Asian Journal of Chemistry; Vol. 25, No. 4 (2013), 1981-1984

ASIAN JOURNAL OF CHEMISTRY

http://dx.doi.org/10.14233/ajchem.2013.13260

One-Pot Multi Component Synthesis of Xanthenediones and Acridinediones at Room Temperature

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(*Received*: 15 December 2011; *Accepted*: 10 October 2012) AJC-12262

A facile and efficient protocol for the synthesis of xanthenedione and acridinrdione derivatives has been developed *via* the one-pot condensation reaction between dimedone and aldehydes (for the synthesis of xanthenediones) and dimedone, aldehydes and primary amines (for the synthesis of acridinediones) in presence of the formic acid as solvent at room temperature. All the products were obtained in good to excellent yields and all reactions were completed in short times.

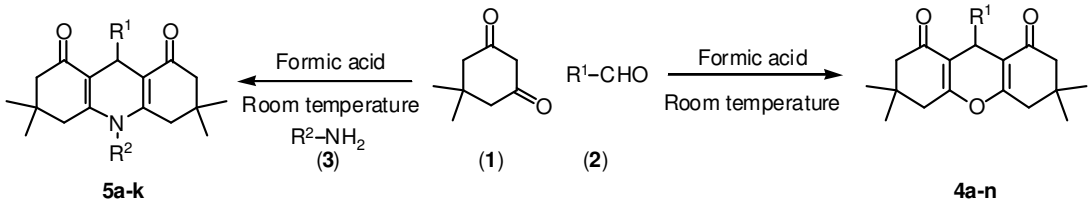
Key Words: Xanthenedione, Acridinediones, Formic acid, Multi component reactions.

INTRODUCTION

Multi component reactions (MCRs) provide unmatched opportunities for the expeditious increase of complexity and diversity in synthetic outcomes. The strategy offers significant advantages over classical stepwise approaches, allowing the formation of several bonds and the construction of complex molecular architectures from simple precursors in a single synthetic operation without the need for isolation of intermediates[1]. One-pot multi component synthesis of xanthenediones and acridinediones are two important examples of the application of multi component reactions for the synthesis of biologically active compounds.

Xanthene-based compounds are important because of their use in medicine as they possess antimicrobial activities[2]. These compounds have also been investigated for agricultural bactericidal activity[2a], photo dynamic therapy, antiinflammatory effects[3] and antiviral activity[4]. In particular, octahydroxanthene constitutes a structural unit in several natural products[5] and they are valuable synthons because of the inherent reactivity of the in built pyran ring[6]. A number of xanthene-based compounds are also available from natural sources. Santalin pigments are popularly known, have been isolated from a number of plant species[7]. The wide-ranging biological activities associated with xanthenes, both naturally occurring and synthetic, ensure that the synthesis of these compounds remains a topic of current interest. Moreover, acridinediones and their derivatives are poly functionalized 1,4-dihydropyridine derivatives. In recent years, 1,4-dihydropyridines and their derivatives have attracted strong interest for the treatment of cardiovascular diseases, such as angina pectoris[8] and hypertension[9]. Acridine derivatives have been used to synthesize labeled conjugates with medicinals, peptides, proteins and nucleic acids[10-12] that exhibit antitumor and DNA-binding properties. There are several reports in the literature for the synthesis of xanthenediones and acridinediones include $InCl_3{\cdot}4H_2O$ in ionic liquid[13], solid-state condensation by grinding at room temperature[14], diammonium hydrogen phosphate[15], *p*-dodecylbenzenesulfonic acid in water[16], Fe^{3+}-montmorilonite[17], $NaHSO_4$-SiO_2 or silica chloride[18], amberlyst-15[19], silica sulfuric acid[20], tetrabutyl ammonium hydrogen sulphate[21], trimethylsilylchloride[22], 1-butyl-3-methylimidazolium hydrogen sulphate[23], montmorillonite K-10-supported[24] and covalently anchored sulfonic acid on silica gel[25]. Each of these methods have their own advantages but also some of them often suffer from one or more disadvantages such as prolonged reaction time, tedious work-up processes, low yield[25], expensive reagents[26] and hazardous organic solvents[25]. Moreover most of these reported methods were applied at reflux temperature. It is well known that for many chemical processes, a major adverse effect to environment is the consumption of energy for heating and cooling. So design of methods for the synthesis of organic compounds at room temperature is a useful strategy to overcome this problem.

