

1,3,5-Trichloro-2,4,6-Triazinetrion: A Versatile Heterocycle for the One-Pot Synthesis of 14-Aryl- or Alkyl -14*H*-Dibenzo[a,j]xanthene, 1,8-Dioxooctahydroxanthene and 12-Aryl-8,9,10,12-Tetrahydrobenzo[a]xanthene-11-one Derivatives under Solvent-Free Conditions

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Received December 14, 2010, Accepted January 23, 2011

A facile, green, efficient and environment-friendly protocol for the synthesis of 14-aryl- or alkyl-14*H*-dibenzo[a,j]xanthene, 1,8-dioxooctahydroxanthene and 12-aryl-8,9,10,12-tetrahydrobenzo[a]xanthene-11-one have been developed by one-pot condensation of various aldehydes with (i) β -naphthol (ii) cyclic 1,3-dicarbonyl compounds and (iii) β -naphthol and cyclic 1,3-dicarbonyl compounds, in the presence of 1,3,5-trichloro-2,4,6-triazinetrion (trichloroisocyanuric acid, TCCA) as catalyst under solvent-free conditions. The present approach offers the advantages of clean reaction, simple methodology, short reaction time, easy purification, and economic availability of the catalyst.

Key Words : β -Naphthol, Aldehydes, Trichloroisocyanuric acid, Cyclic 1,3-dicarbonyl compounds; One-pot synthesis

Introduction

Organic syntheses involving greener process and under solvent-free conditions have been investigated world wide due to stringent environment and economic regulations.¹⁻⁴ In addition, with increasing environmentally concerns and the regulatory constraints faced in the chemical and pharmaceutical industries, development of environmentally benign organic reactions has become a crucial and demanding research area in modern organic chemical research.⁵

Xanthenes and benzoxanthenes are important biologically active heterocyclic compounds, which possess antiviral,⁶ antibacterial⁷ and anti-inflammatory⁸ activities. These are being utilized as antagonists for paralyzing action of zoxazolamine⁹ and in photodynamic therapy.¹⁰ Furthermore, these compounds can be used as dyes,¹¹ in laser technologies¹² and as pH sensitive fluorescent materials for visualization of biomolecules.¹³

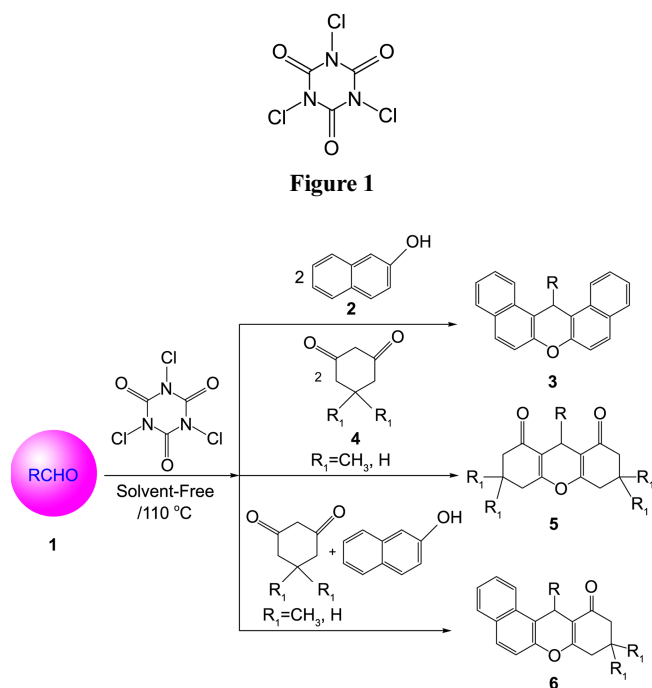
Because of their wide range of pharmacological, industrial and synthetic application, many methods for their preparation are reported in the literature.¹⁴⁻²¹ of these methods, the condensation reaction of aldehydes with (i) β -naphthol (ii) cyclic 1,3-dicarbonyl compounds and (iii) β -naphthol and cyclic 1,3-dicarbonyl compounds are one of the most simple and straightforward approaches for the synthesis of 14-aryl or alkyl-14*H*-dibenzo[a,j]xanthenes, 1,8-dioxooctahydroxanthene and 12-aryl-tetrahydrobenzo[a]xanthene-11-ones, respectively. A variety of reagents such as P_2O_5/Al_2O_3 under microwave irradiation,²² silica sulfuric acid,^{23,24} boric acid,²⁵ $BF_3 \cdot SiO_2$,²⁶ oxalic acid,²⁷ $TaCl_5$,²⁸ $KAl(SO_4)_2 \cdot 12H_2O$ (alum),²⁹ tetrabutylammonium bromide,³⁰ 1-methyl-3-propane sulfonic-imidazolium hydrosulfate,³¹ methanesulfonic acid,³²

