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Research Article

A novel basic dicationic molten salt based on 4, 4'-bipyridine for synthesis of 1, 8-dioxooctahydroxanthene and 1, 8-dioxodecahydroacridine derivatives under solvent-free conditions

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ABSTRACT

Xanthene and acridine derivatives are important classes of heterocyclic compounds exhibiting remarkable pharmacological activities. In this study, a versatile and efficient catalytic protocol was developed for the synthesis of 1,8-dioxooctahydroxanthene and 1,8-dioxodecahydroacridine derivatives. The reactions were promoted by a novel dicationic Brönsted molten salt catalyst, 1, 1'-sulfinyl bis(4, 4'-bipyridinium)dichloride ([$(BPy)_2SO$][Cl_2]) under solvent-free conditions. The catalytic system proved to be highly efficient, affording 1, 8-dioxooctahydroxanthene derivatives in 30 min-60 min with 80%-95% yield, and 1, 8-dioxodecahydroacridine derivatives in 50 min-70 min with 83%-92% yield. Moreover, the catalyst showed outstanding reusability and could be recycled for at least six consecutive runs without any noticeable loss of activity.

Keywords: basic dicationic molten salt; bipyridine, 1, 8-dioxooctahydroxanthene derivatives; 1, 8-dioxodecahydroacridine derivatives; solvent-free; multi-component reaction

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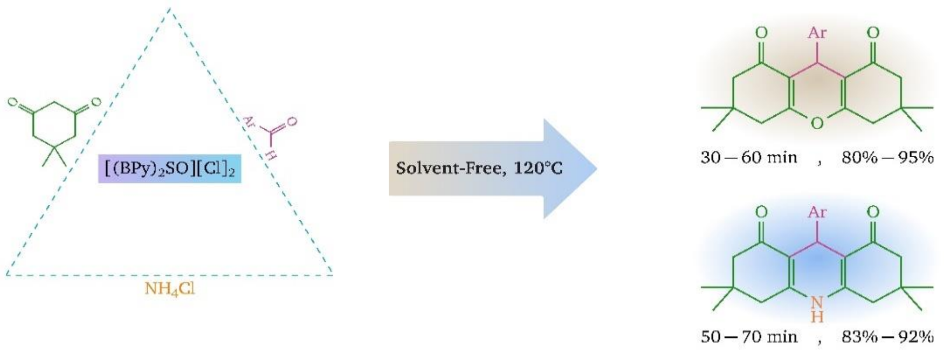
Introduction

Ionic liquids (ILs), a special group of molten salts, are organic salts composed of large unsymmetrical organic cations containing various substituents and weakly coordinated organic or inorganic anions [1]. These interesting compounds have versatile properties, including excellent solubility, high thermal and chemical stability, negligible vapor pressure, recyclability, wide liquid range, and designable structure via suitable modification of the cation and the anion [2,3]. Recently, a new class of IL research has emerged, focusing on developing dicationic ionic liquids (DILs) [4]. Such ILs contain two monocations combined into a dication and linked by a rigid or flexible organic spacer [5]. In recent years, the utilization of DILs for a variety of research areas, such as stationary phase for chromatography (GC), electrochemistry, organic reaction catalyst, extractants, lubricants, and electrolytes, have blossomed [4,6].

Basic ionic liquids (BILs), a kind of ionic liquid, can be considered safer alternatives to organic and inorganic bases in many reactions. They exhibit remarkable properties, such as a wide range of melting points to remain in a liquid state, reasonable moisture and thermal stability, the ability to dissolve inorganic, organic, and polymeric materials, lower vapor pressure, and recyclable as homogeneous catalysts or reactant medium [7]. BILs are used in various organic transformations such as Knovenagel condensation [8-10], Michael addition [11-13], Aza-Michael reaction [7], synthesis of cyclic carbonate [14], conjugate cyanation of CF_3-substituted alkyliden malonets, synthesis of 2-amino-4*H*-chromenes and spirochromenes [15], glucose aqueous isomerization [13], synthesis of *sec*-butanol from *sec*-butyl acetate [17], synthesis of 2-(phenylsulfonyl)-1*H*-benzo[a]pyrano[2,3-c]phenazin-3-amine derivatives [18], and synthesis of xanthenes [19].