Considering the above facts herein we report a convenient and efficient method for one-pot multi component synthesis



Scheme-I: One-pot multi component synthesis of xanthenediones and acridinediones at room temperature

of xanthenedione and acridinedione derivatives at room temperature (**Scheme-I**).

EXPERIMENTAL

All chemicals were purchased from Merck or Fluka Chemical Companies. All synthesized compounds are known and were identified by comparison of their melting points and ^{1}H NMR data with those in the authentic samples. The ^{1}H NMR (500 MHz) and ^{13}C NMR (125 MHz) were run on a Bruker Avance DPX-250, FT-NMR spectrometer. Melting points were recorded on a Stuart Scientific Apparatus SMP3 (UK) in open capillary tubes.

General procedure for the synthesis of xanthenedione and/or acridinedione derivatives: In a typical example, dimedone (2 mmol) and aromatic aldehyde (1 mmol) were added [in the case of the synthesis of acridinedione primary amine (1 mmol) was also added] in a 25 mL round-bottomed flask contained formic acid (1 mL) and the resulting mixture was stirred magnetically at room temperature. The completion of the reaction was followed by TLC using *n*-hexane/ethyl acetate 3:1 as an eluent. After completion, water (10 mL) was added and insoluble products were separated by simple filtration and recrystallized from ethanol for more purification.

3,4,6,7-Tetrahydro-3,3,6,6-tetramethyl-9-phenyl-2*H*-xanthene-1,8-(5*H*,9*H*)-dione (4a): ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 1.02 (s, 6H), 1.14 (s, 6H), 2.18 (d, 2H, *J* = 16.0 Hz), 2.25 (d, 2H, *J* = 16.0 Hz), 2.51 (s, 4H), 4.77 (s, 1H), 7.12 (t, 1H, *J* = 7.0 Hz), 7.25 (t, 2H, *J* = 7.5 Hz), 7.31 (d, 2H, *J* = 7.6 Hz). ^{13}C NMR ($CDCl_3$, 125 MHz), δ: 27.6, 29.5, 32.2, 32.6, 41.3, 51.1, 116.0, 126.7, 128.5, 128.8, 144.5, 162.7, 196.6.

3,4,6,7-Tetrahydro-3,3,6,6-tetramethyl-9-(4-chlorophenyl)-2*H*-xanthene-1,8-(5*H*,9*H*)-dione (4b): ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 1.02 (s, 6H), 1.15 (s, 6H), 2.21 (d, 2H, *J* = 16.3 Hz), 2.29 (d, 2H, *J* = 16.3 Hz), 2.51 (s, 4H), 4.77 (s, 1H), 7.21 (d, 2H, *J* = 8.5 Hz), 7.26 (d, 2H, *J* = 8.5 Hz). ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 27.7, 29.7, 31.7, 32.6, 41.2, 51.2, 115.6, 128.6, 130.1, 132.5, 143.0, 162.8, 196.7.

3,4,6,7-Tetrahydro-3,3,6,6-tetramethyl-9-*p*-tolyl-2*H*-xanthene-1,8-(5*H*,9*H*)-dione (4f): ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 0.97 (s, 6H), 1.07 (s, 6H), 2.14 (d, 2H, *J* = 16.3 Hz), 2.22-2.24 (m, 5H), 2.45 (s, 4H), 4.72 (s, 1H), 7.02 (d, 2H, *J* = 8.0 Hz), 7.17 (d, 2H, *J* = 8.0 Hz). ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 21.3, 27.9, 29.7, 31.8, 32.7, 41.3, 51.2, 116.2, 128.6, 129.2, 136.1, 141.6, 162.4, 196.7.

3,4,6,7-Tetrahydro-3,3,6,6-tetramethyl-9-(pyridin-3-yl)-2*H*-xanthene-1,8(5*H*,9*H*)-dione (4m): ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 1.02 (s, 6H), 1.12 (s, 6H), 2.17 (d, 2H, *J* = 16.3 Hz), 2.26 (d, 2H, *J* = 16.3 Hz), 2.51 (s, 4H), 4.77 (s, 1H), 7.16-7.18 (m, 1H), 7.72-7.75 (dt, 1H, J_1 = 7.8 Hz, J_2 = 1.9 Hz), 8.37 (dd, 1H, J_1 = 4.7 Hz, J_2 = 1.5 Hz), 8.46 (d, 1H, *J* = 1.9 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 27.8, 29.6, 32.6, 41.2, 51.0, 115.1, 123.4, 136.9, 140.0, 148.1 149.9, 163.3, 196.8.

