ICC
Iranian Chemical Communication
Original Research Article
Payame Noor University of Ilam http://icc.journals.pnu.ac.ir

Melamine trisulfonic acid as a highly efficient catalyst for the synthesis of 14-aryl-14H-dibenzo[a,j]xanthenes under solvent-free conditions

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Received: 20 October 2013 , Accepted: 9 November 2013, Published: 1 February 2014

Abstract

Melamine trisulfonic acid (MTSA) is utilized as a highly efficient catalyst for the solvent-free condensation of 2-naphthol with arylaldehydes under conventional thermal or microwave irradiation to give 14-aryl-14*H*-dibenzo[*a,j*]xanthenes in high to excellent yields and in short reaction times.

Keywords: 14-Aryl-14*H*-dibenzo[*a,j*]xanthene, melamine trisulfonic acid (MTSA), 2-naphtol, arylaldehyde, solvent-free, microwave.

Introduction

14-Aryl-14H-dibenzo[a,j]xanthene derivatives are of importance as they have various industrial, pharmaceutical and biological applications. For example, they have been used as antibacterial [1], antiviral [2] and anti-inflammatory agents [3], and in photodynamic therapy [4]. Moreover, benzoxanthenes have been applied in industry as dyes in laser technology [5], and as fluorescent materials for visualization of biomolecules [6]. Many benzoxanthene derivatives are also potent non-peptidic inhibitors of recombinant human calpain I [7], and novel CCR1 receptor antagonists [8]. Therefore, synthesis of xanthenes has attracted the attention of chemists at large. The best method for synthesis of 14-aryl-14H-dibenzo[a,j]xanthenes is the condensation reaction between 2-naphthol and aromatic aldehydes [9-19]. Some catalysts have been employed for this transformation, e.g. $ZrO(OTf)_2$ [9], $H_3PW_{12}O_{40}$ supported MCM-

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41 [10], selectfluor™ [11], $KAl(SO_4)_2.12H_2O$ (alum) [12], nano-TiO_2 [13], cyanuric chloride [14], $[Et_3N\text{-}SO_3H]Cl$ [15], $Sc[N(SO_2C_8F_{17})_2]_3$ [16], $TaCl_5$ [17], Dowex-50W [18] and $Yb(OTf)_3$ [19]. However, most of the reported methods are associated with one or more of the following drawbacks: long reaction time, moderate yield, high catalyst loading, the use of toxic solvents, a need for special apparatus, and poor agreement with the green chemistry protocols. Thus, search for finding a method without possessing the mentioned drawbacks, is still of importance.

In recent years, the use of SO_3H-containing catalysts and reagents has gained considerable attention in organic synthesis; it is so because of their unique properties such as enhanced reactivity as well as selectivity, efficiency, straightforward workup, easy availability of their starting materials, eco-friendly reaction conditions and ability to promote a wide range of reactions [20-23]. They are also non-toxic, non-corrosive and inexpensive. Melamine trisulfonic acid (MTSA) (Figure 1) is certainly one of the most interesting SO_3H-containing catalysts, which has been utilized in some organic transformations [24-28].

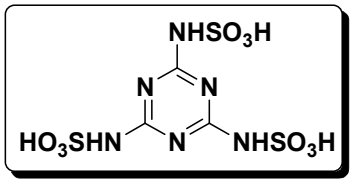


Figure 1. The structure of MTSA

Solvent-free organic reactions have been applied as a useful protocol in organic synthesis [29]. Solvent-free method is an efficient technique for various organic transformations instead of using harmful organic solvents. Performing these reactions under thermal or microwave conditions often leads to a remarkable decrease in reaction times, increased yields, easier workup, matching with the green chemistry protocols, and may enhance the regioselectivity and stereoselectivity of reactions [29-31].