$ZrOCl_2 \cdot 8H_2O$,³³ polyaniline-*p*-toluenesulfonate salt,³⁴ antimony trichloride- SiO_2 ,³⁵ trichloroisocyanuric acid,³⁶ $NaHSO_4 \cdot SiO_2$,³⁷ *p*-TSA in ionic liquid ($[bmim]BF_4$),³⁸ $InCl_3$ or P_2O_5 ,³⁹ strontium triflate,⁴⁰ praline triflate,⁴¹ $HBF_4 \cdot SiO_2$,⁴² 1-butyl-3-methylimidazolium hydrogen sulfate $[bmim]HSO_4$,⁴³ $NH_4H_2PO_4/SiO_2$,⁴⁴ cyanuric chloride,^{45,46} Dowex-50W,⁴⁷ H_3PO_4 or $HClO_4$,⁴⁸ dodecylbenzenesulphonic acid,⁴⁹ diammonium hydrogen phosphate,⁵⁰ and Caros acid⁵¹ have been employed to accomplish this transformation. Unfortunately, many of these existing methods, used for the synthesis of xanthenes derivatives accompanying with one or other kinds of the disadvantages, such as, use of volatile organic solvents, prolonged reaction time, tedious work-up procedures, expensive reagents, high catalyst loading and strongly acidic conditions. Therefore, to avoid these limitations, the development of a new readily available catalyst with high catalytic activity, short reaction time and simple work-up for the preparation of xanthenes is highly desirable.

Result and Discussions

In continuous demand to develop synthetic method for the synthesis of xanthenes compounds under mild conditions and by using non-toxic and not expensive reagents has prompted the our group research to investigate the use of TCCA. 1,3,5-Trichloro-2,4,6-triazinetrion (trichloroisocyanuric acid or TCCA) (Fig. 1) have traditionally found applications in analytical chemistry as complexation agents, in electrochemistry as multi-step redox systems and as beaching agents, disinfectants, bactericides and pesticide or herbicide components in agriculture.^{52,53}

During the course of our studies towards the development



of useful synthetic methodologies,⁵⁴⁻⁵⁹ we wish to report a simple, green, efficient and practical for the synthesis of xanthenes derivatives using TCCA as eco-friendly and neutral catalyst with high catalytic activity under solvent-free condition at 110 °C (Scheme 1).

Initially, 4-chlorobenzaldehyde was selected as a probe aldehyde to optimize the reaction conditions, and the results are listed in Table 1. Obviously, the temperature and the amount of catalyst had important effects on the reaction. The results in Table 1 showed some interesting points. First of all, increasing amount of TCCA did not improve obviously the yields of **3b** (entry 5). So, the optimum amount of TCCA was found to be 5 mol % relative to reactants (β -naphthol, 2 mmol and 4-chlorobenzaldehyde, 1 mmol). The second important point which could be elicited evidently from these results is that raising the reaction temperature from 110 to 120 °C did not increase the yield and also did not improve the reaction rates (entry 3). As shown in Table 1, nearly no xanthene could be detected in the absence of TCCA (entry 6).

Using the best conditions reported in Table 1 (entry 2), we continued to investigate the reaction at temperature 110 °C

Table 1. Optimizing the reaction conditions

Entry	Catalyst (mol %)	Temperature (°C)	Time (min)	Yield (%) ^a
1	5	100	50	74
2	5	110	50	86
3	5	120	50	84
4	2	110	50	57
5	10	110	50	80
6	—	110	50	—

^aIsolated Yields.