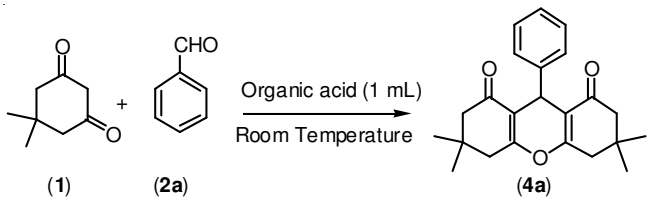
9-(4-Chlorophenyl)-3,4,6,7-tetrahydro-3,3,6,6-tetramethyl-10-*p*-tolylacridine-1,8-(2*H*,5*H*,9*H*,10*H*)-dione (5d): ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 0.83 (s, 6H), 0.99 (s, 6H), 1.88 (d, 2H, *J* = 17.5 Hz), 2.08-2.14 (m, 4H), 2.21 (d, 2H, *J* = 16.2 Hz), 2.55 (s, 3H), 5.25 (s, 1H), 7.12 (d, 2H, *J* = 8.0 Hz), 7.23 (d, 2H, *J* = 8.0 Hz), 7.36-7.42 (m, 4H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 21.6, 27.1, 30.1, 32.9, 32.9, 42.1, 50.6, 114.7, 128.5, 129.2, 129.7, 130.0, 131.6, 136.6, 140.2, 145.3, 150.6, 196.1.

3-(1,2,3,4,5,6,7,8-Octahydro-3,3,6,6-tetramethyl-9-(3-nitrophenyl)-1,8-dioxo-acridin-10(9*H*)-yl)benzonitrile (5h): ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 0.85 (s, 6H), 1.00 (s, 6H), 1.82 (d, 2H, *J* = 17.5 Hz), 2.14 (d, 2H, *J* = 17.5 Hz), 2.18 (d, 2H, *J* = 16.4 Hz), 2.28 (d, 2H, *J* = 16.4 Hz), 5.37 (s, 1H), 7.45 (t, 1H, *J* = 7.8 Hz), 7.63-7.68 (m, 2H), 7.84 (t, 1H, *J* = 7.8 Hz), 7.91-7.95 (m, 2H), 8.01 (dd, 1H, J_1 = 8.0 Hz, J_2 = 1.4 Hz), 8.21 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 27.1, 30.1, 33.1, 33.2, 42.5, 50.6, 114.9, 117.5, 121.7, 122.6, 129.7, 133.2, 135.2, 140.7, 148.2, 148.8, 149.0, 196.01.

3,4,6,7-Tetrahydro-3,3,6,6-tetramethyl-9-(4-(methylthio)-phenyl)-10-*p*-tolyl-acridine-1,8-(2*H*,5*H*,9*H*,10*H*)-dione (5k): ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 0.88 (s, 6H), 0.99 (s, 6H), 1.85 (d, 2H, *J* = 17.5 Hz), 2.12 (d, 2H, *J* = 17.5 Hz), 2.17 (d, 2H, *J* = 16.5 Hz), 2.25 (d, 2H, *J* = 16.5 Hz), 2.44 (s, 3H), 2.55 (s, 3H), 5.25 (s, 1H), 7.10 (d, 2H, *J* = 7.5 Hz), 7.19 (d, 2H, *J* = 8.0 Hz), 7.36-7.40 (m, 4H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 16.3, 21.6, 27.2, 30.1, 32.7, 32.9, 42.1, 50.6, 114.7, 127.3, 128.8, 135.4, 136.8, 139.9, 144.0, 150.8, 196.2.

RESULTS AND DISCUSSION

At first the one-pot condensation reaction between dimedone (**1**) (2 mmol) and benzaldehyde (**2a**) (1 mmol) was selected as a model reaction (**Scheme-II**). In order to find the best reaction conditions, we examined the model reaction in the presence of various organic acids at room temperature and the obtained results are summarized in Table-1.