Microwave-assisted organic synthesis has been used as a powerful technique in organic chemistry. Microwave irradiation often leads to remarkable decrease in reaction times, increase in yields, easier work-up, compliance with green chemistry protocols, and may enhance the regio and stereoselectivity of reactions. Moreover, its unique capabilities permits its elaboration in synthesis of compounds which are difficult or impossible to prepare by means of conventional methods [32,33].

Here, we report a highly efficient, clean and simple solvent-free method for the synthesis of 14-aryl-14*H*-dibenzo[*a,j*]xanthenes from 2-naphthol and arylaldehydes using melamine trisulfonic acid (MTSA) under conventional thermal and also microwave conditions (Scheme 1). It is worth noting that our protocol has none of the above-mentioned drawbacks at all.

Experimental

All chemicals were purchased from Merck or Fluka Chemical Companies. All known compounds were identified by comparison of their melting points and spectral data with those reported in the literature. Progress of the reactions was monitored by TLC using silica gel SIL G/UV 254 plates. The reactions (in microwave conditions) were performed using a domestic microwave oven model LG-5607KR. The ^{1}H NMR (400 or 300 MHz) and ^{13}C NMR (100 or 75 MHz) were run on a Bruker Avance DPX, FT-NMR spectrometer (δ in ppm). Melting points were recorded on a Büchi B-545 ap- paratus in open capillary tubes.

General procedure for the synthesis of 14-aryl-14*H*-dibenzo[*a,j*]xanthene derivatives under conventional thermal conditions

A mixture of 2-naphthol (0.288 g, 2 mmol) and arylaldehyde (1 mmol) was heated and stirred at 110 °C in a test tube and, then, MTSA (0.009 g, 2.5 mol%) was added to it. At first, the resulting mixture was stirred magnetically at 110 ºC, and after solidification of the reaction mixture and also at the same temperature, it was vigorously stirred with a small rod. After completion of the reaction, as monitored by TLC, the reaction mixture was cooled to room temperature, and the resulting solid was re-crystallized from EtOH (95%) to give the pure product.

General procedure for the synthesis of 14-aryl-14*H*-dibenzo[*a,j*]xanthenes under-microwave conditions

A mixture of 2-naphthol (0.289 g, 2 mmol), arylaldehyde (1 mmol) and MTSA (0.009 g, 2.5 0.002 g, 0.5 mol%) was irradiated in a domestic microwave oven in a test tube at 540 W for 1 min-intervals. After completion of the reaction, as monitored by TLC, the reaction mixture was cooled to room temperature, and recrystallized from EtOH (95%) to give the pure product.

Selected spectral data of the synthesized 14-aryl-14*H*-dibenzo[*a*,*j*]xanthenes

14-Phenyl-14*H*-dibenzo[*a*,*j*]xanthene (1): ^{1}H NMR (400 MHz, DMSO-d$_6$): δ 6.74 (s, 1H), 6.97 (t, *J*= 7.6 Hz, 1H), 7.14 (t, *J*= 7.6 Hz, 2H), 7.45 (t, *J*= 7.2 Hz, 2H), 7.57 (d, *J*= 8.8 Hz, 2H), 7.62-7.66 (m, 4H), 7.91-7.93 (m, 4H), 8.70 (d, *J*= 8.8, 2H); ^{13}C NMR (100 MHz, DMSO-d$_6$): δ 37.0, 117.9, 118.2, 123.9, 124.9, 126.7, 127.4, 128.4, 128.8, 129.1, 129.5, 131.1, 131.4, 146.0, 148.5.

14-(4-Nitrophenyl)-14*H* dibenzo[*a*,*j*]xanthene(2)

^{1}H NMR (300 MHz, DMSO-d$_6$): δ 6.95 (s, 1H), 7.43 (t, *J*= 7.2 Hz, 3H), 7.58-7.65 (m, 4H), 7.81 (d, *J*= 7.5 Hz, 1H), 7.90-7.95 (m, 4H), 8.14 (d, *J*= 7.8, 1H), 8.45 (s, 1H), 8.72 (d, *J*= 8.4 Hz, 2H); ^{13}C NMR (75 MHz, DMSO-d$_6$): δ 36.4, 116.9, 118.2, 122.0, 123.6, 125.2, 127.7, 129.1, 130.0, 130.4, 131.11, 134.7, 147.9, 148.3, 148.6.

