

GREEN SYNTHESIS OF POLYSUBSTITUTED QUINOLINES AND XANTHENE DERIVATIVES PROMOTED BY TARTARIC ACID AS A NATURALLY GREEN CATALYST UNDER SOLVENT-FREE CONDITIONS

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Abstract. Tartaric acid was employed as a naturally green catalyst for economical and facile preparation of polysubstituted quinolines *via* Friedländer hetero-annulation/condensation, 12-aryl-tetrahydrobenzo[*a*]xanthene-11-ones, 1,8-dioxo-octahydroxanthenes and 14-aryl-14*H*-dibenzo[*a,j*]xanthenes in solvent-free, one-pot multi-component reactions. This environmental friendly protocol provides high to excellent yields, short reaction times, clean reactions, simplicity and easy work up and mild conditions compared to the traditional method of synthesis. Furthermore, naturally green, low-cost and non-toxic catalyst made this protocol economic and sustainable.

Keywords: tartaric acid, polysubstituted quinoline, 12-aryl-tetrahydrobenzo[*a*]xanthene-11-one, 1,8-dioxo-octahydroxanthene, 14-aryl-14*H*-dibenzo[*a,j*]xanthene.

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Introduction

One of the dominating factors in recent organic synthetic routes is green chemistry. Atom economy, reduction in byproduct, in the number of steps of organic synthesis, in energy cost, in produced waste, use of non-hazardous reagents in catalytic protocols are among the most important goals of green chemistry. Furthermore, organic reactions under solvent-free conditions for green and clean synthesis of organic compounds have attracted much interest by organic chemists. And, herein, our recent studies focused on the development of a green catalyst [1,2] in multi-component reactions [3-6].

In recent years, the use of tartaric acid as a catalyst has received considerable attention in organic chemistry [7-9]. It is well known that tartaric acid has many applications in pharmacy, textile and food [10] industries because of its important advantages, such as green, natural origin, low-cost, high efficiency. Tartaric acid is added to food in order to give a sour taste, and usually is used as an antioxidant [11]. This compound is also used in the production of jams, sweets, jelly, tinned fruit and vegetables, cocoa powder and frozen dairy products mainly as an acidity adjuster but also in the form of as an

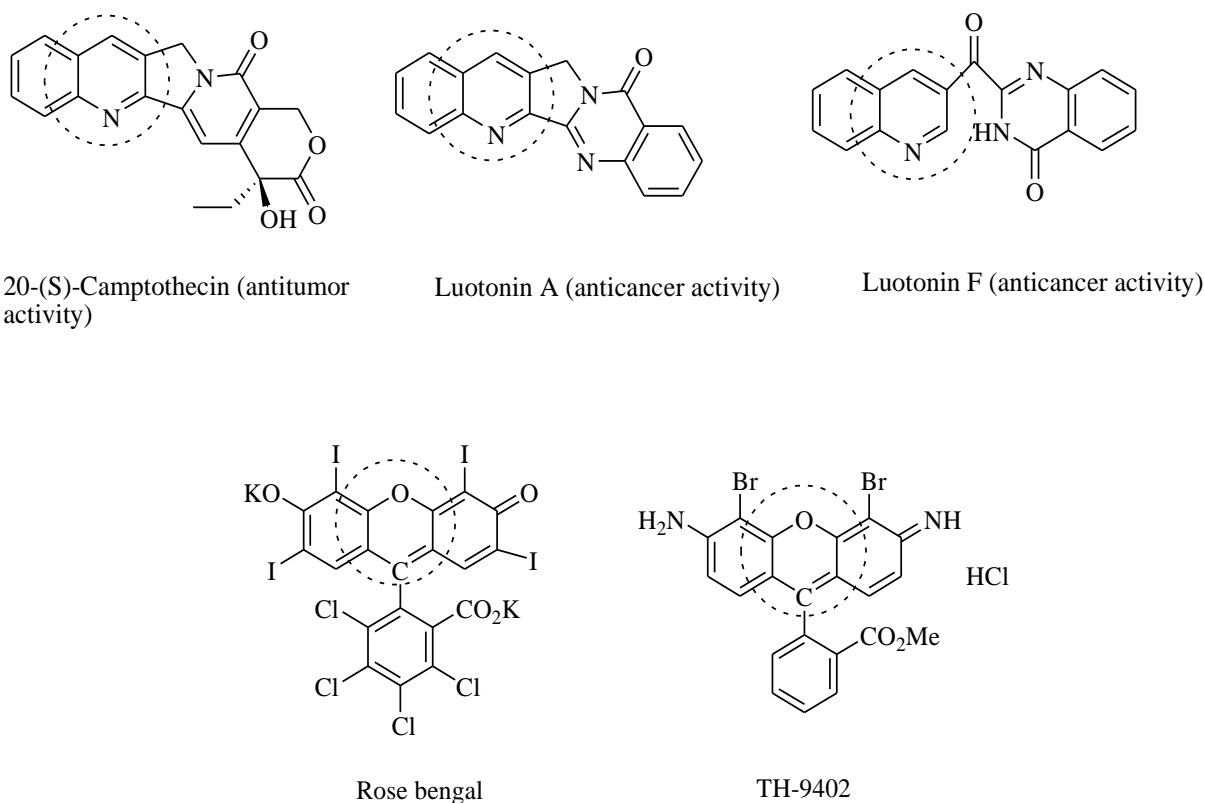
emulsifier [12]. In regards to acid adjustment, it is one of the strongest acids naturally occurring in fruit and is the strongest acid in grapes and wine ($pK_{a1} = 2.90$) [13]. Tartaric acid is relatively stable microbiologically, compared to the other naturally occurring organic acids, such as maleic and citric acids in wine industry [12].