Acridines are among the important class of heterocyclic compounds that have been exploited for their remarkable pharmaceutical and biological activities, such as anticancer [20], anti-tumor [21], anti-malaria [22], anti-microbial [23] and antifungal properties [24] as well as calcium β-blockers [25]. Acridines can be synthesized by multi-component reactions (MCRs), which is a powerful strategy for assembling complicated molecules in a single synthetic operation. As a commonly used protocol for the synthesis of these heterocyclic compounds, a three-component reaction of various aldehydes, dimedone, and nitrogen sources such as ammonium chloride [26], ammonium acetate [27], and aniline [28] are used. For accelerating this MCR, various catalysts have been used such as [ImSi][PF_6]@xanthan [29], 1-methylimidazolium tricyanomethanide [HMIM]$C(CN)_3$ [30], pulsed laser ablated zeolite nanoparticles [31], betainium-based ionic liquid [32], N-propyl benzoguanamine sulfonic acid supported on magnetic Fe_3O_4 nanoparticles [33], KH_2PO_4 [34], biodegrable chitosan SO_3H [35], $Fe_3O_4@SiO_2$ [36], graphene-based nanoparticles [37], nano-$Fe_3O_4@SiO_2@(CH_2)_3$-Imidazole-SO_3HCl [38], hydroxylamine-O-sulfonic acid [39], and sulfonic acid functionalized imidazolium salts [40]. Catalytic condensation of aldehydes with dimedone is a usual procedure for the synthesis of 1, 8-dioxooctahydroxanthene derivatives such as graphene oxide incorporated strontium nanoparticles [41], Brönsted acidic ionic liquid [42], $ZrCl_4$ or NH_4VO_3 [43], cyclodextrin nanosponges [44], immobilized Lewis acidic ionic liquid on perlite nanoparticle surface [45], $Na_2Ca(HPO_4)_2$ [46], SnP_2O_7 [47], [H-Pyrr][HSO_4] [48], 1,10-Phenanthrolin-1-ium Trinitromethanide [49], and sulfonic acid-functionalized titana-coated magnetic nanoparticles [50]. Although the literature survey outlines numerous methods for the synthesis of acridines and xanthenes, many of them suffer from several drawbacks, such as the use of expensive catalysts, long reaction times, low yields, the need for harsh reaction conditions (e.g., refluxing and strongly acidic media), and tedious, time-consuming work-up procedures. In line with our research on the synthesis of novel ionic liquids [51-64] herein, we report the synthesis and characterization of the novel basic dicationic molten salt and its application in the synthesis of 1, 8-dioxooctahydroxanthene and 1, 8-dioxodecahydroacridine derivatives under solvent-free conditions (Scheme 1).



Scheme. 1. Schematic synthesis of 1, 8-dioxooctahydroxanthene and 1, 8-dioxooctahydroacridine derivatives using $[(BPy)_2SO][Cl]_2$.

2. Results and discussion

2.1 Characterization of the basic dicationic molten salt

2.1.1. Infrared spectroscopy

FT-IR spectra for the 4, 4'-bipyridine and the synthesized basic dicationic molten salt are shown in Figure 1. In this figure, the peak appeared at 3032 cm^{-1}, which is responsible for C-H stretching vibration of the aromatic framework. In addition, the peaks at 1020 and 1189 cm^{-1} are referred to S=O symmetric and asymmetric stretching, respectively.

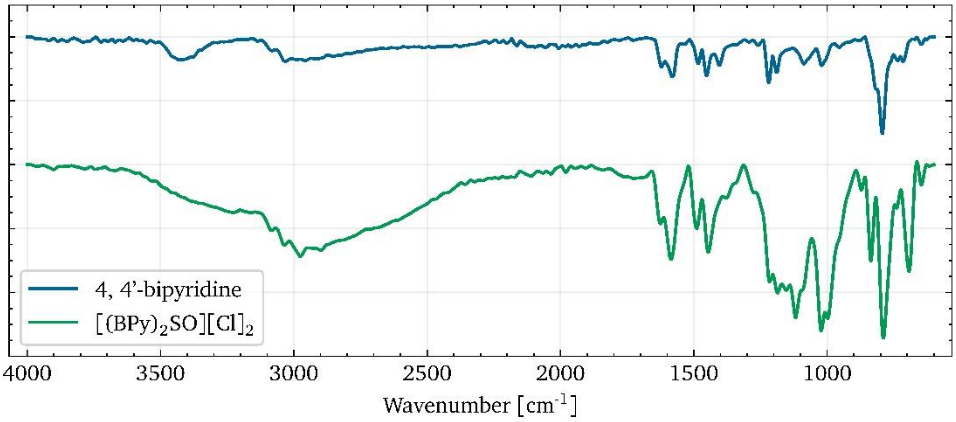
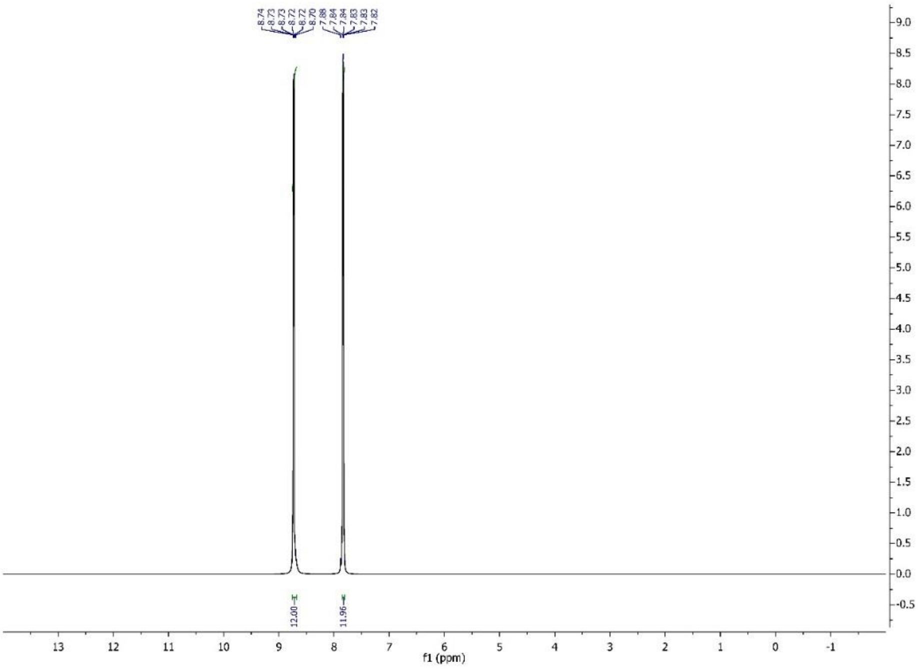


