best of our knowledge, this is the first report on the use of Ru<sup>III</sup> salts as catalyst for the preparation of 14-substituted-14-*H*-dibenzo[*a*,*j*]xanthene derivatives.

This method offers several significant advantages such as simplicity of the reaction procedure, short reaction times, low catalyst loading, exellent yields of products, use of ethanol as an ecofriendly solvent, efficiency, and mild reaction conditions. Further manipulation of this reaction is under way.

#### EXPERIMENTAL

All products were characterized by physical data (mp), spectral data [Infrared (IR), <sup>1</sup>H NMR, <sup>13</sup>C NMR] and elemental analysis. IR spectra were obtained on a Shimadzu Fourier transform (FT)–IR-8400S spectrometer. <sup>1</sup>H NMR spectra were measured on a Bruker DRX-500 Avance spectrometer, and <sup>13</sup>C NMR spectra were obtained on a Bruker DRX-125 Avance spectrometer. Chemical shifts of <sup>1</sup>H and <sup>13</sup>C NMR spectra were expressed in parts per million (ppm) downfield from tetramethylsilane (TMS) as internal standard. Melting points were measured on a Büchi meltingpoint B-540 instrument and are uncorrected. Elemental analyses were made by a Carlo-Erba EA1110 CNNO-S analyzer and results agreed with the calculated values.

#### Materials

 $\beta$ -Naphthol, aldehydes, RuCl<sub>3</sub>·nH<sub>2</sub>O, and solvents were purchased from Merck and used without further purification.

#### Ruthenium-Catalyzed Condensation of β-Naphthol and Benzaldehyde

A mixture of  $\beta$ -naphthol (288.34 mg, 2 mmol), benzaldehyde (106.13 mg, 1 mmol), and RuCl<sub>3</sub> · *n*H<sub>2</sub>O (21.43 mg, 0.1 mmol) in ethanol (5 mL) was refluxed in an oil bath at 100 °C for 2 h and yielded a solid precipitate. The progress of the reaction was monitored by thin-layer chromatography (TLC). After completion of the

reaction, as indicated by TLC, simple filtration of the reaction mixture and washing with ethanol provided a pure product (322.62 mg, 90%). The same procedure was also used for the other products listed in Table 1. In the case of entries 10–12 in Table 1, purification of the reaction mixture by preparative TLC (petroleum ether–ethyl acetate, 10:3) provided pure products.

#### Selected Characterization Data

**14-(3,4-Dimethoxyphenyl)-14-***H***-dibenzo**[*a,j*]**xanthene (3k).** Solid; mp 208–210 °C, IR (KBr):  $\nu$  (cm<sup>-1</sup>); 3060, 2920, 1620, 1595, 1505, 1457, 1260, 1245, 1210, 1130, 1020, 810, 740; <sup>1</sup>H NMR [500 MHz, (CD<sub>3</sub>)<sub>2</sub>SO, 25 °C]  $\delta$  = 8.1 (d, 2H, *J* = 8.5 Hz), 7.72 (d, 2H, *J* = 7.8 Hz), 7.65 (d, 2H, *J* = 8.6 Hz), 7.23 (t, 2H, *J* = 7.1 Hz), 7.17 (t, 2H, *J* = 7.2 Hz), 7.10 (d, 2H, *J* = 8.6 Hz), 6.96 (s, 1H), 6.73 (d, 1H, *J* = 8.1 Hz), 6.72 (s, 1H), 6.44 (d, 1H, *J* = 8.1 Hz), 3.68 (s, 3H), 3.50 (s, 3H) ppm; <sup>13</sup>C NMR [125 MHz, (CD<sub>3</sub>)<sub>2</sub>SO, 25 °C]  $\delta$  = 149.30, 147.69, 137.14, 135.11, 129.34, 129.25, 129.22, 126.91, 126.63, 124.48, 122.73, 121.21, 120.79, 120.28, 113.58, 112.20, 56.34, 56.27, 41.92 ppm. Anal. calcd. for C<sub>29</sub>H<sub>22</sub>O<sub>3</sub>: C, 83.23; H, 5.30. Found: C, 83.29; H, 5.32.

**14-(Fluorene-2-yl)-14-***H***-dibenzo[***a,j***]xanthene (31). Solid; mp 260–265 °C, IR (KBr): \nu (cm<sup>-1</sup>); 3070, 2900, 1590, 1510, 1457, 1430, 1400, 1250, 1240, 815, 765, 740; <sup>1</sup>H NMR [500 MHz, (CD<sub>3</sub>)<sub>2</sub>SO, 25 °C] \delta = 8.72 (d, 2H,** *J* **= 8.4 Hz), 7.91–7.93 (m, 4H), 7.83 (d, 1H,** *J* **= 7.7 Hz), 7.61–7.65 (m, 5H), 7.59 (d, 2H,** *J* **= 8.8 Hz), 7.44 (t, 2H,** *J* **= 7.3 Hz), 7.37 (d, 1H,** *J* **= 7.2 Hz), 7.22 (t, 1H,** *J* **= 7.2 Hz), 7.16 (t, 1H,** *J* **= 7.2 Hz), 6.77 (s, 1H), 3.63 (s, 2H) ppm; <sup>13</sup>C NMR [125 MHz, (CD<sub>3</sub>)<sub>2</sub>SO, 25 °C] \delta = 148.85, 145.20, 144.21, 143.80, 141.28, 140.27, 131.82, 131.54, 129.87, 129.45, 127.78, 127.72, 127.40, 125.73, 125.38, 124.34, 120.55, 120.42, 118.63, 118.38, 37.55, 37.02 ppm. Anal. calcd. for C<sub>34</sub>H<sub>22</sub>O: C, 91.45; H, 4.97. Found: C, 91.40; H, 5.0.** 

Supplementary data are also available for the other products listed in Table 1.

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