Synthesis of heterocyclic compounds has attracted great interests due to their wide applicability in life and nature. Such compounds with quinoline and xanthene ring systems exhibit DNA binding capability [14], antitumor [15,16] (Figure 1) and antihypertensive [17] activities, tyro kinase platelet-derived growth factor receptors inhibiting [18] and antiplasmodial activity [19]. In addition to medical applications, quinoline derivatives are found to undergo hierarchical self assembly into a variety of nanostructures and meso structures with enhanced electronic and photonic functions [20]. Besides, xanthene derivatives have been widely used as pH sensitive fluorescent materials for visualization of biomolecules [21], xanthene dyes have been used in antiviral therapy [22] and as sensitizers in photodynamic therapy [23,24] (Figure 1).

In recent decades, a number of methodologies for synthesis of these compounds

have been reported, which include various catalysts, such as [25-51]. Some of these methodologies have limitations such as difficult work-up, toxic and expensive catalysts, low yields, use of strongly acidic conditions, long time reactions and high temperature. As part of our ongoing research program on the development of green methodologies, herein, we report a clean and facile one-pot synthesis of polysubstituted quinolines *via* Friedländer hetero-annulation/condensation [52], 12-aryl-tetrahydrobenzo[α]xanthene-11-ones, 1,8-dioxo-octahydroxanthenes and 14-aryl-14H-dibenzo[α , j]xanthenes in the presence of catalytic amount of tartaric acid under thermal and solvent-free conditions. Tartaric acid as a mild and green

acidic catalyst in organic synthesis has several advantages: it is environmentally friendly, highly efficient and non-toxic. Tartaric acid is commercially available, inexpensive and can be easily handled. The advantages of this acidic catalyst do not end here; it can be further investigated and considered for applications in other types of condensation reactions involving C-C and bond formations. Furthermore, one of the causes of environmental pollution is the usage of organic solvents under reflux conditions and the need for column chromatography to purify the products. In the present work, the products were obtained through simple filtering without the need of column chromatographic separation.



Dye Sensitizers for Photodynamic Therapy

Figure 1. Pharmaceutical active compounds with quinoline and xanthene units.

Results and discussion

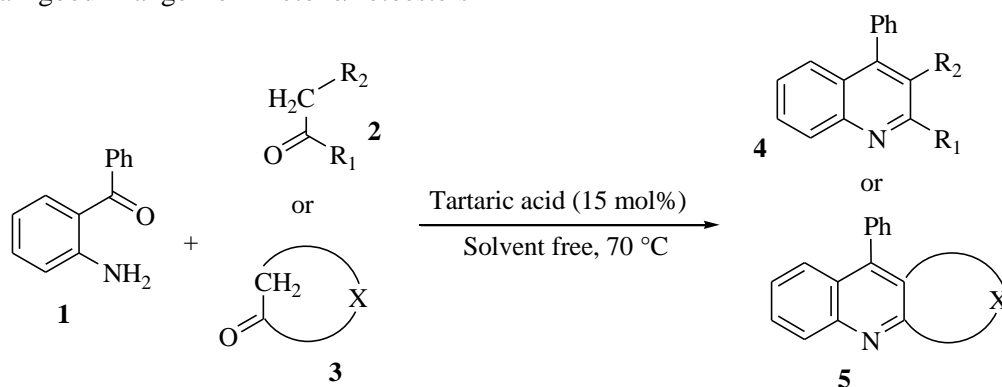
In the pursuit of our continued interest in the development of solvent-free and green synthetic procedures, we decided to explore the use of tartaric acid catalyst for the synthesis of polysubstituted quinolines *via* Friedländer condensation in high to excellent yields at 70°C under solvent-free conditions. Initially, the reaction between 2-aminobenzophenone (1.0 mmol) and dimedone (1.0 mmol), as the

model reaction, was examined in the presence of various amounts of tartaric acid as catalyst and the results are presented in Table 1S (see Supplementary material). The best result was achieved by carrying out the reaction with 15 mol% of catalyst (Table 1S, entry 4). Use of a higher amount of catalyst did not improve the yield, while a decrease in the amount of catalyst decreases the yield (Table 1S). In the absence of catalyst the reaction did not proceed even after a

long reaction time (Table 1S, entry 1). The effect of temperature was also studied, by carrying out the model reaction at different temperatures under solvent-free conditions (room temperature, 40, 60, 70, 80°C) and the best results were obtained at 70°C (Table 1S, entry 4).

To study the generality of this process, a good range of ketone/ketoesters

(**2** or **3**, 1.0 mmol) and 2-aminobenzophenone (**1**, 1.0 mmol) were condensed to the corresponding quinoline derivatives in the presence of catalytic amount of tartaric acid and the related quinoline derivatives were obtained without observation of any by-product in high to excellent yields (Scheme 1, Tables 1 and 2).



Scheme 1. Synthesis of polysubstituted quinolines.

Table 1

Tartaric acid catalyzed synthesis of polysubstituted quinolines (compounds 2 and 4).							
Entry	R_1	R_2	Product	Time, min	Isolated yield, %	M.p., °C	
						This work	Literature
1	CH ₃	COCH ₃	4a	10	92	111-113	113-114 [26]
2	CH ₃	COOCH ₃	4b	15	94	108-110	107-108 [26]
3	CH ₃	COOC ₂ H ₅	4c	15	91	96-98	98-99 [26]

Table 2

Tartaric acid catalyzed synthesis of polysubstituted quinolines (compounds 3 and 5).							
Entry	X	Product	Time, min	Isolated yield, %	M.p., °C		
					This work	Literature	
1	CH ₂ C(CH ₃) ₂ CH ₂ CO	5a	10	94	192-194	192-194 [27]	
2	(CH ₂) ₄	5b	15	89	152-154	153-154 [27]	
3	(CH ₂) ₃	5c	15	87	130-132	129-131 [27]	
4	(CH ₂) ₃ CO	5d	10	93	158-160	157-159 [26]	