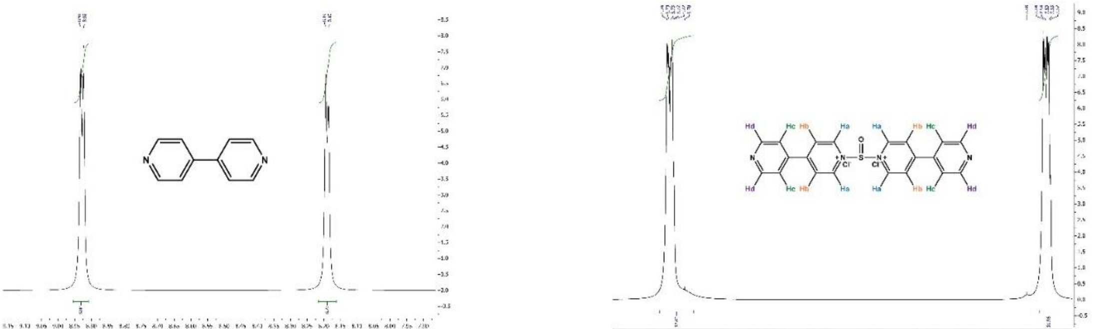
Fig. 1: FT-IR spectra of pure 4, 4'-bypiridine and [(BPy)$_2$SO][Cl]$_2$.

2.1.2. NMR analysis

The ^{1}H NMR spectrum of [(BPy)$_2$SO][Cl]$_2$ is shown in Figure 2a. As shown in Figure 2b, the ^{1}H NMR of 4, 4'-bipyridine shows two signals, each with doublet multiplicity at the aromatic region. After the reaction with thionyl chloride, the numbers, their chemical shifts, and their multiplicities of the signals are changed. In the ^{1}H NMR of [(Bpy)$_2$SO][Cl]$_2$, four expected peaks with doublet multiplicity were observed due to four different types of hydrogen in its structure. The chemical shift of hydrogen is depicted in Figure 2c. The expanded ^{1}H NMR spectrum of [(Bpy)$_2$SO][Cl]$_2$ is shown in Figure 2d and Figure 2d.

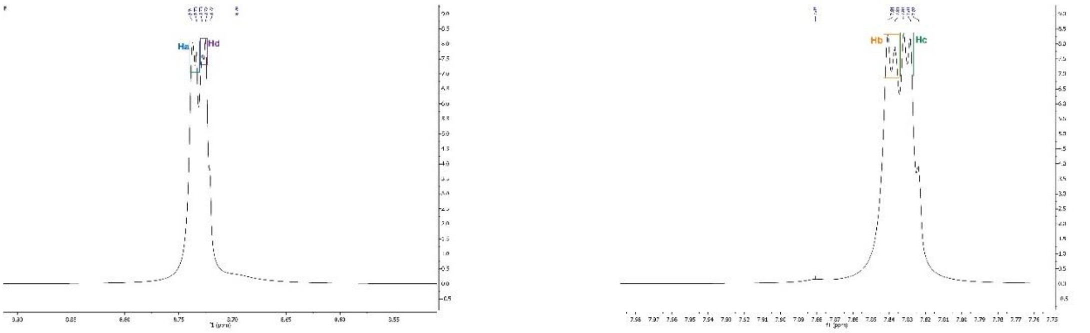


(a) ^{1}H NMR of $[(Bpy)_2SO][Cl]_2$.



(b) ^{1}H NMR of 4, 4’-bipyridine (expanded).

€ ^{1}H NMR of $[(Bpy)_2SO][Cl]_2$ (expanded).



(d) The part of expanded ^{1}H NMR of $[(Bpy)_2SO][Cl]_2$.

€ The other part of expanded of ^{1}H NMR of $[(Bpy)_2SO][Cl]_2$.

Fig. 2. The ^{1}H NMR of the 4, 4’-bipyridine and $[(Bpy)_2SO][Cl]_2$ in DMSO-d_6.

For further determination of the structure of the prepared molten salt, the following experiments were performed:

- Reaction of 4, 4’-bipyridine (1 mmol) with thionyl chloride (1 mmol) was done in CH_2Cl_2 at 0°C. The ^1H NMR of the product is shown in Figure 3.