14-(3-Nitrophenyl)-14*H*-dibenzo[*a*,*j*]xanthene (3)

^{1}H NMR (300 MHz, DMSO-d$_6$):δ 6.91 (s, 1H), 7.11-7.25 (m, 1H), 7.43-7.48 (m, 2H), 7.55-7.71 (m, 4H), 7.88-8.03 (m, 7H), 8.66 (d, *J*= 8.4 Hz, 2H); ^{13}C NMR (75 MHz, DMSO-d$_6$):δ 36.7, 116.6, 118.2, 120.1, 123.6, 124.2, 125.2, 127.6, 129.1, 129.5, 130.0, 131.1, 131.2, 134.2, 146.3, 148.4, 153.1.

14-(2-Nitrophenyl)-14*H*-dibenzo[*a*,*j*]xanthene (4)

^{1}H NMR (300 MHz, DMSO-d$_6$): δ 6.94 (s, 1H),7.41-7.46 (m, 3H), 7.56-7.65 (m, 4H), 7.79 (d, *J*= 2.1, 1H), 7.89-7.94 (m, 4H), 8.13 (d, *J*= 7.8, 1H), 8.45 (s, 1H), 8.70 (d, *J*= 6, 2H); ^{13}C NMR (75 MHz, DMSO-d$_6$): δ 36.4, 116.9, 118.1, 122.0, 122.5, 123.6, 125.2, 127.7, 129.1, 130.0, 130.4, 131.1, 134.7, 138.2, 147.9, 148.3, 148.6.

14-(4-Chlorophenyl)-14*H*-dibenzo[*a*,*j*]xanthene (5)

^{1}H NMR (300 MHz, DMSO-d$_6$): δ 6.76 (s, 1H), 7.18 (d, *J*= 6.8 Hz, 2H), 7.46-7.64 (m, 10H), 8.92 (d, *J*= 7.79 Hz, 2H), 8.66 (d, *J*= 7.6 Hz, 2H); ^{13}C NMR (75 MHz, DMSO-d$_6$): δ 36.3, 117.4, 118.1, 123.4, 123.7, 125.0, 127.4, 128.8, 129.1, 129.6, 130.1, 131.1, 131.2, 144.8, 148.4.

14-(3-Chlorophenyl)-14*H*-dibenzo[*a*,*j*]xanthene (6)

^{1}H NMR (300 MHz, DMSO-d$_6$): δ 6.74 (s, 1H), 7.01 (d, *J*= 8.1, 1H), 7.13 (t, *J*= 7.8, 1H), 7.42 (t, *J*= 7.2, 2H), 7.52-7.66 (m, 6H), 7.91 (d, *J*= 8.7, 4H), 8.67 (d, *J*= 8.7, 2H); ^{13}C NMR (75 MHz, DMSO-d$_6$): δ 36.5, 117.2,

118.1, 123.7, 125.1, 126.8, 127.0, 127.5, 127.9, 129.1, 129.7, 130.7, 131.1, 131.2, 133.5, 148.2, 148.5.

14-(2-Chlorophenyl)-14*H*-dibenzo[*a*,*j*]xanthene (7)

^{1}H NMR (300 MHz, DMSO-d_6): δ 6.64 (s, 1H), 6.91-7.03 (m, 2H), 7.27 (d, *J*= 7.7 Hz, 2H), 7.38-7.50 (m, 5H), 7.57-7.70 (m, 2H), 7.76-7.90 (m, 4H), 8.54 (d, *J*= 8.4 Hz, 1H); ^{13}C NMR (75 MHz, DMSO-d_6): δ 34.8, 116.9, 118.2, 123.3, 124.9, 127.4, 128.5, 128.8, 129.2, 129.8, 130.2, 130.3, 130.9, 131.4, 132.0, 143.2, 148.7.