In continuation of these studies we were interested in the preparation of 12-aryl-tetrahydrobenzo[α]xanthene-11-ones and the influence of a catalytic amount of tartaric acid on this type of reaction (Scheme 2). To optimize the amount of catalyst and the temperature, the reaction between β -naphthol, benzaldehyde and dimedone was performed, as a model reaction, in the presence of various amounts of catalyst (Table 2S, see Supplementary material). The product yield increased, and the time for reaction completion decreased upon an increase in the amount of tartaric acid up to 15 mol% catalyst and 70°C. Any further increase in the amount of catalyst or temperature did not significantly improve the results. It is important to note that in the absence of catalyst, a trace of product was observed (Table 2S, entry 1). The role of

temperature in the synthesis of 12-(phenyl)-9,9-dimethyl-8,9,10,12-tetrahydrobenzo[α]xanthenes-11-one was investigated in the presence of tartaric acid as the catalyst. The best results were obtained at 70°C in the presence of 15 mol% of catalyst (Table 2S, entry 4). After the optimization of the reaction conditions and in order to show the generality of the method, we used the optimized conditions for the synthesis of different types of 12-aryl-tetrahydrobenzo[α]xanthene-11-ones by using of β -naphthol (**6**, 1.0 mmol), aromatic aldehyde derivatives (**7**, 1.0 mmol) and dimedone (**8**, 1.0 mmol) (Table 3). The reaction was fairly general, clean, rapid, and efficient. The experimental procedure is very simple and the products are obtained in high yields in relatively short reaction times.

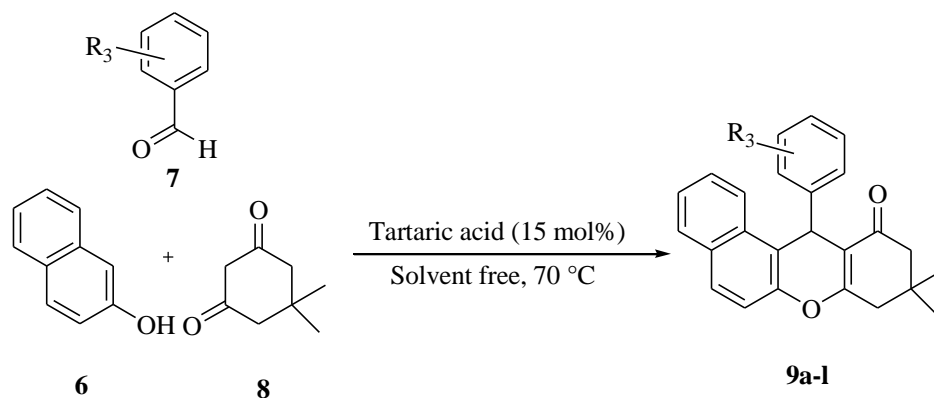
Scheme 2. Synthesis of 12-aryl-tetrahydrobenzo[α]xanthene-11-ones.

Table 3

Tartaric acid catalyzed synthesis of 12-aryl-tetrahydrobenzo[α]xanthene-11-ones.

Entry	R_3	Product	Time, min	Isolated yield, %	M.p., °C	
					This work	Literature
1	H	9a	10	89	149-151	148-150 [28]
2	4-Me	9b	10	91	169-171	171-173 [28]
3	4-OMe	9c	15	89	202-204	202-204 [28]
4	3-OMe	9d	15	88	203-205	204-205 [35]
5	4-OH	9e	20	85	221-223	222-223 [28]
6	4-NO ₂	9f	15	92	174-176	175-178 [40]
7	3-NO ₂	9g	15	93	166-168	167-169 [34]
8	4-Cl	9h	20	87	178-180	176-178 [28]
9	2-Cl	9i	20	89	177-179	179-180 [41]
10	4-Br	9j	20	86	185-187	184-186 [28]
11	3-Br	9k	20	85	160-163	161-164 [40]
12	4-F	9l	10	94	183-185	184-185 [34]

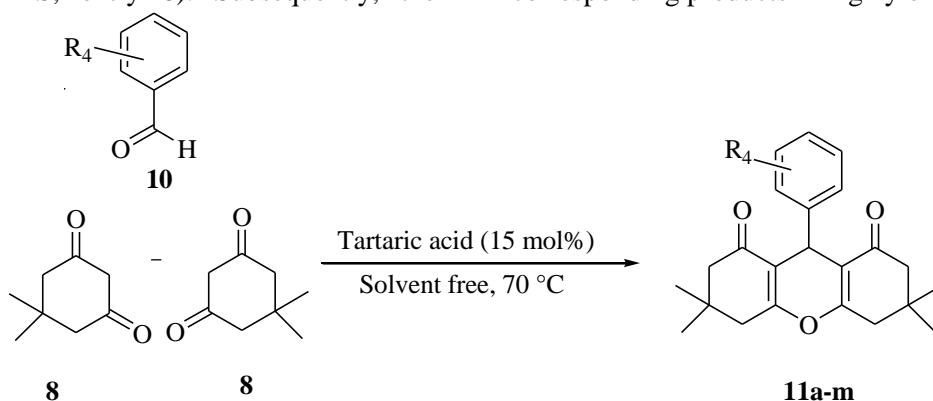
After the successful synthesis of 12-aryl-tetrahydrobenzo[α]xanthene-11-ones, the catalytic activity of uric acid was tested for the reaction of aromatic aldehyde derivatives (**10**, 1.0 mmol) and dimedone (**8**, 2.0 mmol) to give 1,8-dioxo-octahydroxanthenes. For this purpose, the condensation of benzaldehyde (1.0 mmol) with dimedone (2.0 mmol) (Scheme 3) was optimized in terms of the catalyst amount and temperature, under solvent-free conditions; the results are summarized in Table 3S (see Supplementary material). The reasonable results were obtained when 15 mol% of tartaric acid was utilized at 70°C (Table 3S, entry 4). In another study, when the reaction was carried out at 80°C, the product was obtained in 92% yield at 10 min in comparison with 70°C (Table 3S, entry 8). Nevertheless, 70°C was selected as optimal reaction temperature, because one of the aims of this work was performing the reaction in milder reaction conditions with respect to the reported works, and this was more logical (Table 3S). The efficiency and the generality of the catalyst were examined by the reaction of different arylaldehydes (having electron-withdrawing

substituents, electron-donating substituents) with dimedone. The results are shown in Table 4. According to the results presented in Table 4, all reactions were achieved efficiently, and afforded the corresponding 1,8-dioxo-octahydroxanthenes in excellent yields (85-94%), and in short reaction times (10-20 min). Thus, tartaric acid was efficient and general. To show the merit of our catalyst with respect to the reported catalysts for the preparation of 1,8-dioxo-octahydroxanthenes, the results of these catalysts on the reaction of benzaldehyde with dimedone were tabulated in Table 4. As this table indicates, tartaric acid is superior in terms of reaction time, yield or temperature.