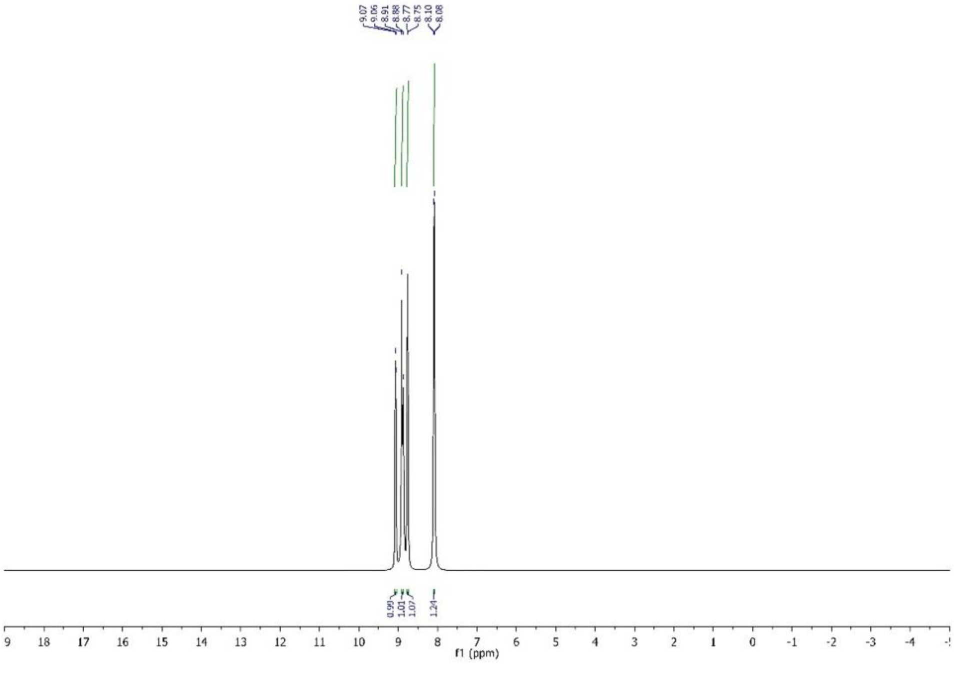
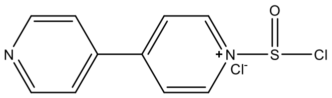


Fig. 3: The ^1H NMR of the product of the reaction of 1 mmol 4,4’-bipyridine with 1 mmol thionyl chloride in CH_2Cl_2 at 0°C

According to Figure 3, the following structure can be proposed:



- Reaction of 4, 4’-bipyridine (1 mmol) with thionyl chloride (2 mmol) was done in CH_2Cl_2 at 0°C.

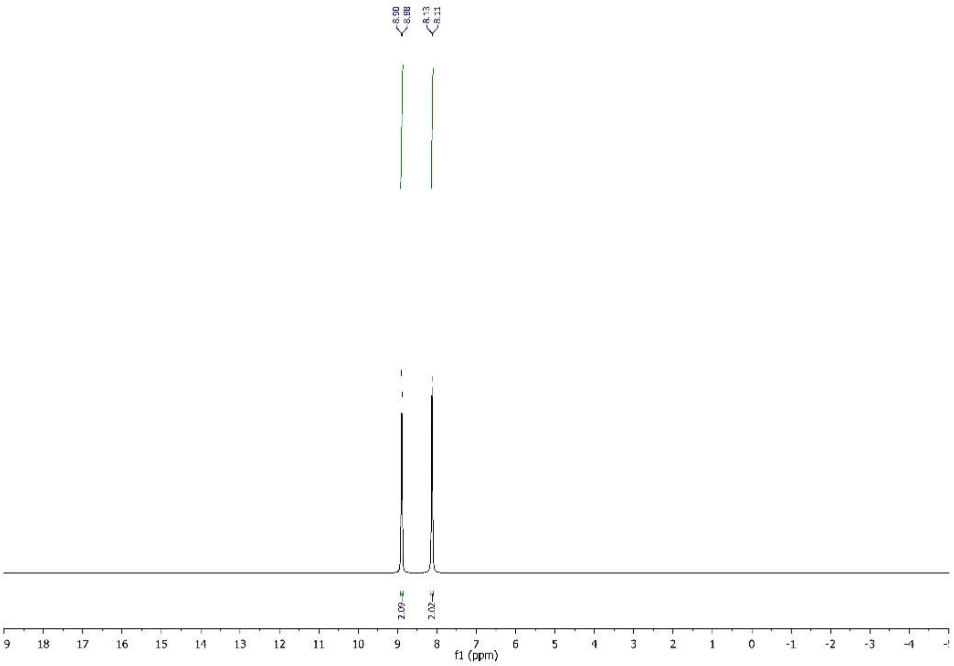
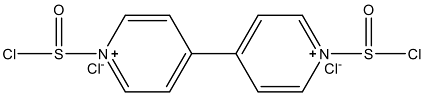


Fig. 4: The ^{1}H NMR of the product of the reaction of 1 mmol 4, 4'-bipyridine with 2 mmol thionyl chloride in CH_2Cl_2 at 0°C.

According to Figure 4, the proposed structure is as follows:



The ^{13}C NMR spectrum of the $[(Bpy)_2SO][Cl]_2$ is shown in Figure 5. The six signals appeared in the spectrum, which shows that the six nonequivalent carbons exist in the structure of prepared molten salt.