Scheme-II: One-pot condensation reaction between dimedone (**1**) (2 mmol) and benzaldehyde (**2a**) (1 mmol) in the presence of some organic acids at room temperature

TABLE-1
ONE-POT CONDENSATION REACTION BETWEEN DIMEDONE (2 mmol) AND BENZALDEHYDE (1 mmol) IN THE PRESENCE OF SOME ORGANIC ACIDS (1 mL) AT ROOM TEMPERATURE

Entry	Catalyst	Time (h)	Yield (%)[a]
1	Formic acid	2	90
2	Acetic acid	5	75
3	Propanoic acid	8	69
4	Butanoic acid	8	60
5	Pantanoic acid	8	55

[a]Isolated yield.

As it is clear from Table-1, the best results were obtained in the presence of formic acid at room temperature. These observations demonstrate that there are a direct relationship between the acid strength of the applied organic acid and the rate of the one-pot condensation reaction between dimedone and benzaldehyde. As it is clear from Table-1, as the acid strength decreased, the reaction time was increased and the yield was decreased obviously.

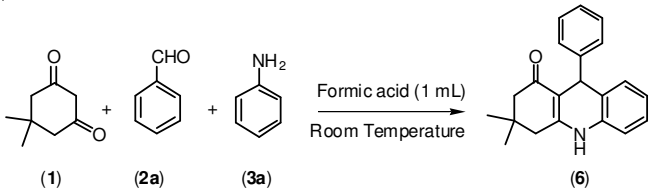
In the next step, a broad range of structurally diverse aldehydes were condensed with dimedone to furnish the corresponding products in high yield and the obtained results are presented in Table-2. We investigated further the electronic effect of different substituents present on the aldehyde component. As it is shown in Table-2, electron withdrawing substituents in aromatic ring of aryl-alkyl ketones accelerated the reaction rate (entries 10, 11, 12) whereas electron releasing substituents reduced the reaction rate (entries 7, 8, 9) but nature of substituents is not affected the yield of the reaction. So a wide range of aldehydes having both electron-donating and electron withdrawing groups were equally facile for the reaction, resulting in the formation of xanthenedione derivatives in very good yields. An important feature of this method is that the heterocyclic functionality present in the molecule remains unaffected. This fact was amply demonstrated by the reaction of pyridine-3-caboxaldehyde with dimedone, which gave 9-(pyridine-3-yl)-1,8-dioxo-octahydroxanthene (**4m**) in excellent yield.

In the next step, we decided to apply other method for the synthesis of acridinedione derivatives. For this purpose, the one-pot multi component condensation reaction between dimedone (**1**), aromatic aldehydes (**2**) and primary amines (**3**) were investigated in presence of the formic acid at room temperature and obtained results are summarized in Table-3.

As it is clear from Table-3, here also the aromatic aldehydes containing both electron-donating and electron-withdrawing groups afforded the products in high yields. Moreover it is obviously showed that various aniline derivatives reacted smoothly under the reaction conditions.

The experimental procedure is remarkably simple. After the completion of the reaction, water was added to the reaction mixture and the insoluble crude products were isolated by simple filtration and recrystalized from ethanol for more purification. To the best of our knowledge, this is the first report on the synthesis of acridinedione derivatives at room temperature and all of the last reported procedures were applied under reflux conditions.

In another study we examined the one-pot three component condensation reaction between dimedone (**1**) (1 mmol), benzaldehyde (**2a**) (1 mmol) and aniline (**3a**) (1 mmol) in the presence of formic acid at room temperature to afford compound (**6**) and unfortunately only a mixture of unknown products were obtained even after a long time (24 h) (**Scheme-III**).



(**1**) (**2a**) (**3a**) (**6**)

Scheme-III: Oone-pot three component condensation reaction between dimedone (**1**) (1 mmol), benzaldehyde (**2a**) (1 mmol) and aniline (**3a**) (1 mmol) in the presence of formic acid (1 mL) at room temperature

In order to assess the capability of the present method with respect to the reported methods for the preparation of titled compounds, the synthesis of compound **5a** was compared with the reported methods (Table-4). As it is clear from Table-4, the present method is more efficient.

