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SOLVENT-FREE SYNTHESIS OF 1,8-DIOXO-OCTAHYDROXANTHENES AND 1,8-DIOXO-DECAHYDROACRIDINES USING [BPY]HSO_4 AS AN EFFICIENT REUSABLE CATALYST

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ABSTRACT
An efficient synthesis of 1,8-dioxo-octahydroxanthenes and 1,8-dioxo-decahydroacridines using [BPy]HSO_4 as catalyst under thermal, solvent-free conditions is described. This new approach has advantages such as short reaction time, high yields, cleaner reaction profiles, simple work-up, and reusable catalyst.

KEYWORDS
[BPy]HSO_4, 1,8-dioxo-octahydroxanthenes, 1,8-dioxo-decahydroacridines, aromatic aldehydes, 5,5-dimethyl-1,3-cyclohexanedione,solvent-free conditions

INTRODUCTION

Xanthenes have been used as dyes[i], and pH-sensitive fluorescent materials for the visualization of biomolecular assemblies[ii]. Furthermore, these compounds are an important family of organic compounds because they have wide range of biological and pharmaceutical properties such as anti-inflammatory[iii], anti-depressants and antimalarial agents[iv]. A typical method for preparation of 1,8-dioxo-octahydroxanthenes involves condensation of aromatic aldehydeswith5,5-dimethyl-1,3-cyclohexanedione in the presence of catalysts such as$ZnCl_2$-choline chloride[v], ZnO-acetyl chloride[vi], Amberlyst-15[vii], $ZrOCl_2 \bullet 8H_2O$[viii], trichloroisocyanuricacid[ix], silica sulfuric acid[x], nanosized MCM-41-SO_3H[xi], Fe_3O_4nanoparticles[xii],[bmim][HSO_4][xiii-xiv] [Hmim]TFA[xv], [TMPSA]HSO_4[xvi], [DDPA][HSO_4][xvii], [Et_3N-SO_3H]Cl[xviii]and[Hbim]BF_4[xix]as catalysts. Rahmati reported that the synthesis of xanthenedione derivatives could be proceeded in ionic liquid tetramethylguanidiniumtrifluoroacetate catalyzed by trifluoroacetateacid[xx]. Fan et al and Ma et al reported that the synthesis of xanthenedione derivatives could be proceeded in ionic liquid [bmim]BF_4 catalyzed by $NaHSO_4$[xxi].

It has been reported that acridine-1,8-diones can be used as laser dyes with very high-lasing efficiencies[xxii-xxiii]. In addition, acridine derivatives have attracted considerable attention in medicinal chemistry due to their highly biological and physiological activities such as anti-cancer [xxiv], cytotoxic[xxv], anti-multidrug-resistant[xxvi] and antimicrobial[xxvii].Some methods are available in the literature for the synthesis of 1,8-dioxo-decahydroacridines from aldehyde, dimedone and ammonium acetate in the

presence of several catalysts such asHY-zeolite[xxviii],1-n-butyl-3-methylimidazolium bromide[xxix],$Zn(OAc)_2$, NH_4Cl[xxx], L-proline[xxx-xxxi],triethylbenzylammonium chloride[xxxii],carbon-based solid acid[xxxiii],MCM-41-SO_3H[xxxiv],n-butylpyridiumtetrafluoroborate, n-butylimidazoliumtetrafluoroborate, n-butylimidazoliumhexafluorophosphate[xxxv],tetrabutylammoniumhexatungstate[xxxvi],salicylic acid[xxxvii]and indium(III) chloride[xxxviii].

Unfortunately, many of the synthetic protocols for 1,8-dioxo-octahydroxanthenes and 1,8-dioxo-decahydroacridines reported above suffer from one or more disadvantages, such as low yields, prolonged reaction time, use of hazardous and often expensive acid catalysts, use of organic solvents, tedious work-up procedures, the requirement of special apparatus, and difficulty in recovery and reusability of the catalysts. Therefore, the development of simple, efficient, high-yielding and environmentally friendly methods using new catalysts for the synthesis of these compounds would be highly desirable.