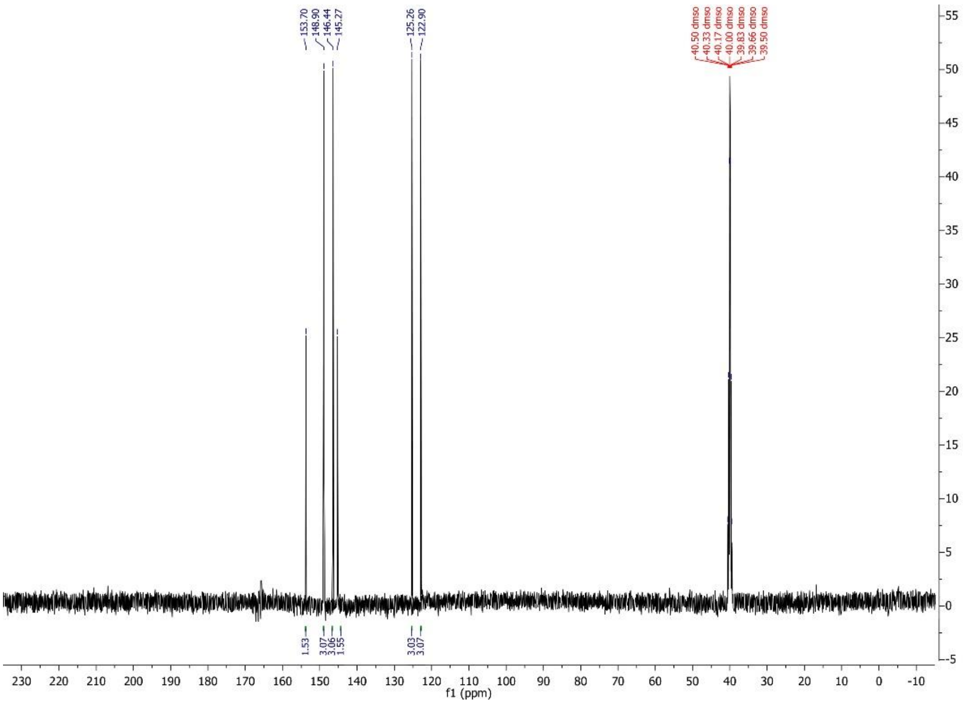
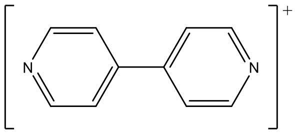


Fig. 5: The ^{13}C NMR of the $[(Bpy)_2SO][Cl]_2$ in the DMSO-d_6.

2.1.3. Mass Spectroscopy

The mass spectrum of $[(Bpy)_2SO][Cl]_2$ is shown in Figure 6. The correct molecular ion peak in this spectrum appears at 431 *m/z*. The base peak (156 *m/z*) is related to



Species. Another important peak is related to $C_5H_4N^+$ species which appears at 78 *m/z*.

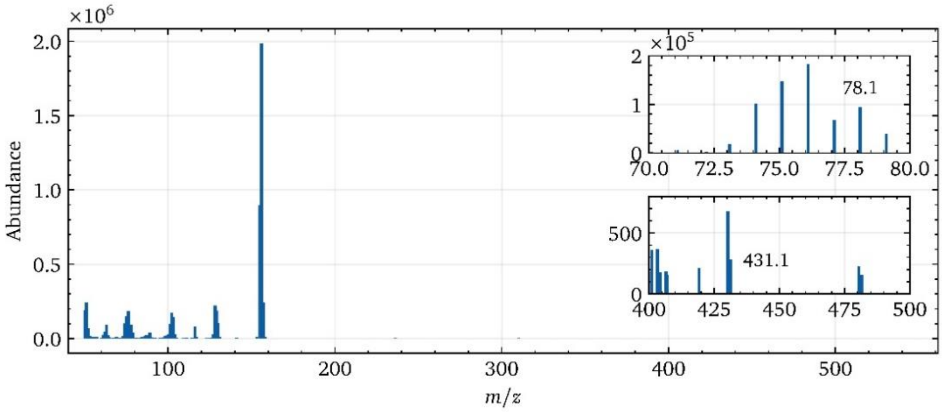


Fig. 6: Mass spectrum of $[(BPy)_2SO][Cl]_2$.

2.1.4. Thermal Analysis

The thermo-gravimetric analysis of $[(BPy)_2SO][Cl]_2$ under nitrogen atmosphere on increasing the temperature at the rate of $20^{o}C.min^{-1}$ is shown in Figure 7. The TGA and DTG profiles of the compound exhibit a single major decomposition event. The sample remains thermally stable up to approximately $250^{o}C$, with negligible weight loss, confirming the absence of residual solvent or moisture. A pronounced DTG peak centered at approximately $300^{o}C$ probably corresponds to the cleavage of S–N bond in the molten salt. Above $330^{o}C$, the TGA curve shows a gradual weight decrease attributed to the progressive decomposition of the residual organic group.

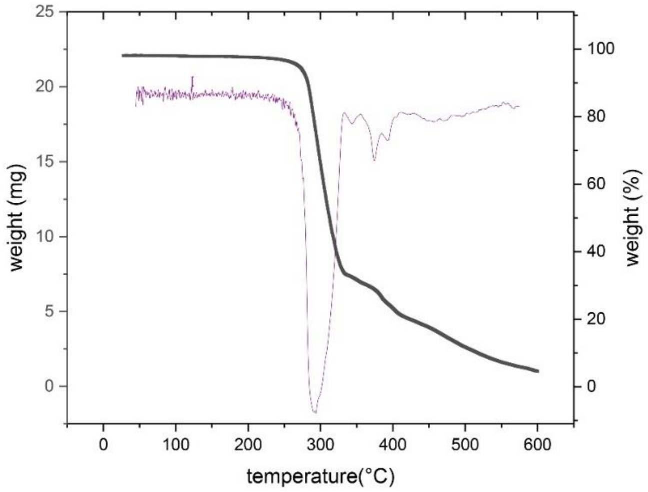


Fig. 7: Thermo-gravimetric analysis (TGA/DTG) of $[(BPy)_2SO][Cl]_2$.