Table 2. The one-pot synthesis of 14-aryl or alkyl-14*H*-dibenzo-[a,j]xanthenes using TCCA

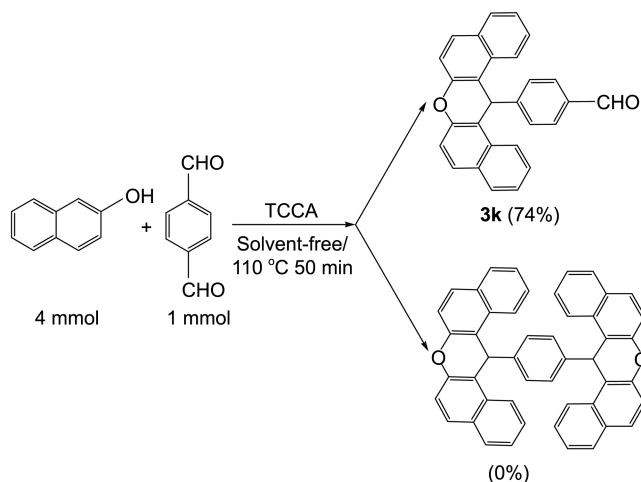
Product (3)	R	Time (min)	Yield (%) ^a	Mp (°C)	
				Found	Reported
a	C ₆ H ₅	50	82	186-187	183-185
b	4-ClC ₆ H ₄	50	86	290-292	289-291
c	2-ClC ₆ H ₄	50	80	216-218	213-215
d	3-BrC ₆ H ₄	50	82	190-192	192-193
e	4-BrC ₆ H ₄	50	86	301-302	298-300
f	3-NO ₂ C ₆ H ₄	50	80	212-213	210-211
g	4-NO ₂ C ₆ H ₄	50	74	312	310-312
h	2-CH ₃ OC ₆ H ₄	60	80	259-260	258-259
i	4-CH ₃ OC ₆ H ₄	60	86	200-202	203-205 [35]
j	4-FC ₆ H ₄	50	85	241-243	238-240 [35]
k	4-CHOC ₆ H ₄ ^b	50	74	308-310	310-312
l	Pyridyl-2-yl	70	62	236-237	236-237
m	Pyridyl-3-yl	70	80	201-203	200-202
n	4-CH ₃ C ₆ H ₄	50	83	228-229	226-228
o	Isopropyl	60	82	157-158	154-156
p	<i>n</i> -Propyl	60	81	152-154	152-154

^aThe yields refer to the isolated pure products which were characterized from their spectral data and were compared with authentic sample. ^b β -naphthol/aldehyde (4:1).

under solvent-free with 5 mol % of TCCA. The desired product was obtained in satisfactory yields. Encouraged by these results, we then continued to study the reaction using various aldehydes in the presence of 5 mol % of TCCA. The results are summarized in Table 2 indicating that aromatic, aliphatic and heteroaromatic aldehydes underwent smooth reaction with β -naphthol to give high yields of products.

The practical synthetic efficiency of this reaction was highlighted by the reaction of terephthalaldehyde with β -naphthol to give structurally complex xanthenes derivative (**3k**) (Scheme 2).

We expected that both of the formyl groups on the aromatic ring of terephthalaldehyde would react with β -naphthol. However, we observed that one of the formyl



Scheme 2. Reaction between terephthalaldehyde and excess amount of β -naphthol.