Acidic ionic liquid $[BPy]HSO_4$ has been reported to be an efficient, and environmentally benign catalyst for synthesis of 1,5-benzodiazepines[xxxix].This ionic liquid wasalso employed as phase-transfer catalyst for phase-transfer catalytic oxidation of dibenzothiophene[xl].Herein, we report a simple, efficient and environmentally benign procedure for synthesis of 1,8-dioxo-octahydroxanthenes and 1,8-dioxo-decahydroacridinesunder solvent-free conditions using$[BPy]HSO_4$ as reusable catalyst.

EXPERIMENTAL

Melting points were determined on an X-4 micro melting point apparatus and are uncorrected. FT-IR spectra were obtained as KBr pellets on a Nexus 470 spectrophotometer. ^{1}H NMR spectra were recorded on a Bruker AvanceIII400 with TMS as internal standard. All chemicals were commercial products. $[BPy]HSO_4$ was prepared according to the literature method[xxxix].

General procedure for the synthesis of 1,8-dioxo-octahydroxanthenes

To a mixture of aldehyde (2.5 mmol) and 5,5-dimethyl-1,3-cyclohexanedione (5 mmol), $[BPy]HSO_4$ (0.25 mmol) was added and the mixture was heated on an oil bath at 100°C with good stirring. The progress of the reaction was monitored by thin layer chromatography (ethyl acetate/hexane=1/3 as eluent). After completion of the reaction, the obtained solid was recrystallized from 95% ethanol to afford the corresponding 1,8-dioxo-octahydroxanthenes. All products obtained are known compounds and were identified by comparing of their physical and spectra data with the reported ones.

General procedure for the synthesis of 1,8-dioxo-decahydroacridines

A mixture of aromatic aldehyde (3 mmol), 5,5-dimethyl-1,3-cyclohexanedione (6 mmol), ammonium acetate (3.6 mmol) and $[BPy]HSO_4$ (10 mol%) was stirred at 100°C. The progress of the reaction was monitored by thin-layer chromatography(ethyl acetate/petroleum ether=1/2 as eluent). After completion of reaction, the reaction mixture was cooled to room temperature. The resulting solid was recrystallized from ethanol (80%) to get the corresponding 1,8-dioxo-decahydroacridines. All products obtained are known compounds and were identified by comparing of their physical and spectra data with the reported ones. The spectral data of some representative products are given below.

3,3,6,6-Tetramethyl-9-(phenyl)-1,8-dioxo-decahydroacridine (Table 3, entry 1)

IR (KBr): 3282, 3064, 2958, 1638, 1603, 1479, 1368, 1217, 1141 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz) δ:0.97 (s, 6H, $2CH_3$), 1.08 (s, 6H, $2CH_3$), 2.14-2.34(m, 8H, $4CH_2$), 5.11 (s, 1H, CH), 7.08 (t, J=7.6 Hz, 1H, ArH), 7.20 (t, J=7.6 Hz, 2H, ArH), 7.35 (d, J=8.0 Hz, 2H, ArH), 7.54 (s, 1H, NH).

3,3,6,6-Tetramethyl-9-(4-methylphenyl)-1,8-dioxo-decahydroacridine (Table 3, entry 2)

IR (KBr): 3183, 3067, 2958, 1651, 1607, 1494, 1366, 1221, 1149 cm^{-1}. ^{1}H NMR ($CDCl_3$, 400 MHz) δ:0.95

(s, 6H, $2CH_3$), 1.05 (s, 6H, $2CH_3$), 2.12-2.31(m, 11H, $4CH_2$, CH_3), 5.05 (s, 1H, CH), 6.99 (d, *J*=8.0 Hz, 2H, ArH), 7.22 (d, *J*=8.0 Hz, 2H, ArH), 7.88 (s, 1H, NH).

3,3,6,6-Tetramethyl-9-(4-chlorophenyl)-1,8-dioxo-decahydroacridine (Table 3, entry 9)

IR (KBr): 3176, 3059, 2956, 1647, 1611, 1490, 1365, 1221, 1148 cm^{-1}. ^1H NMR ($CDCl_3$, 400 MHz) δ: 0.95 (s, 6H, $2CH_3$), 1.07 (s, 6H, $2CH_3$),2.13-2.33 (m, 8H, $4CH_2$), 5.05 (s, 1H, CH), 7.15 (d, *J*=8.0 Hz, 2H, ArH), 7.27 (d, *J*=8.0 Hz, 2H, ArH), 7.51 (s, 1H, NH).