TABLE-2
ONE-POT MULTI COMPONENT SYNTHESIS OF XANTHENEDIONE DERIVATIVES IN THE PRESENCE OF FORMIC ACID AT ROOM TEMPERATURE

Entry	R^1	Compound	Time (h)	Yield (%)[a]	m.p. (ºC)	
					Founded	Reported [Ref.]
1	C_6H_5	**4a**	2.0	90	201-202	203-204[26]
2	4-Cl-C_6H_4	**4b**	2.0	91	231-232	230-232[26]
3	3-Cl-C_6H_4	**4c**	2.0	91	186-187	184-186[26]
4	2-Cl-C_6H_4	**4d**	3.0	90	227-228	225-227[26]
5	4-Br-C_6H_4	**4e**	2.5	93	240-242	240-241[26]
6	4-CH_3-C_6H_4	**4f**	3.0	90	213-216	215-217[26]
7	4-CH_3O-C_6H_4	**4g**	5.0	89	243-245	242-244[26]
8	4-OH-C_6H_4	**4h**	6.0	85	250-252	249-251[17]
9	3-OH-C_6H_4	**4i**	5.0	90	224-226	225-226[17]
10	4-NO_2-C_6H_4	**4j**	1.0	92	220-221	222-223[26]
11	3-NO_2-C_6H_4	**4k**	1.0	91	172-174	170-172[26]
12	2-NO_2-C_6H_4	**4l**	1.0	93	250-252	252-254[26]
13	3-pyridine	**4m**	3.0	92	185-187	184-186[26]
14	C_6H_5-C_2H_2	**4n**	4.0	90	173-175	174-176[26]

[a]Isolated yields.

TABLE-3
ONE-POT MULTI COMPONENT SYNTHESIS OF XANTHENEDIONE DERIVATIVES IN THE PRESENCE OF FORMIC ACID AT ROOM TEMPERATURE

Entry	R^1	R^2	Compound	Time (h)	Yield (%)[a]	m.p. (ºC)	
						Founded	Reported [Ref.]
1	C_6H_5	C_6H_5	**5a**	3	91	255-257	254-256[27]
2	4-CH_3-C_6H_4	4-CH_3-C_6H_4	**5b**	3	90	296-298	297-299[28]
3	3-NO_2-C_6H_4	4-CH_3-C_6H_4	**5c**	2	91	283-285	284-285[28]
4	4-Cl-C_6H_4	4-CH_3-C_6H_4	**5d**	3	90	273-275	273-275[26]
5	4-Cl-C_6H_4	3-OH-C_6H_4	**5e**	2	92	266-268	267-269[26]
6	4-NO_2-C_6H_4	3-OH-C_6H_4	**5f**	2	92	>300	> 300[26]
7	3-NO_2-C_6H_4	4-CH_3-C_6H_4	**5g**	2	91	288-290	289-291[26]
8	3-NO_2-C_6H_4	3-CN-C_6H_4	**5h**	3.5	90	267-269	26-268[26]
9	3-OH-C_6H_4	3-CN-C_6H_4	**5i**	4	90	>300	> 300[26]
10	3-CN-C_6H_4	4-CH_3-C_6H_4	**5j**	3	92	256-258	256-257[26]
11	4-CH_3S-C_6H_4	4-CH_3-C_6H_4	**5k**	3	91	237-138	239[26]

[a]Isolated yields.

TABLE-4
COMPARATIVE THE SYNTHESIS OF COMPOUND **5a** USING THE REPORTED METHODS VERSUS THE PRESENT METHOD

Entry	Reagents and conditions	Time (min)	Yield (%)	Ref.
1	Proline (10 mol %), EtOH/H_2O 5:1, reflux	360	79	28
2	Amberlyst-15 (200 mg), CH_3CN, reflux	300	81	18
3	[Hmim]TFA (0.1 g), 80 ºC	270	86	29
4	Silica-bonded S-sulphunic acid (30 mg), EtOH, reflux	120	94	26
5	Formic acid (1 mL), room temperature	30	95	This work

[a]Isolated yield.

Conclusion

We have developed an efficient method for the synthesis of xanthenedione and acridinedione derivatives in high yields in the presence of formic acid at room temperature. The mild reaction conditions and simplicity of the procedure offers improvements over many existing methods.

ACKNOWLEDGEMENTS

The authors are thankful to the Research Council of Islamic Azad University, Shiraz branch for the financial support of this work.

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