Table 3. Preparation of 1,8-dioxooctahydroxanthene using TCCA (5 mol %)

Product (5)	R	R ¹	Time (min)	Yield (%) ^a	Mp (°C)	
					Found	Reported
a	C ₆ H ₅	CH ₃	20	86	203-205	203-204
b	4-ClC ₆ H ₄	CH ₃	20	84	233-235	230-232
c	2-ClC ₆ H ₄	CH ₃	20	83	225-227	225-227
d	2-NO ₂ C ₆ H ₄	CH ₃	20	85	251-253	252-254
e	4-NO ₂ C ₆ H ₄	CH ₃	20	86	224-226	222-223
f	3-NO ₂ C ₆ H ₄	CH ₃	20	88	168-170	170-172
g	4-CH ₃ C ₆ H ₄	CH ₃	20	80	218-219	215-217
h	4-BrC ₆ H ₄	CH ₃	20	90	237-239	240-241
i	4-FC ₆ H ₄	CH ₃	20	84	226-227	226-227
j	2-NO ₂ C ₆ H ₄	H	15	81	240-242	245-247
k	4-NO ₂ C ₆ H ₄	H	15	82	224-226	224-226
l	C ₆ H ₅	H	15	72	203-205	201-203
m	4-ClC ₆ H ₄	H	15	70	229-231	231-233
n	4-CH ₃ C ₆ H ₄	H	15	82	218-220	216-218
o	4-BrC ₆ H ₄	H	15	86	229-231	228-231

^aIsolated yields.

groups was condensed with β -naphthol and another group was intact because of the steric effects between *o*-hydrogens of benzene ring and the xanthenes ring.²²

After successfully synthesizing of a series of 14-aryl or alkyl-14*H*-dibenzo[*a,j*]xanthenes in good yields. We turned out attention toward the synthesis of 1,8-dioxooctahydroxanthene under similar conditions. we replaced the cyclic 1,3-dicarbonyl compounds (1,3-cyclohexanedione or 5,5-dimethyl-1,3-cyclohexanedione) instead of β -naphthol in same conditions (Scheme 1). We next examined the reaction with 2 equivalents of cyclic 1,3-dicarbonyl compounds **4** with various aldehydes. As expected, these substrates underwent smooth, one-pot conversion to give the corresponding 1,8-dioxooctahydroxanthene in good yields (Table 3).

Finally, we have developed this synthetic method for one-pot efficient synthesis of 12-aryl-tetrahydrobenzo[*a*]xanthene-11-ones by condensation aldehydes with β -naphthol and cyclic 1,3-dicarbonyl (1,3-cyclohexanedione or 5,5-dimethyl-1,3-cyclohexanedione).

A multicomponent reaction is a one-pot reaction in which three or more reactants are combined together to generate a desired product without the isolation of any intermediate.^{60,61} The MCR strategy has gained increasing attention in the past decade because of its capability to prepare compound libraries in the field of modern medicinal and combinatorial chemistry. The combination of solvent-free and MCR reactions represents a very powerful method from both economical and synthetic points of view since the isolation of the intermediates is skipped, the overall reaction time is significantly decreased, higher yields of products are obtained, and due to less use of solvents and reagents the costs are lowered.^{62,63}

We first studied a reaction between 5,5-dimethyl-1,3-cyclohexanedione (1.2 mmol), 4-chlorobenzaldehyde (1 mmol) and β -naphthol (1mmol) by screening the reaction

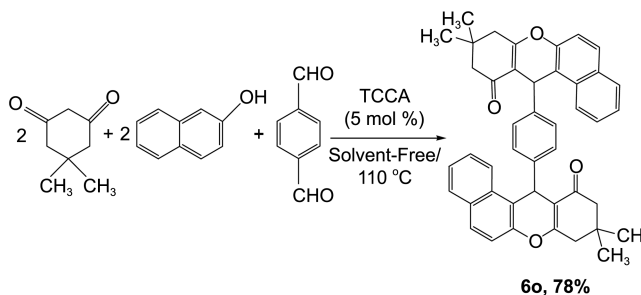
Table 4. Preparation 12-aryl-tetrahydrobenzo[*a*]xanthene-11-ones TCCA (5 mol %)