14-(3-Bromophenyl)-14*H*-dibenzo[*a*,*j*]xanthene (8)

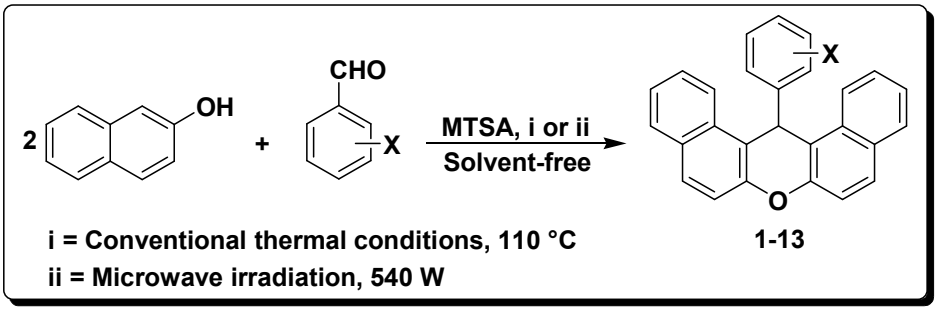
^{1}H NMR (300 MHz, DMSO-d_6): δ 6.63 (s, 1H),6.90-7.02 (m, 2H), 7.26 (d, *J*= 7.7 Hz, 2H), 7.37-7.49 (m, 5H), 7.58 (t, *J*= 7.9 Hz, 2H), 7.85-7.89 (m, 4H), 8.53 (d, *J*= 8.5 Hz, 1H); ^{13}C NMR (75MHz, DMSO-d_6): δ 34.8, 116.9, 118.2, 123.3, 124.9, 127.4, 128.5, 128.8, 129.1, 129.8, 130.2, 130.3, 130.9, 131.4, 132.0, 143.2, 148.6.

14-(4-Methylphenyl)-14*H*-dibenzo[*a*,*j*]xanthene (11)

^{1}H NMR (300 MHz, DMSO-d_6): δ 2.01 (s, 3H), 6.63 (s, 1H), 6.90 (d, *J*= 7.2, 2H), 7.40-7.61 (m, 8H), 7.88 (t, *J*= 2.7, 4H), 8.61 (d, *J*= 8.7, 2H); ^{13}C NMR (75 MHz, DMSO-d_6): δ 20.8, 37.4, 117.9, 118.1, 123.8, 124.9, 127.3, 128.2, 129.0, 129.3, 129.3, 131.1, 131.3, 135.8, 143.0, 148.3.

14-(4-Methoxyphenyl)-14*H*-dibenzo[*a*,*j*]xanthene (12)

^{1}H NMR (300 MHz, DMSO-d_6): δ 3.52 (s, 3H), 6.63-6.67 (m, 3H), 7.19-7.62 (m, 8H), 7.87-7.92 (m, 4H), 8.64 (d, *J*= 8.7, 2H); ^{13}C NMR (75 MHz, DMSO-d_6): δ 36.0, 55.3, 114.1, 118.1, 123.9, 124.9, 127.9, 124.9, 127.3, 129.2, 131.1, 135.7, 138.1, 147.0, 149.4, 157.9.



Scheme 1. The preparation of 14-aryl-14H-dibenzo[a,j]xanthenes using MTSA

Results and discussion

To optimize the reaction conditions under conventional thermal conditions, at first, the condensation of 2-naphthol (2 mmol) with 3-nitrobenzaldehyde (1 mmol) was selected as a model reaction to provide the desired 14-aryl-14*H*-dibenzo[*a*,*j*]xanthene (Scheme 1), and it was examined in the presence of 5 mol% of MTSA at range of 90-120 ºC in the absence of solvent (Table 1). As Table 1 indicates, the reasonable results were observed when the reaction was performed at 110 °C. The Increment of the temperature up to 120 °C didn't significantly improve the reaction results.