Following our work on the development of simple and environmentally friendly synthesis of xanthene derivatives, the treatment of β -naphthol (**6**, 2.0 mmol) and aromatic aldehyde derivatives (**12**, 1.0 mmol) in tartaric acid at 70°C, resulted in the corresponding 14-aryl-14H-dibenzo[α , j]xanthenes (**13a-n**) in high yields (Scheme 4). Using the conversion of β -naphthol and benzaldehyde as a model reaction, we explored different temperatures and loading

amount of catalyst to obtain an optimal condition, as shown in Table 4S (see Supplementary material). Different amounts of tartaric acid were tested, the experimental results indicate that the most effective conversion occurred when a 15 mol% of catalyst was used (Table 4S, entry 4). A trace of product **13a** was detected in tartaric acid at room temperature (Table 4S, Entry 1), and was obtained successfully at higher reaction temperature, reaching a maximum of 89% yield at 80°C (Table 4S, entry 8). Subsequently, the

optimized conditions were applied for the conversion of various kinds of aldehydes into the corresponding 14-aryl-14*H*-dibenzo[*α,j*]xanthenes (**13a-n**) in high yields (Table 5, entries 1–14). The results are summarized in Table 5. It is observed that the process can tolerate both electron-donating and electron-withdrawing substituents in the benzaldehydes. In all cases, the reactions efficiently proceeded at 70°C under solvent-free conditions to afford the corresponding products in high yields.

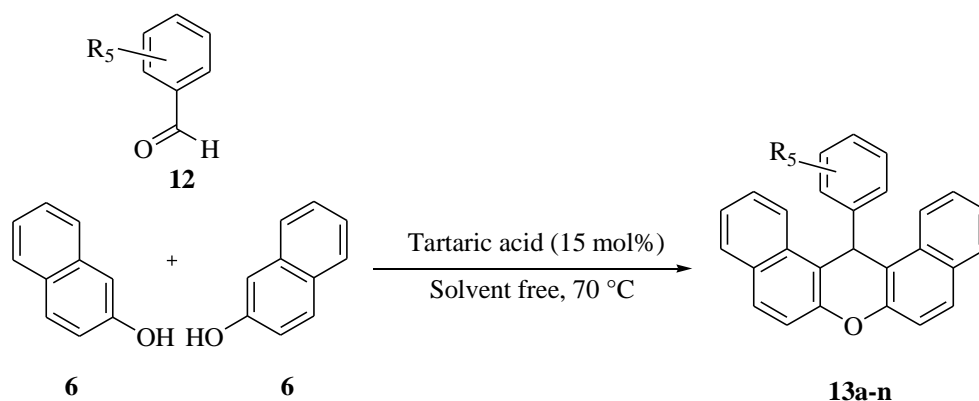


Scheme 3. Synthesis of 1,8-dioxo-octahydroxanthenes.

Table 4

Tartaric acid catalyzed synthesis of 1,8-dioxo-octahydroxanthenes.

Entry	R_4	Product	Time, min	Isolated yield, %	M.p., °C	
					This work	Literature
1	H	11a	10	92	205-207	206-208 [24]
2	4-Me	11b	10	94	218-220	216-218 [43]
3	4-OMe	11c	15	92	240-242	241-243 [34]
4	3,4-(OMe) ₂	11d	15	89	172-174	174-176 [43]
5	3,4,5-(OMe) ₃	11e	15	87	186-188	186-188 [43]
6	4-OH	11f	20	85	245-247	246-248 [43]
7	4-NO ₂	11g	15	91	223-225	222-224 [43]
8	3-NO ₂	11h	15	92	172-174	171-172 [34]
9	4-Cl	11i	20	87	236-238	235-238 [34]
10	2-Cl	11j	20	88	226-228	224-227 [34]
11	4-Br	11k	20	86	238-240	239-241 [34]
12	3-Br	11l	20	85	191-193	192-194 [43]
13	4-F	11m	10	93	195-197	193-195 [27]



Scheme 4. Synthesis of 14-aryl-14*H*-dibenzo[*α,j*]xanthenes.

Proposed mechanistic route for the synthesis of polysubstituted quinolines in the presence of tartaric acid is shown in Scheme 5. Starting from 2-aminobenzophenone (**1**) and carbonyl compounds (**2**), the first pathway involves an initial rate-limiting step, the intramolecular aldol reaction, to afford the aldol product **D**, which gives the enones **B** after loss of water; the following cyclization reaction gives the quinoline **4**.