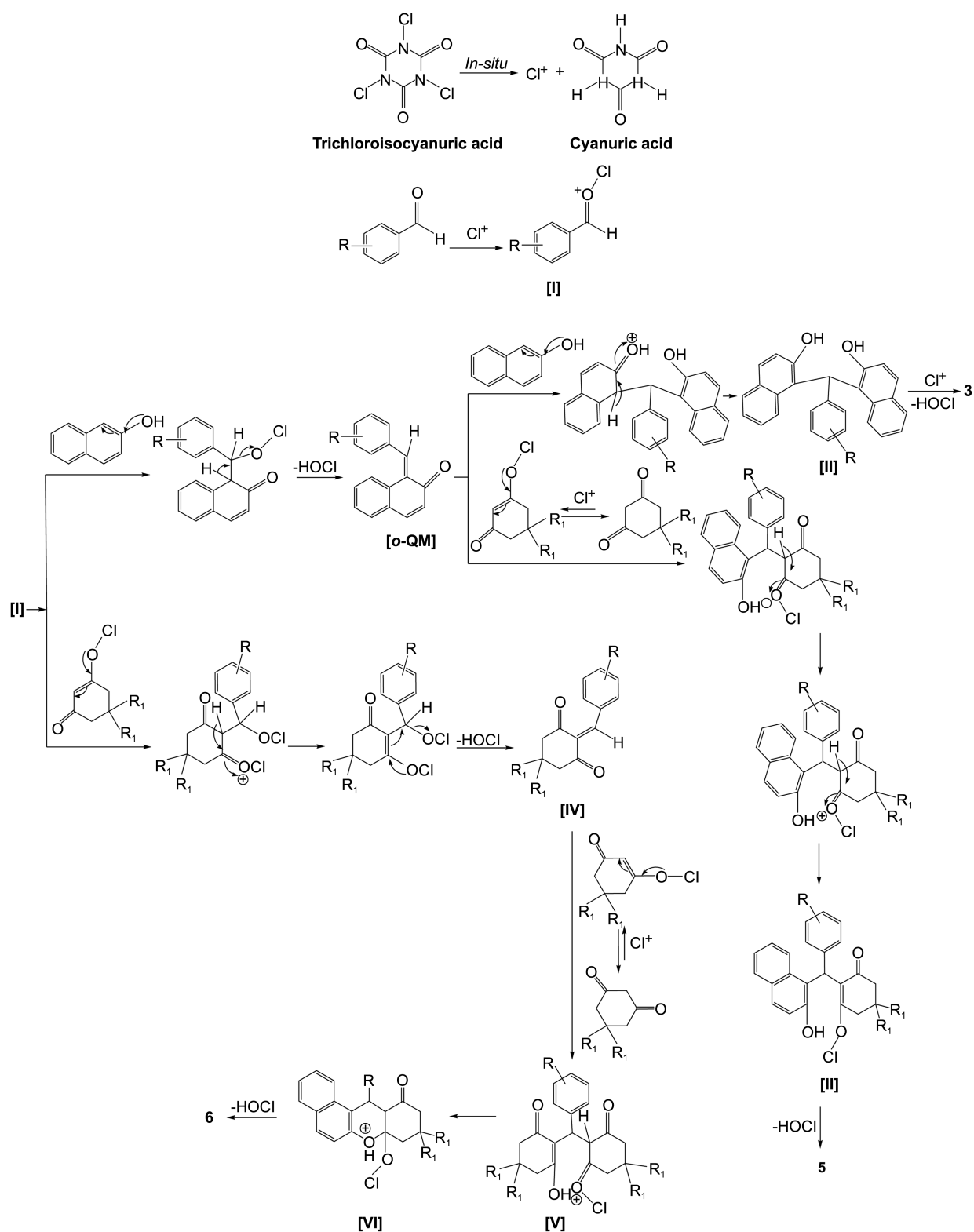
Product (6)	R	R ¹	Time (min)	Yield (%) ^a	Mp (°C)	
					Found	Reported
a	C ₆ H ₅	CH ₃	40	80	150-151	151-153
b	4-ClC ₆ H ₄	CH ₃	40	84	184-185	186-188
c	2-ClC ₆ H ₄	CH ₃	40	86	180-181	179-180
d	2-NO ₂ C ₆ H ₄	CH ₃	40	74	220-222	223-225
e	4-NO ₂ C ₆ H ₄	CH ₃	40	78	175-177	178-180
f	3-NO ₂ C ₆ H ₄	CH ₃	40	80	167-168	168-170
g	4-MeC ₆ H ₄	H	25	86	208-210	205-206
h	4-FC ₆ H ₄	H	25	75	214-216	210-212
i	2-CH ₃ OC ₆ H ₄	CH ₃	30	88	167-168	163-165
j	4-ClC ₆ H ₄	H	25	90	207-208	205-206
k	4-CH ₃ C ₆ H ₄	H	25	80	208-210	205-207
l	3-NO ₂ C ₆ H ₄	H	25	86	230-232	235-236
m	4-NO ₂ C ₆ H ₄	H	25	75	232-234	234-235
n	C ₆ H ₅	H	25	74	192-194	189-190
o	4-CHOC ₆ H ₄ ^b	CH ₃	40	78	307-309	310-311