Table 1. Effect of temperature on the condensation of 2-naphthol with 3-nitrobenzaldehyde

Entry	Temp. (°C)	Time (min)	Yield[a](%)
1	90	50	94
2	100	25	96
3	110	14	98
4	120	13	98

[a]Yield of isolated product.

In another study, the reaction of 2-naphthol (2 mmol) with 3-nitrobenzaldehyde (1 mmol) was tested in the presence of different molar ratios of MTSA at 110 ºC under solvent-free conditions (Table 2). As it is shown in Table 2, 2.5 mol% of the catalyst was sufficient to promote the reaction efficiently at 110 °C. After optimization of the reaction conditions, the efficiency and generality of the method were evaluated by the reaction of 2-naphthol with arylaldehydes bearingelectron withdrawing substituents, electron-donating substituents or halogens on their aromatic rings. The results are summarized in Table 3. As it can be seen in Table 3, the method was general and efficient; all reactions were performed successfully to furnish the corresponding 14-aryl-14*H*-dibenzo[*a*,*j*]xanthenes in high to excellent yields and in relatively short reaction times.

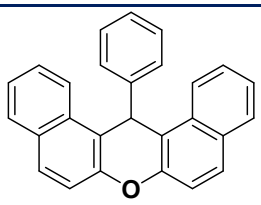
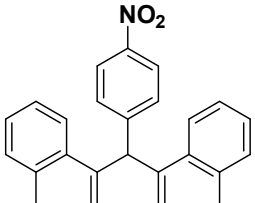
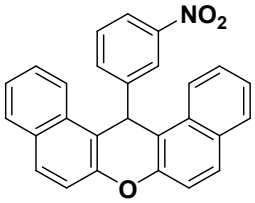
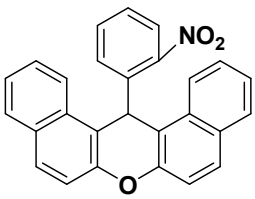
Table 2. Effect of the catalyst amount on the condensation of 2-naphthol with 3-nitrobenzaldehyde

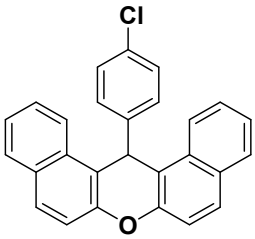
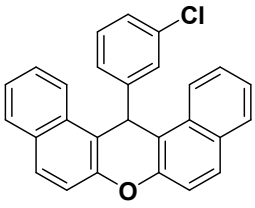
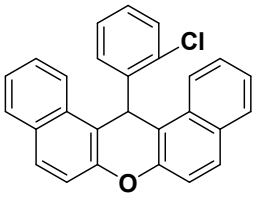
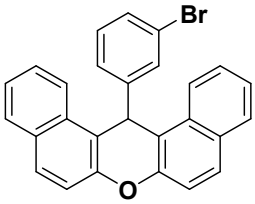
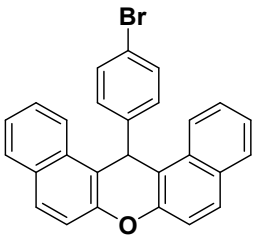
Entry	Mol% of MTSA	Time (min)	Yield[a] (%)

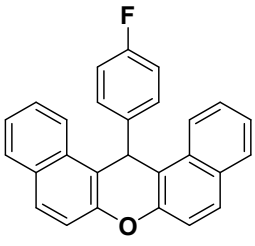
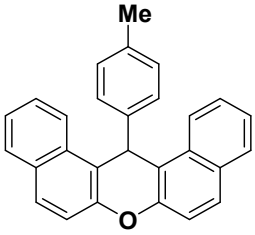
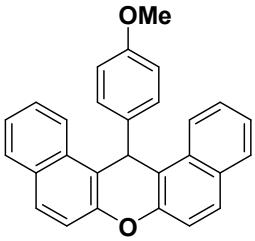
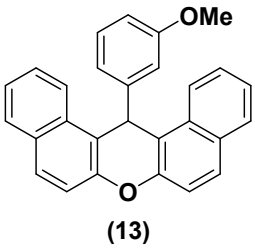
1	-	120	9
2	1	27	95
3	2.5	16	98
4	5	16	98

[a]Yield of isolated product.