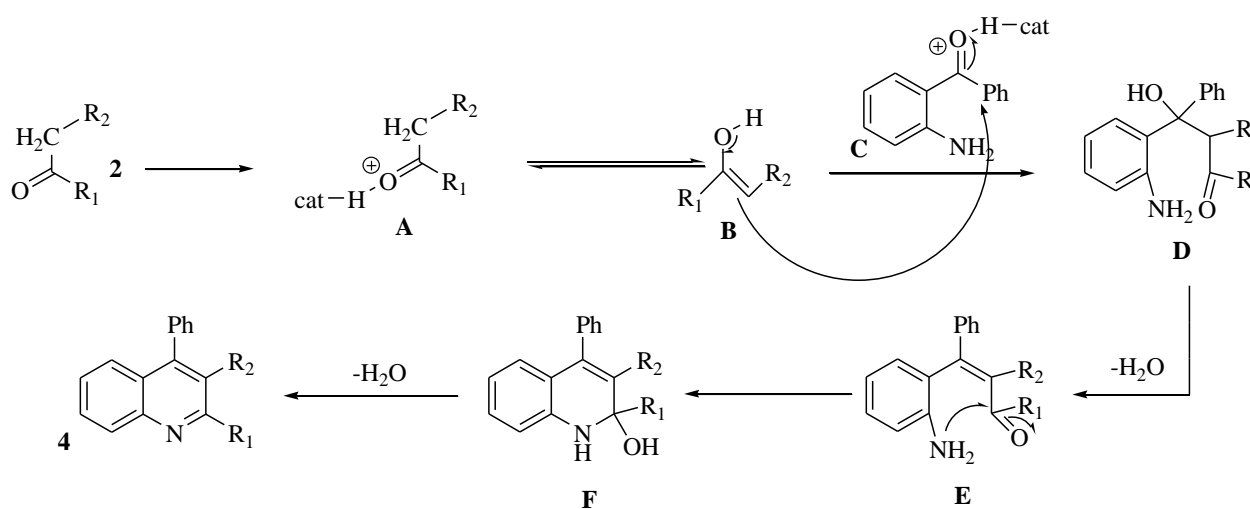
The suggested mechanistic route for the synthesis of 12-aryl-tetrahydrobenzo[*a*]xanthene-11-ones (**9**), 1,8-dioxo-octahydroxanthenes (**11**) and 14-aryl-14*H*-dibenzo[*a,j*]xanthenes (**13**) in the presence of tartaric acid is shown in Scheme 6. On the basis of this mechanism, tartaric acid donates the proton to the oxygen

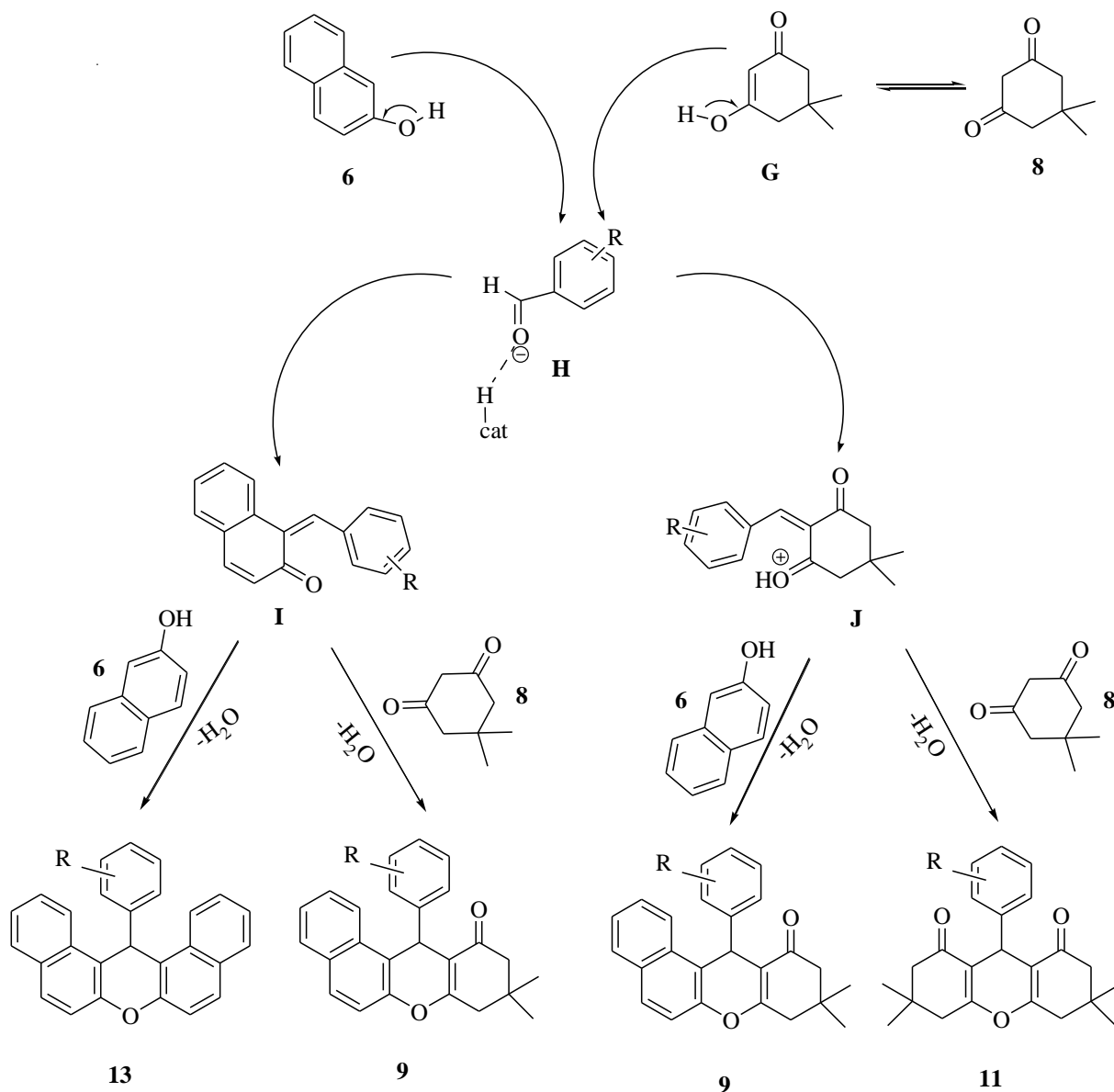
atom of the aldehyde and activates it. Then, nucleophilic β -naphthol (**6**) or dimedone (**8**) attacks the carbonyl group of the activated aldehyde and by removing H₂O, the Knoevenagel products (**D** or **E**) are generated. The following addition of these intermediates to **6** or **8**, gives the acyclic adduct intermediate, which undergoes an intramolecular cyclization with the participation of two hydroxyl groups to afford the xanthene derivatives (Scheme 6).