2.1.5. Elemental Analysis and Mohr Titration

The elemental analysis of the $[(BPy)_2SO][Cl]_2$ showed that the calculated value for each element is approximately consist with the found one (Table 1).

Table. 1. Elemental analysis of $[(BPy)_2SO][Cl]_2$.

	CHN Analysis					
Compound	C (%)		H (%)		N (%)	
	Calc.	Obs.	Calc.	Obs.	Calc.	Obs.
	55.68	55.19	3.71	4.12	12.99	12.92

Mohr titration of 5.011×10^{-3} *M* solution of $[(BPy)_2SO][Cl]_2$ was obtained with a standard solution of $AgNO_3$ (0.100 *M*). According to the calculations, it can be concluded that 2.5 mL of $AgNO_3$ is need for the precipitation of each mmol Cl^-. The experimental result was 5.23 mL. The information indicated that $[(BPy)_2SO][Cl]_2$ involves 2 mmol Cl^-.

$$M_1V_1 = M_2V_2 \Rightarrow (5.011 \times 10^{-3})(50\ \text{mL}) = (0.100)V_2$$

$$V_2 = 2.5\ \text{mL (required } AgNO_3 \text{ solution against 1 mmol } Ag^+ \text{ for } [(BPy)_2SO][Cl]_2\text{)}$$

The above-mentioned structural analyses confirm that the proposed structure for the basic dicationic molten salt is correct. In addition, these data implicitly indicate that during the formation of $[(BPy)_2SO][Cl]_2$, only one nitrogen atom is involved. It should be noted that the reaction conditions, *i.e.*, the use of an aprotic solvent (CH_2Cl_2), low temperature (0^oC), and the rapid precipitation of the product, along with the above information, suggest that polymerization cannot occur.

2.1.6. SEM Analysis

The surface morphology was studied using SEM analysis (Figure 8). This figure shows that the cuboid-shaped particles are observed for 1, 1'-sulfinyl bis(4, 4')bipyridinium dichloride.

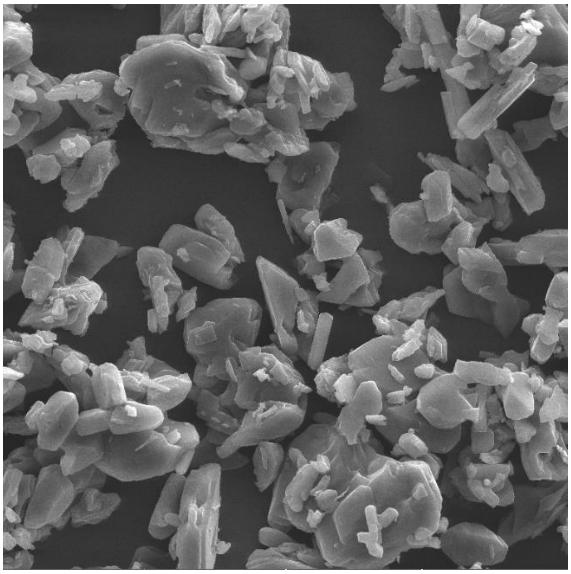


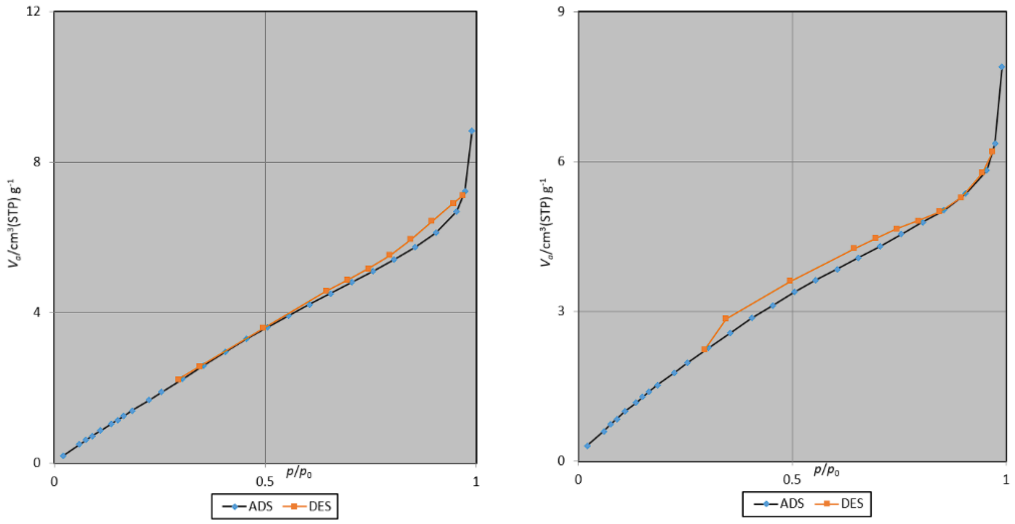
Fig. 8. SEM of $[(BPy)_2SO][Cl]_2$.