Table 3. The MSTA-catalyzed condensation of 2-naphthol with arylaldehydes leading to 14-aryl-14*H*-dibenzo[*a*,*j*]xanthenes under thermal and solvent-free conditions (110 °C)

Product	Time (min)	Yield[a] (%)	M.p. °C (Lit.)
 **(1)**	16	98	186-188 (186-188) [15]
 **(2)**	19	97	314-316 (312-314) [15]
 **(3)**	16	98	211-213(215-217) [15]
 **(4)**	23	98	214-216 (212-214) [15]

 (5)	12	98	292-294 (289-290) [12]
 (6)	16	98	206-208 (207-209) [15]
 (7)	18	98	212-214 (209-211) [15]
 (8)	14	97	189-191 (186-188) [15]
 (9)	11	98	299-301 (296-298) [12]

 **(10)**	16	98	238-240 (238) [11]
 **(11)**	12	96	226-228 (225-227) [15]
 **(12)**	40	90	202-204 (203-205) [12]
 **(13)**	22	97	167-169 (164-165) [35]

[a]Yield of isolated product.

Considering the significant importance of microwave irradiation to promote organic reactions, we decided to apply this energy for thesynthesis of 14-aryl-14*H*-dibenzo[*a*,*j*]xanthenes using MTSA. In order to reach the aim, a set of experiments varying microwave power and amount of the catalyst were carried out on the condensation of 2-naphthol with 4-bromobenzaldehyde under solvent-free conditions (Scheme 1).

First of all, the condensation of 2-naphthol with 4-bromobenzaldehyde was examined at different microwave powers (180-540 W) in the presence of 2.5 mol% of MSTA. The results are displayed in Table 4. As Table 4 shows, higher yield and shorter reaction time were obtained at 540 W.

Table 4. Effect of different microwave powers on the reaction in the presence of 2.5 mol% of MSTA

Entry	MW Power (W)	Time (min)	Yield[a] (%)
1	180	15	20
2	360	12	96
3	540	4	98

[a]Yield of isolated product.

To optimize the reaction conditions relative to the catalyst amount, the solvent-free condensation of 2-naphthol with 4-bromobenzaldehyde was tested in the presence of different molar ratios of MSTA at 540 W (Table 5). As it can be seen in Table 5, 0.5 mol% of MSTA was sufficient to catalyze the reaction efficiently. Increasing the catalyst amount up to 2.5 mol% had not significant influence on the reaction.

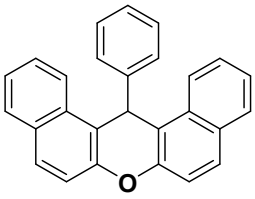
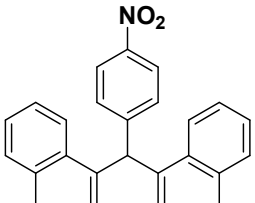
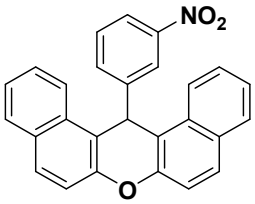
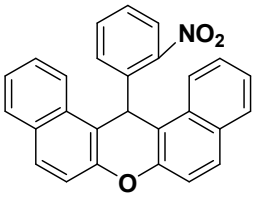
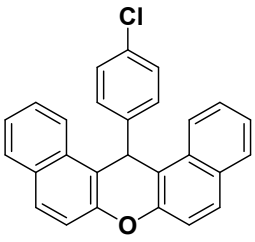
Table 5. Effect of the catalyst amount on the condensation of 2-naphthol with 4-bromobenzaldehyde under micro-wave irradiation (540 W)