Comparison of catalytic ability of some catalysts reported in the literature for the synthesis of polysubstituted quinolines (**4** or **5**), 12-aryl-tetrahydrobenzo[*a*]xanthene-11-one (**9**), 1,8-dioxo-octahydroxanthenes (**11**) and 14-aryl-14*H*-dibenzo[*a,j*]xanthenes (**13**) are shown in Tables 6-9.

Table 5

Tartaric acid catalyzed synthesis of 14-aryl-14 <i>H</i> -dibenzo[<i>a,j</i>]xanthenes.						
Entry	R ₅	Product	Time, min	Isolated yield, %	M.p., °C	
					This work	Literature
1	H	13a	15	87	180-182	183-184 [51]
2	4-Me	13b	15	89	226-228	227-228 [28]
3	3-Me	13c	15	91	195-197	197-198 [50]
4	4-OMe	13d	20	87	204-206	204-205 [28]
5	3-OMe	13e	20	88	171-173	172-173 [50]
6	4-OH	13f	25	81	139-141	138-140 [50]
7	4-NO ₂	13g	15	87	306-308	308-309 [50]
8	3-NO ₂	13h	15	89	213-215	212-213 [50]
9	2-NO ₂	13i	15	90	211-213	213-214 [50]
10	4-Cl	13j	25	83	290-292	289-290 [51]
11	2-Cl	13k	25	85	213-215	212-213 [51]
12	4-Br	13l	25	84	295-297	297-298 [28]
13	3-Br	13m	25	86	190-192	191-193 [34]
14	4-F	13n	15	92	241-243	240-242 [29]

Scheme 5. Proposed mechanistic route for the synthesis of polysubstituted quinolines (**4**).



Scheme 6. Proposed mechanistic route for the synthesis of 12-aryl-tetrahydrobenzo[α]xanthene-11-one (9), 1,8-dioxo-octahydroxanthenes (11) and 14-aryl-14H-dibenzo[α, j]xanthenes (13).

Table 6

Comparison of catalytic ability of some catalysts reported in the literature for the synthesis of polysubstituted quinolines*.

Entry	Catalyst	Conditions	Time, min	Yield, %	Reference
1	1,3-disulfonic acid imidazolium hydrogen sulfate	Solvent-free, 70°C	25	89	[27]
2	Rice husk ash supported FeCl ₂ ·2H ₂ O	Solvent-free, 90°C	35	86	[30]
3	P ₂ O ₅ /SiO ₂	Solvent-free, 80°C	35	94	[31]
4	Cellulose sulfuric acid	Solvent-free, 100°C	35	70	[32]
5	Starch sulfuric acid	Solvent-free, 100°C	45	75	[32]
6	Amberlyst-15	EtOH, Reflux	210	72	[33]
7	Tartaric acid	Solvent-free, 70°C	10	94	This work

*Based on reaction of 2-aminobenzophenone (1.0 mmol) and dimedone (1.0 mmol).

Table 7

Comparison of catalytic ability of some catalysts reported in the literature for the synthesis of 12-aryl-tetrahydrobenzo[α]xanthene-11-ones*.

Entry	Catalyst	Conditions	Time, min	Yield, %	Reference
1	1,3-disulfonic acid imidazolium hydrogen sulfate	Solvent-free, 55°C	20	93	[28]
2	Fe ₃ O ₄ -SiO ₂ -SO ₃ H	Solvent-free, 110°C	30	95	[34]
3	NaHSO ₄ /SiO ₂	CH ₂ Cl ₂ , Reflux	300	91	[36]
4	Fe(III) tetranitrophthalocyanine immobilized on activated carbon	EtOH, Reflux	30	91	[37]
5	Ceric ammonium nitrate	Microwave irradiation, 120°C	120	85	[39]
6	Strontium triflate	1,2-Dichloroethane, 80°C	300	85	[42]
7	Tartaric acid	Solvent-free, 70°C	10	89	This work

*Based on the three-component reaction of β -naphthol (1.0 mmol); benzaldehyde (1.0 mmol) and dimedone (1.0 mmol).

Table 8

Comparison of catalytic ability of some catalysts reported in the literature for the synthesis of 1,8-dioxo-octahydroxanthenes*.

Entry	Catalyst	Conditions	Time, min	Yield, %	Reference
1	1,3-disulfonic acid imidazolium hydrogen sulfate	Solvent-free, 55°C	4	95	[28]
2	Fe ₃ O ₄ -SiO ₂ -SO ₃ H	Solvent-free, 110°C	4	94	[34]
3	[cmmim][BF ₄]	Microwave irradiation	2	92	[43]
4	[Hbim]BF ₄	Microwave irradiation	45	85	[45]
5	[BMim][BF ₄]	Mg(BF ₄) ₂ , 80°C	30	97	[46]
6	Tartaric acid	Solvent-free, 70°C	10	92	This work

*Based on the three-component reaction of dimedone (2.0 mmol) and benzaldehyde (1.0 mmol).

Table 9

Comparison of catalytic ability of some catalysts reported in the literature for the synthesis of 14-aryl-14H-dibenzo[α, j]xanthenes*.