3,3,6,6-Tetramethyl-9-(2,4-dichlorophenyl)-1,8-dioxo-decahydroacridine (Table 3, entry 10)

IR (KBr): 3322, 3063, 2961, 1641, 1609, 1473, 1363, 1222, 1142 cm^{-1}. ^1H NMR ($CDCl_3$, 400 MHz) δ: 0.94 (s, 6H, $2CH_3$), 1.05 (s, 6H, $2CH_3$), 2.11-2.37 (m, 8H, $4CH_2$), 5.26 (s, 1H, CH), 5.89 (s, 1H, NH), 7.12 (d, *J*=8.4 Hz, 1H, ArH), 7.23 (s, 1H, ArH), 7.47 (d, *J*=8.4 Hz, 1H, ArH).

3,3,6,6-Tetramethyl-9-(4-hydroxyphenyl)-1,8-dioxo-decahydroacridine (Table 3, entry 12)

IR (KBr): 3283, 3066, 2957, 1636, 1616, 1480, 1367, 1219, 1143 cm^{-1}. ^1H NMR ($CDCl_3$, 400 MHz) δ: 0.95 (s, 6H, $2CH_3$), 1.07 (s, 6H, $2CH_3$), 2.13-2.35 (m, 8H, $4CH_2$), 5.08 (s, 1H, CH), 7.08 (t, *J*=7.6 Hz, 1H), 7.14 (s, 1H), 7.20 (t, *J*=7.6 Hz, 2H, ArH), 7.35 (d, *J*=7.2 Hz, 2H, ArH).

RESULTS AND DISCUSSION

In order to find optimum reaction conditions, the condensation of benzaldehyde with 5,5-dimethyl-1,3-cyclohexanedione in the presence of catalytic amount of $[BPy]HSO_4$ under solvent-free conditions was examined as a model reaction. As can be seen from Table 1, the reaction was completed within 10 min to furnish the desired 1,8-dioxo-octahydroxanthene in 90% yield at 100°C in the presence of 10 mol%$[BPy]HSO_4$. Further increase in temperature did not increase the yield and also did not improve the reaction rates. It was also observed that the yield of 1,8-dioxo-octahydroxanthene decreased to 87% when the reaction was carried out at 90 °C. Furthermore, the effect of the amount of catalyst was examined. The optimum catalyst loading for $[BPy]HSO_4$ was found to be about 10 mol%. Larger amount of the catalyst did not improve the yield while decreasing the amount of catalystdecreased the yield.

Table 1 Effect of different reaction conditions on $[BPy][HSO_4]$ catalyzed synthesis of 1, 8-dioxo-octahydroxanthenes[a]

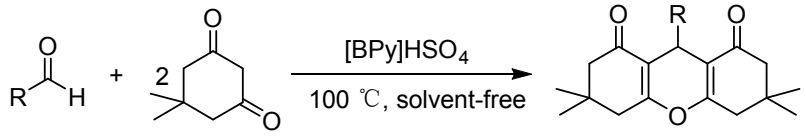
Entry	Temperature (°C)	Catalyst (mol%)	Time (min)	Yield [b] (%)
1	80	10	10	77
2	80	0	15h	41[xiv]
3	90	10	10	87
4	100	5	10	86
5	100	10	10	90
6	100	15	10	91
7	110	10	10	90

[a]Reaction condition: benzaldehyde (5 mmol), 5,5-dimethyl-1,3-cyclohexanedione (10mmol).

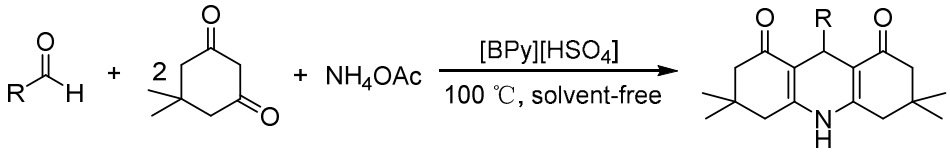
[b] Isolated yield.