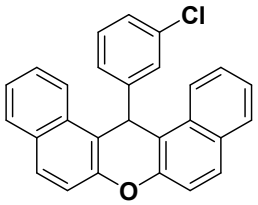
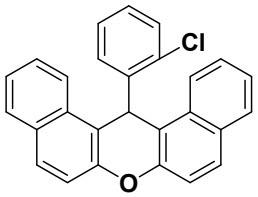
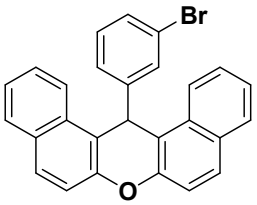
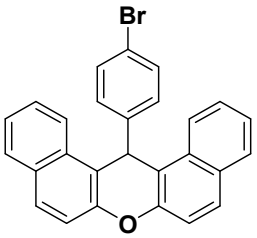
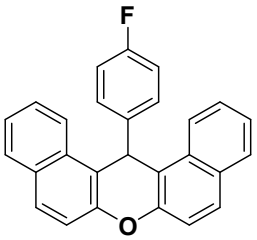
Entry	Mol% of MTSA	Time (min)	Yield[a] (%)
1	-	20	12
2	0.5	5	97
3	1	5	97
4	2.5	4	98

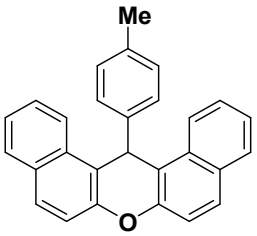
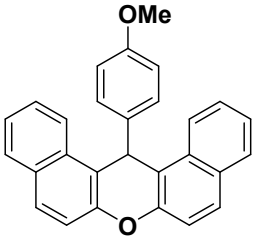
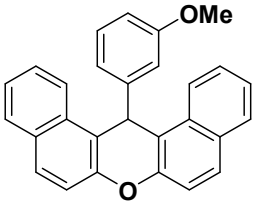
[a]Yield of isolated product.

Under the optimal reaction conditions, 2-naphthol was condensed with different aromatic aldehydes (including benzaldehyde and arylaldehydes bearing electron-withdrawing substituents, electron-releasing substituents or halogens on their aromatic ring) to afford the desired products in high yields and in short reaction times.

Table 6. The solvent-free synthesis of 14-aryl-14*H*-dibenzo[*a,j*]xanthenes using 0.5 mol% of MSTA at 540 W of microwave power

Product	Time (min)	Yield[a] (%)
 **(1)**	3	98[b]
 **(2)**	10	93[b]
 **(3)**	8	94
 **(4)**	3	98[b]
 **(5)**	4	98

 (6)	5	96
 (7)	5	97[b]
 (8)	6	97
 (9)	5	97
 (10)	7	96

 **(11)**	5	98
 **(12)**	20	88[b]
 **(13)**	5	96[b]

[a]Yield of isolated product.

[b]This product was produced using 2.5 mol% of the catalyst.