Entry	Catalyst	Conditions	Time, min	Yield, %	Reference
1	1,3-disulfonic acid imidazolium hydrogen sulfate	Solvent-free, 90°C	3	94	[28]
2	Fe ₃ O ₄ -SiO ₂ -SO ₃ H	Solvent-free, 110°C	30	94	[34]
3	[BMim][BF ₄]	Mg(BF ₄) ₂ , 80°C	15	95	[46]
4	Sulfonic acid-functionalized phthalimide	Solvent-free, 90°C	30	98	[47]
5	Sulfonic acid functionalized silica	Solvent-free, 125°C	20	98	[48]
6	[H-NMP][HSO ₄]	Solvent-free, 110°C	12	94	[50]
7	Diatomite-SO ₃ H	Solvent-free, 90°C	10	93	[51]
8	Tartaric acid	Solvent-free, 70°C	15	87	This work

*Based on the three-component reaction of β -naphthol (2.0 mmol) and benzaldehyde (1.0 mmol).

Conclusions

In summary, a naturally green, highly efficient and environmentally benign acidic catalyst *i.e.* tartaric acid was developed and exploited for clean, facile and economical one-pot synthesis of polysubstituted quinolines *via* Friedländer hetero-annulation/condensation, 12-aryl-tetrahydrobenzo[α]xanthene-11-ones, 1,8-dioxo-octahydroxanthenes and 14-aryl-14H-dibenzo[α, j]xanthenes from starting

materials under solvent-free conditions. This method gave an insight on the credibility of pathway followed by the aforementioned green catalyst in aiding the heterocyclic compounds formation. Cleaner reaction profile, simple column-free work up condition, shorter reaction times, high to excellent yields, eco-friendly and high catalytic activity make this present procedure an interesting alternative to multistep approaches.

Experimental

Generalities

The melting point of all compounds was determined using an Electro thermal 9100 apparatus. The IR spectra of compounds were determined using a JASCO FTIR 460 Plus spectrometer. Also, ^1H NMR spectra were recorded on a Bruker DRX-400 Avance instruments with CDCl_3 as solvent. In this article, all reagents and solvents were purchased from Merck, Fluka and Acros chemical companies and were used without further purification.

Synthesis of polysubstituted quinolines (4 or 5)

A mixture of 2-aminobenzophenone (**1**, 1.0 mmol), ketone/ketoester (**2** or **3**, 1.0 mmol) and tartaric acid (15 mol%) was heated at 70°C for an appropriate time. After reaction completion (by thin layer chromatography TLC), 3 mL EtOH were added. The mixture was poured into cold water and the resulting precipitate was recrystallized in ethanol to give pure product (**4a-c** and **5a-d**).

Synthesis of 12-aryl-tetrahydrobenzo[a]xanthene-11-ones (9)

A mixture of β -naphthol (**6**, 1.0 mmol), aromatic aldehydes derivatives (**7**, 1.0 mmol), dimedone (**8**, 1.0 mmol) and tartaric acid (15 mol%) was heated at 70°C for an appropriate time. After completion of the reaction by TLC, the mixture was cooled to room temperature and ethanol was added and the precipitated was separated by filtration and the solid was recrystallized from ethanol to give the pure products (**9a-l**).

Synthesis of 1,8-dioxo-octahydroxanthenes (11)

A mixture of dimedone (**8**, 2.0 mmol), aromatic aldehydes derivatives (**10**, 1.0 mmol), and tartaric acid (15 mol%) was heated at 70°C for an appropriate time. After completion of the reaction by TLC, the mixture was cooled to room temperature and ethanol was added and the precipitated was separated by filtration and the solid was recrystallized from ethanol to afford the pure products (**11a-m**).

Synthesis of 14-aryl-14H-dibenzo[a,j]xanthenes (13)

A mixture of β -naphthol (**6**, 2.0 mmol), aromatic aldehydes derivatives (**12**, 1.0 mmol) and tartaric acid (15 mol%) was heated at 70°C for an appropriate time. After completion of the reaction by TLC, the mixture was cooled to room temperature and ethanol was added and the precipitated was separated by filtration and the

solid was recrystallized from ethanol to afford the pure products (**13a-n**).

The products have been characterized by melting points and ^1H NMR spectroscopy. Spectra data of selected and known products are represented below:

3,4-Dihydro-3,3-dimethyl-9-phenylacridine-1(2H)-one (Table 2, entry 1) White solid; Yield: 94%; M.p. $192\text{--}194^\circ\text{C}$; ^1H NMR (400 MHz, CDCl_3): δ 1.19 (6H, s, 2CH_3), 2.59 (2H, s, CH_2), 3.33 (2H, s, CH_2), 7.19–7.22 (2H, m, ArH), 7.45 (1H, t, $J = 8.0$ Hz, ArH), 7.50–7.56 (4H, m, ArH), 7.80 (1H, t, $J = 8.0$ Hz, ArH), 8.13 (1H, brs, ArH).

9,9-dimethyl-12-phenyl-8,9,10,12-tetrahydrobenzo[a]xanthene-11-one (Table 3, entry 1) White solid; Yield: 89%; M.p. $149\text{--}151^\circ\text{C}$; IR (KBr, cm^{-1}): 2950, 1646, 1591, 1462, 1371, 1224, 1024; ^1H NMR (400 MHz, CDCl_3): δ 0.89 (3H, s, CH_3), 1.07 (3H, s, CH_3), 2.14 (1H, d, $J = 16.0$ Hz, CH_2), 2.35 (1H, d, $J = 16.0$ Hz, CH_2), 2.60 (1H, d, $J = 17.6$ Hz, CH_2), 2.70 (1H, d, $J = 17.2$ Hz, CH_2), 5.59 (1H, s, CHAr), 7.04–8.07 (11H, m, ArH).

9,9-dimethyl-12-(4-methylphenyl)-8,9,10,12-tetrahydrobenzo[a]xanthene-11-one (Table 3, entry 2). White solid; Yield: 91%; M.p. $169\text{--}171^\circ\text{C}$; IR (KBr, cm^{-1}): 2949, 1648, 1597, 1371, 1226, 1186, 813. ^1H NMR (400 MHz, CDCl_3): δ 1.01 (3H, s, CH_3), 1.15 (3H, s, CH_3), 2.23 (3H, s, CH_3), 2.28 (1H, d, $J = 16.0$ Hz, CH_2), 2.34 (1H, d, $J = 16.0$ Hz, CH_2), 2.60 (2H, s, CH_2), 5.71 (1H, s, CHAr), 7.00–8.06 (10H, m, ArH).