2.1.7. Characterization by BET

N_2 adsorption–desorption analysis was carried out to examine the textural properties of the fresh and reused catalysts, and the corresponding results are presented in Table 2 and Figure 9. For the reused catalyst, the BET surface area and pore volume decreased slightly from 11.474 $m^2.g^{-1}$ and 0.013608 $cm^3.g^{-1}$ to 10.319 $m^2.g^{-1}$ and 0.012142 $cm^3.g^{-1}$, respectively, indicating that the catalyst retained its structural integrity and catalytic activity even after six successive cycles.

Table. 2. The surface area and pore volumes of $[(BPy)_2SO][Cl]_2$ and its reused.

Compound	BET Surface Area ($m^2.g^{-1}$)	Pore Volume ($cm^3.g^{-1}$)
$[(BPy)_2SO][Cl]_2$	11.474	0.013608
Reused $[(BPy)_2SO][Cl]_2$	10.319	0.012142



(a) Before. (b) After.

Fig. 9. N_2 adsorption-desorption isotherms of $[(BPy)_2SO][Cl]_2$ and its reused.

2.2. Study of Solvent Miscibility of $[(BPy)_2SO][Cl]_2$

This investigation was performed mixing 0.1 g of pure BIL in 3 mL of solvent at room temperature. As shown in Table 3, the $[(BPy)_2SO]$ is soluble in H_2O, MeOH, and DMSO.

Table. 3. Solubility of $[(BPy)_2SO][Cl]_2$ in different solvents.

Temp.	H_2O	CH_3OH	CH_2Cl_2	DMSO	$CHCl_3$	*n*-hexane	Et_2O	CH_3CN	THF	EtOH
r.t.	+[a]	+	-[b]	+	-	-	-	-	-	-

[a] +: good solubility
[b] -: Poor solubility

2.3. Catalytic Performance

To conduct an initial study to establish the optimal condition for the synthesis of 1, 8-dioxooctahydroxanthene derivatives, a reaction involving 4-chlorobenzaldehyde **1a** and dimedione **2** to afford 3, 4, 6, 7-tetrahydro-3, 3, 6, 6-tetramethyl-9-(4-chlorophenyl)-2H-xanthene-1, 8 (5H, 9H) dione (**3a**) was selected as the model reaction for optimizing different parameters such as solvent, temperature, and amount of the catalyst (Table 4). In addition, we test the solubility of the synthesized molten salt in various solvents, presented in Table 3. The results indicate that the condition having 25 mol% of the catalyst, 120°C of the reaction temperature under solvent-free conditions is the best to obtain the highest reaction yield (Table 4, entry 10).

Table. 4. Effect of solvents, temperature, and amount of the catalyst on the model reaction [a].

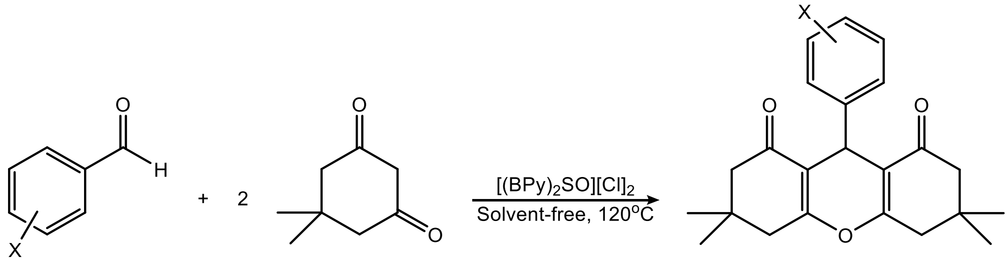
Entry	Solvent	Amount of Cat. (mol%)	Temp. (°C)	Time (min)	Yield (%)[b]
1	Water	25	Reflux	90	20
2	EtOH	25	Reflux	90	30
3	Water/EtOH	25	Reflux	90	30
4	Hexane	25	Reflux	90	25
5	$CHCl_3$	25	Reflux	90	35
6	CH_2Cl_2	25	Reflux	90	28
7	Acetone	25	Reflux	60	7
8	EtOAc	25	Reflux	60	10
9	2-Buthanol	25	Reflux	60	25
10	Solvent-free	25	120	60	95
11	Solvent-free	25	110	60	88
12	Solvent-free	25	100	60	75
13	Solvent-free	20	120	60	85
14	Solvent-free	30	120	60	90

[a] Reaction conditions: 4-chlorobenzaldehyde (**1a**, 1 mmol), dimedone (**2**, 2 mmol), different solvent (15 mL, entries 1–9, refluxing temperature), solvent-free (entries 10–14), temperature 100°C to 120°C, catalyst (20 mol% to 30 mol%).