With these results in hand, we investigated one-pot synthesis of 1, 8-dioxo-octahydroxanthenes by condensation of a variety of aromatic aldehydes with5,5-dimethyl-1,3-cyclohexanedione at 100°C in the presence of 10 mol% of $[BPy]HSO_4$ under solvent-free conditions(Scheme 1). The results are summarized in Table 2. Aromatic aldehydes with electron-withdrawing or electron-donating substituents could react very well with 5,5-dimethyl-1,3-cyclohexanedione in short time and high to excellent yields were achieved.The same reaction conditions were applied for the synthesis of 1,8-dioxo-decahydroacridines via

the one-pot, three-component condensation of 5,5-dimethyl-1,3-cyclohexanedione, an aromatic aldehyde, and ammonium acetate (Scheme 2). Aromatic aldehydes bearing electron-donating and electron-withdrawing substituents undergo this reaction to furnish the products with high yields (Table 3).



Scheme 1. $[BPy]HSO_4$ catalyzed synthesis of 1,8-dioxo-octahydroxanthenes



Scheme 2. $[BPy]HSO_4$ catalyzed synthesis of 1,8-dioxo-decahydroacridines

Table 2 $[BPy]HSO_4$ catalyzed synthesis of 1,8-dioxo-octahydroxanthenes[a]

Entry	R	Time (min)	Yield [b] (%)	mp (°C)	mp (°C) [Lit.]
1	C_6H_5	10	90	205-206	203-204[xiv]
2	4-$CH_3C_6H_4$	20	93	208-209	208-209[xxi]
3	4-$CH_3OC_6H_4$	20	97	243-244	242-244[xiv]
4	2-ClC_6H_4	45	86	223-224	223-225[xviii]
5	4-FC_6H_4	50	85	226-228	226-227[viii]
6	3-$CH_3OC_6H_4$	30	92	182-184	177-180[ix]
7	4-BrC_6H_4	50	89	240-242	240-241[xiv]
8	3-$NO_2C_6H_4$	30	88	170-171	170-172[xiv]
9	4-HO-3-$MeOC_6H_3$	30	90	226-228	226-228[xxi]
10	4-ClC_6H_4	20	96	230-232	230-232[xiv]
11	2,4-$Cl_2C_6H_3$	20	87	250-252	248-251[viii]
12	4-$NO_2C_6H_4$	20	90	225-226	225-227[ix]
13	2-HOC_6H_4	40	86	204-205	202-205[ix]
14	4-HOC_6H_4	20	89	247-249	248-250[xxi]
15	C_6H_4CH=CH	15	83	174-175	173-174[viii]

[a]Reaction condition: aromatic aldehyde (2.5 mmol), 5,5-dimethyl-1,3-cyclohexanedione (5 mmol), $[BPy]HSO_4$ (0.25 mmol) at 100 °Cunder solvent-free conditions.

[b] Isolated yield.

From an environmental and an economic point of view, it is highly desirable that the catalyst can be recovered and reused. We also investigated the recycling of the catalyst under solvent-free conditions. After completion of the reaction,the reaction mixture was cooled to room temperature and the resulting solid was recrystallized by using aqueous ethanol to afford the pure product. The filtrate containing the ionic liquid was then evaporated to dryness under reduced pressure and the resulting catalyst was reused directly for the next run. The reactions using the recycled catalyst were conducted in a similar manner. As shown in Table 4, the recovered catalyst can be reused at least five additional times in subsequent reactions without any loss in catalytic activity. This indicates that $[BPy]HSO_4$ as a catalyst for the preparation of

1,8-dioxo-octahydroxanthenes and 1,8-dioxo-decahydroacridines is reusable.

Table 3 $[BPy]HSO_4$ catalyzed synthesis of 1,8-dioxo-decahydroacridines[a]

Entry	R	Time (min)	Yield [b] (%)	mp (°C)	mp (°C) [Lit.]
1	C_6H_5	15	87	269-271	258-260[xli]
2	4-$CH_3C_6H_4$	20	88	>300	>300[xxix]
3	4-$CH_3OC_6H_4$	25	92	272-274	272-274[xxix]
4	2-ClC_6H_4	15	89	218-220	217-219[xxviii]
5	4-FC_6H_4	20	92	217-219	215-218[xxviii]
6	4-BrC_6H_4	25	84	240-242	241-243[xxx]
7	3-$NO_2C_6H_4$	30	83	284-286	285-286[xlii]
8	4-HO-3-$MeOC_6H_3$	20	91	>300	>300[xxxii]
9	4-ClC_6H_4	25	91	227-229	228-229[xxviii]
10	2,4-$Cl_2C_6H_3$	30	97	>310	>300[xxix]
11	4-$(CH_3)_2NC_6H_4$	15	89	265-267	264-266[xliii]
12	4-HOC_6H_4	15	89	>300	>300[xxxii]
13	2-$NO_2C_6H_4$	40	82	281-283	281-282[xxviii]
14	4-$NO_2C_6H_4$	30	81	281-283	282-283[xxviii]