9,9-dimethyl-12-(4-methoxyphenyl)-8,9,10,12-tetrahydrobenzo[a]xanthene-11-one (Table 3, entry 3) White solid; Yield: 89%; M.p. $202\text{--}204^\circ\text{C}$; IR (KBr, cm^{-1}): 2957, 1647, 1595, 1379, 1226, 1180, 747; ^1H NMR (400 MHz, CDCl_3): δ 1.00 (3H, s, CH_3), 1.15 (3H, s, CH_3), 2.27 (1H, d, $J = 16.4$ Hz, CH_2), 2.34 (1H, d, $J = 16.4$ Hz, CH_2), 2.59 (2H, s, CH_2), 3.72 (3H, s, OCH_3), 5.69 (1H, s, CHAr), 6.72–8.03 (10H, m, ArH).

3,3,6,6-tetramethyl-9-(4-methylphenyl)-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione (Table 4, entry 2) White solid; Yield: 94%; M.p. $218\text{--}220^\circ\text{C}$; IR (KBr, cm^{-1}): 3039, 2958, 1679, 1664, 1467, 1357, 1198, 1137; ^1H NMR (400 MHz, CDCl_3): δ 1.02 (6H, s, 2CH_3), 1.12 (6H, s, 2CH_3), 2.18 (2H, d, $J = 16.2$ Hz, 2CH_2), 2.26 (2H, d, $J = 16.2$ Hz, 2CH_2), 2.26 (3H, s, CH_3), 2.48 (4H, s, 2CH_2), 4.73 (1H, s, CHAr), 7.04 (2H, d, $J = 8.1$ Hz, ArH), 7.20 (2H, d, $J = 8.1$ Hz, ArH).

9-(4-methoxyphenyl)-3,3,6,6-tetramethyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione (Table 4, entry 3) White solid; Yield: 92%;

M.p. 240-242°C; IR (KBr, cm^{-1}): 3028, 2958, 1678, 1665, 1461, 1358, 1260, 1234, 1164; ^1H NMR (400 MHz, CDCl_3): δ 1.02 (6H, s, 2 CH_3), 1.12 (6H, s, 2 CH_3), 2.19 (2H, d, $J = 16.0$ Hz, 2 CH_2), 2.26 (2H, d, $J = 16.4$ Hz, 2 CH_2), 2.48 (4H, s, 2 CH_2), 3.75 (3H, s, OCH_3), 4.72 (1H, s, CHAr), 6.78 (2H, d, $J = 8.8$ Hz, ArH), 7.23 (2H, d, $J = 8.8$ Hz, ArH).

9-(3,4-dimethoxyphenyl)-3,3,6,6-tetramethyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione (Table 4, entry 4) White solid; Yield: 89%; M.p. 172-174°C; IR (KBr, cm^{-1}): 3007, 2957, 1666, 1627, 1465, 1360, 1262, 1228, 1139; ^1H NMR (400 MHz, CDCl_3): δ 1.02 (6H, s, 2 CH_3), 1.12 (6H, s, 2 CH_3), 2.20 (2H, d, $J = 16.2$ Hz, 2 CH_2), 2.27 (2H, d, $J = 16.5$ Hz, 2 CH_2), 2.48 (4H, s, 2 CH_2), 3.81 (3H, s, OCH_3), 3.87 (3H, s, OCH_3), 4.27 (1H, s, CHAr), 6.73 (2H, d, $J = 8.1$ Hz, ArH), 6.78 (2H, d, $J = 8.4$ Hz, ArH), 6.92 (1H, s, ArH).

14-(3-methylphenyl)-14-H-dibenzo[a,j]xanthene (Table 5, entry 3) White solid; Yield: 91%; M.p. 195-197°C; IR (KBr, cm^{-1}): 3043, 2918, 1620, 1592, 1514, 1431, 1252, 1172, 808, 747; ^1H NMR (400 MHz, CDCl_3): δ 2.28 (3H, s, CH_3), 6.48 (1H, s, CHAr), 6.83 (2H, d, $J = 7.2$ Hz, ArH), 7.42-7.63 (8H, m, ArH), 7.81-7.87 (4H, m, ArH), 8.44 (2H, d, $J = 8.4$ Hz, ArH).

14-(4-methoxyphenyl)-14-H-dibenzo[a,j]xanthene (Table 5, entry 4) White solid; Yield: 87%; M.p. 204-206°C; IR (KBr, cm^{-1}): 3072, 2927, 1621, 1592, 1509, 1399, 1250, 1177, 808, 742; ^1H NMR (400 MHz, CDCl_3): δ 3.64 (3H, s, OCH_3), 6.48 (1H, s, CHAr), 6.70 (2H, d, $J = 8.0$ Hz, ArH), 7.39-7.62 (8H, m, ArH), 7.80-7.86 (4H, m, ArH), 8.39 (2H, d, $J = 8.0$ Hz, ArH).

14-(4-hydroxyphenyl)-14-H-dibenzo[a,j]xanthene (Table 5, entry 6) White solid; Yield: 81%; M.p. 139-141°C; IR (KBr, cm^{-1}): 3414, 3056, 2929, 1622, 1593, 1508, 1428, 1250, 1174, 800, 748; ^1H NMR (400 MHz, CDCl_3): δ 6.46 (1H, s, CHAr), 6.61 (2H, d, $J = 8.8$ Hz, ArH), 7.39-7.62 (8H, m, ArH), 7.80-7.87 (4H, m, ArH), 8.39 (2H, d, $J = 8.4$ Hz, ArH).

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Supplementary information

Supplementary data are available free of charge at <http://cjm.asm.md> as PDF file.

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