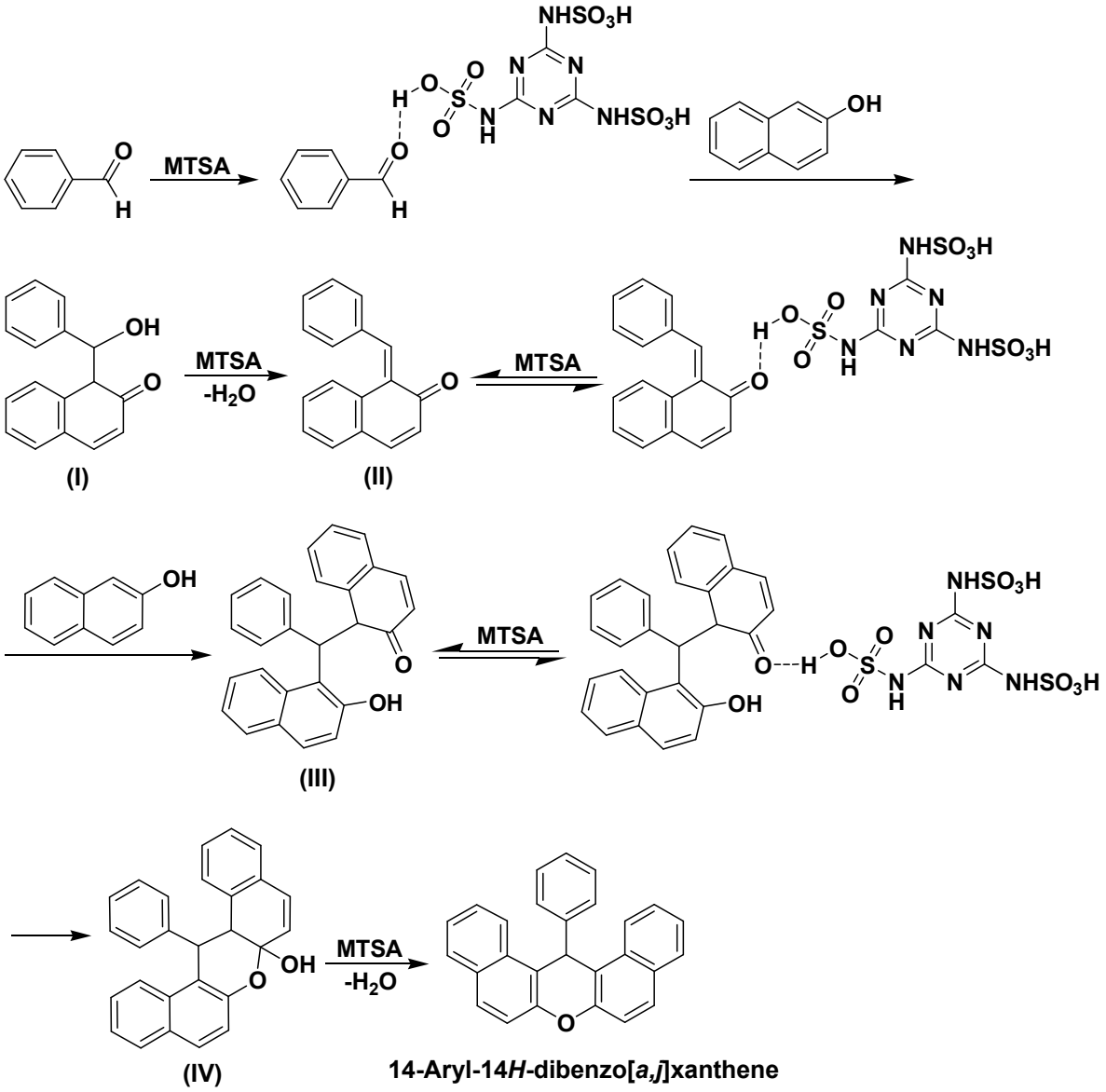
A plausible mechanism is suggested for the reac- tion (Scheme 2), which supported by the literature [36,37].

Conclusion

In sum, we have developed a new solvent-free protocol for the synthesis of 14-aryl-14H-dibenzo [a,j]xanthenes using MTSA under conventional thermal and also microwave conditions. The advantages of this method are, as it was already mentioned, its efficiency, generality, high yield, short reaction time, cleaner reaction profile, simplicity, ease of product isolation, and compliance with the green chemistry protocols.

Acknowledgements

The authors thank Payame Noor University of Bushehr for the financial support of this work.



Scheme 2. The proposed reaction mechanism

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