^aIsolated yields. ^b β -naphthol/5,5-dimethyl-1,3-cyclohexanedione/aldehyde (2:2:1).

conditions. In order to determine the optimum conditions, we examined the influence of the reaction temperature, the reaction time, and the amount catalyst. It can be seen that the best result was obtained with 5 mol % of TCCA at 110 °C. After optimizing the conditions, we next examined the generality of these conditions to other substrates using several aromatic aldehydes (Table 4).

Following the success from synthesis 12-aryl-tetrahydrobenzo[*a*]xanthene-11-ones by condensation aldehydes with β -naphthol and cyclic 1,3-dicarbonyl (1,3-cyclohexanedione or 5,5-dimethyl-1,3-cyclohexanedione), we have developed this synthetic method for the preparation of additional extended bis-12-aryl-tetrahydrobenzo[*a*]xanthene-11-ones derivative in a 2:2:1 molar ratio of β -naphthol/5,5-dimethyl-1,3-cyclohexanedione/terephthalaldehyde using 10 mol % of TCCA (product **6o**) (Scheme 3). As expected, the reaction proceeded smoothly for 40 min with yield 78% under solvent-free conditions at 110 °C [mp 307-309 °C, Lit.⁴⁶ 310-311 °C], ¹H NMR (90 MHz, CDCl₃) δ 0.82 (s, 6H), 1.02 (s, 6H), 2.12-2.18 (m, 4H), 2.43 (s, 2H), 2.55 (s, 2H), 5.64 (s, 1H), 5.82 (s, 1H), 7.09-7.89 (m, 16 H). IR (KBr disc) cm⁻¹ 3040, 2996, 1682, 1622, 1486, 1400, 1375, 1228, 1168.

**Scheme 3.** Reaction between terephthalaldehyde with 5,5-dimethyl-1,3-cyclohexanedione and β -naphthol in the presence of TCCA.



Scheme 4. Proposed mechanisms.

On the basis of previously reported mechanism for applying TCCA to organic transformation^{64,65} and our observation in the course of reaction, one explanation for this process is that the TCCA releases Cl^+ *in-situ*,^{66,67} Which can act as an electrophilic species to activate the carbonyl oxygen [I].

Therefore, the proposed mechanism for this transformation is shown in (Scheme 4). For synthesis of 14-aryl- or alkyl β -14*H*-dibenzo[*a,j*]xanthene, and 12-aryl-8,9,10,12-tetrahydrobenzo[*a*]xanthene-11-one, the reaction may proceed via the ortho-quinone methides intermediate (*o*-QM), which was

formed by the nucleophilic addition of β -naphthol to aldehyde catalyzed by Cl^+ . Subsequent Michael addition of β -naphthol or cyclic 1,3-dicarbonyl (1,3-cyclohexanedione or 5,5-dimethyl-1,3-cyclohexanedione) to the *o*-QM form intermediates [III] and [III], followed by cyclization to give the corresponding products 3 and 6, accompanied by loss of HOCl. Finally for synthesis of 1,8-dioxooctahydroxanthene, one molecule of cyclic 1,3-dicarbonyl (1,3-cyclohexanedione or 5,5-dimethyl-1,3-cyclohexanedione) was firstly condensed with an activated aldehyde [II] to afford [IV]. Then the active methylene of another molecule of cyclic 1,3-dicarbonyl (1,3-cyclohexanedione or 5,5-dimethyl-1,3-cyclohexanedione) reacted with [IV] via conjugate addition reaction to give the intermediate [V]. Then the intermediate [V] cyclized by nucleophilic attack of the OH group on the carbonyl (C=O) moiety and gave the intermediate [VI]. Finally, the expected products 5 resulted from elimination of HOCl.