[a]Reaction condition: aromatic aldehyde (3 mmol), 5,5-dimethyl-1,3-cyclohexanedione (6 mmol), ammonium acetate (3.6 mmol), $[BPy]HSO_4$ (0.3 mmol) at 100 °Cunder solvent-free conditions.
[b] Isolated yield.

Table 4 Recycling of the $[BPy][HSO_4]$catalyst

Run	1,8-dioxo-octahydroxanthenes [a]		1,8-dioxo-decahydroacridines [b]	
	Time (min)	Yield [c] (%)	Time (min)	Yield [c] (%)
1	10	90	15	89
2	10	90	15	93
3	10	91	15	92
4	10	91	15	90
5	10	91	15	88
6	10	90	15	89

[a]Reaction condition: benzaldehyde (5 mmol), 5,5-dimethyl-1,3-cyclohexanedione (10mmol), $[BPy][HSO_4]$ (0.5 mmol) at 100 °Cunder solvent-free conditions.
[b]Reaction condition: 4-hydroxybenzaldehyde (3 mmol), 5,5-dimethyl-1,3-cyclohexanedione (6 mmol), ammonium acetate (3.6 mmol), $[BPy][HSO_4]$ (0.3 mmol) at 100 °Cunder solvent-free conditions.
[c] Isolated yield.

The catalytic efficiency of $[BPy]HSO_4$ was compared with some other reported ionic liquid for the synthesis of 1,8-dioxo-octahydroxanthenes (Table 5) and 1,8-dioxo-decahydroacridines (Table 6). The results proved that $[BPy]HSO_4$ is an efficient catalyst in terms of product yield and reaction times.

Table 5 Comparison of ionic liquids used as catalysts for the synthesis of 1, 8-dioxo-octahydroxanthenes [a]

Entry	Catalyst	Conditions	Time (min)	Yield (%)	Reference
1	[Bmim][HSO_4]	Solvent-free, 100 °C	25	93	xiii
2	[Bmim][HSO_4]	Solvent-free, 80 °C	180	85	xiv
3	[Hmim]TFA	Solvent-free, 80 °C	180	85	xv
4	[TMPSA]HSO_4	H_2O, 100 °C	60	94	xvi
5	[DDPA][HSO_4]	H_2O, 100 °C	60	93	xvii
6	[Et_3N-SO_3H]Cl	Solvent-free, 80 °C	60	97	xviii
7	[Hbim]BF_4	Ultrasonication, rt, MeOH	45	85	xix
8	[BPy][HSO_4]	Solvent-free, 100 °C	10	90	This work

[a] Based on benzaldehyde.

Table 6 Comparison of ionic liquids used as catalysts for the synthesis of 1,8-dioxo-decahydroacridines [a]

Entry	Catalyst	Conditions	Time (min)	Yield (%)	Reference
1	[bmim]Br	[bmim]Br, 90 °C	40	93	xxix
2	[bpy]BF_4	Solvent-free, 90 °C	90	92	xxxv
3	[bmim]BF_4	Solvent-free, 90 °C	90	85	xxxv
4	[bmim]PF_6	Solvent-free, 90 °C	90	85	xxxv
5	[BPy][HSO_4]	Solvent-free, 100 °C	25	91	This work

[a] Based on 4-chlorobenzaldehyde.

CONCLUSION

In summary, we developed a simple and efficient procedure for synthesis of 1,8-dioxo-octahydroxanthenes and1,8-dioxo-decahydroacridines in the presence of [BPy]HSO_4 as catalyst. The remarkable features of this procedure are high yields, cleaner reaction profiles, use of non-toxic, reusable and environmentally benign catalyst, and simple experimental and work-up procedures.

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