Conclusion

In conclusion, we have demonstrated a very simple, efficient, clean, green, and practical method for the synthesis of xanthenes derivatives in good yields. Furthermore, TCCA is a catalyst with cyanuric acid as by-product which is removable by washing with water.

Experimental

Chemicals were obtained from Merck and Fluka. IR spectra were recorded on a Shimadzu 435-U-04 spectrophotometer (KBr pellets). ^1H NMR spectra were obtained using Jeol FT NMR 90 MHz and spectrometer in CDCl_3 using TMS as an internal reference. Melting points were determined in open capillary tubes in a Stuart BI Branstead Electrothermal Cat No:IA9200 apparatus and uncorrected.

General Procedure for the Synthesis of Xanthene Derivatives.

(i) Synthesis of 14-Aryl or Alkyl-14*H*-dibenzo[*a,j*]xanthenes: To a mixture of aldehyde (1, 1 mmol) and β -naphthol (2, 2 mmol), TCCA (5 mol %) was added and the mixture was heated on an oil bath at 110 °C with good stirring for the appropriate time as indicated in Table 2. The progress of the reaction was monitored by TLC (*n*-hexane:ethyl acetate, 8:2), after completion of the reaction, hot EtOH (5 mL, 96%) was added and the mixture stirred for 5 min. Then, the catalyst separated by filtration. The residue was washed with ice-water. A solid precipitated that was collected by filtration and washed with H_2O and then dried to give the corresponding 14-aryl or alkyl-14*H*-dibenzo[*a,j*]xanthenes (3*a-p*). For further purification this product was recrystallized from ethanol to afford the pure products.

(ii) Synthesis of 1,8-Dioxo-octahydroxanthene: To a mixture of β -naphthol (2, 1 mmol) and cyclic 1,3-dicarbonyl compounds (4, 2 mmol), TCCA (5 mol %) was added and the mixture was heated on an oil bath at 110 °C with good stirring for the appropriate time as indicated in Table 3.

Work-up for this reaction is same for reaction of 14-aryl or alkyl-14*H*-dibenzo[*a,j*]xanthenes.

(iii) Synthesis of 12-Aryl-tetrahydrobenzo[*a*]xanthene-11-ones: In a separate set of experiments, To a mixture of β -naphthol (2, 1 mmol), cyclic 1,3-dicarbonyl compounds (4, 1.2 mmol), and aldehyde (1, 1 mmol), TCCA (5 mol %) was added and the mixture was heated on an oil bath at 110 °C for the reasonable time. these reactions were all repeated exactly under the above same conditions. The results obtained are showed in Table 4.

Spectra Data for Selected Compounds.

Compound 3b: ^1H NMR (90 MHz, CDCl_3) δ 6.52 (s, 1H), 7.44-8.40 (m, 19 H). IR (KBr disc) cm^{-1} 3010, 2920, 1580, 1510, 1480, 1460, 1420, 1400, 1240, 1132.

Compound 5h: ^1H NMR (90 MHz, CDCl_3) δ 1.01 (s, 6H), 1.14 (s, 6H), 2.24 (m, 4H), 2.50 (m, 4H), 5.14 (s, 1H), 7.12-7.84 (m, 4 H). IR (KBr disc) cm^{-1} 3020, 2982, 1686, 1662, 1460, 1402, 1378, 1240, 1174.

Compound 6b: ^1H NMR (90 MHz, CDCl_3) δ 0.98 (s, 3H), 1.06 (s, 3H), 2.28 (s, 2H), 2.55 (s, 2H), 5.70 (s, 1H), 7.18-7.82 (m, 12 H). IR (KBr disc) cm^{-1} 3060, 2980, 2880, 1660, 1480, 1400, 1360, 1230, 1140.

Acknowledgments. The authors are thankful to research council of Sabzevar Tarbiat Moallem University for financial support.

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