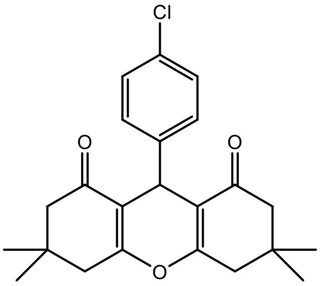
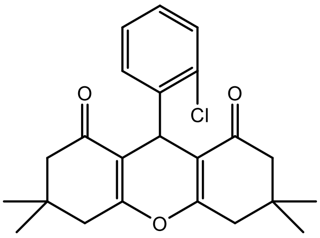
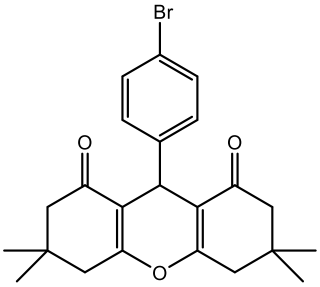
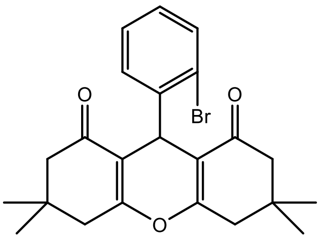
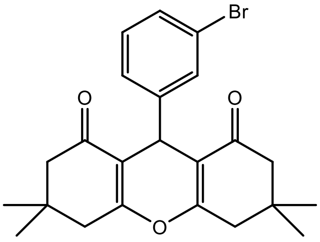
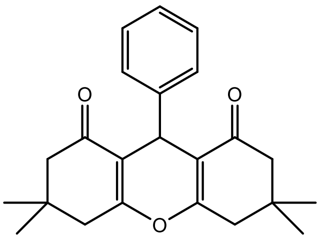
[b] Isolated yields of products.

After ascertaining the optimized reaction conditions, a broad range of aromatic aldehydes was reacted with dimedone under this protocol to synthesize the corresponding 1, 8-dioxooctahydroxanthene derivatives (Table 5). As shown in Table 5, aldehydes with electron-donating groups (-OH, -OMe) as well as electron-withdrawing groups (-Cl, -Br, -F, $-NO_2$) reacted successfully and afforded the corresponding products in high yields. After obtaining successful results with the synthesis of 1, 8-dioxooctahydroxanthene derivatives, synthesis of 1, 8-dioxodecahydroacridines was attempted. For this, the reaction of 4-chlorobenzaldehyde **1a**, dimedone **2**, and ammonium chloride **3** is considered as the model reaction. From these experiments, 25 mol% of the catalyst at 120°C under solvent-free conditions came out as the best reaction conditions in terms of yield and time for the corresponding product (Table 6).

Table. 5. [(BPy)$_2$SO][Cl]$_2$ catalyzed synthesis of 1, 8-dioxooctahydroxanthenes (\textbf{3a-k}) under solvent-free conditions.



1a **2** **3a-k**

		
3a, 95%, 45 min 237°C (237°C-238°C [65]) TON/TOF = 3.8/5.06	**3b**, 85%, 50 min 224°C (226 °C-227°C [66]) TON/TOF = 3.04/4.08	**3c**, 88%, 35 min 242°C (240°C-241°C [65]) TON/TOF = 3.52/6.03
		
3d, 92%, 45 min 225°C (226°C-228°C [66]) TON/TOF = 3.68/4.90	**3e**, 80%, 50 min 189°C (190 °C-191°C [66]) TON/TOF = 3.20/3.84	**3f**, 82%, 50 min 202°C (202°C-203°C [66]) TON/TOF = 3.28/3.93

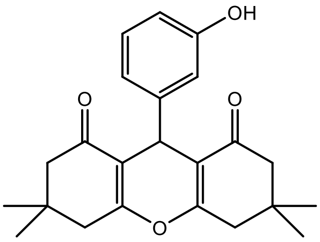
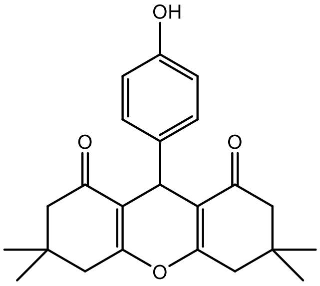
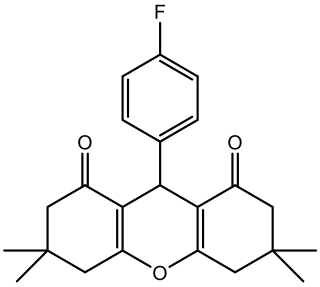
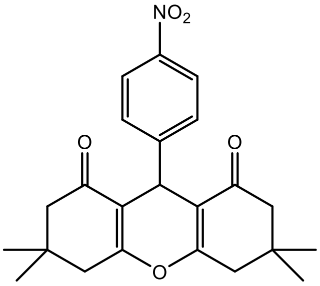
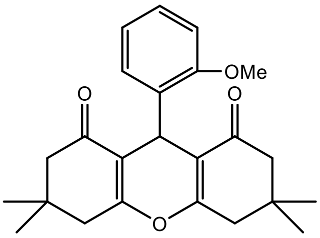
		
3g, 84%, 60 min 226°C (223°C-225°C [67]) TON/TOF = 3.36/3.36	**3h**, 91%, 52 min 249°C (247 °C-248°C [66]) TON/TOF = 3.64/4.20	**3i**, 92%, 40 min 224°C (223°C-224°C [66]) TON/TOF = 3.68/5.52
		
3j, 82%, 30 min 222C (221°C-223°C [65]) TON/TOF = 3.28/6.56	**3k**, 84%, 60 min 192°C (190 °C-191°C [68]) TON/TOF = 3.36/3.36	

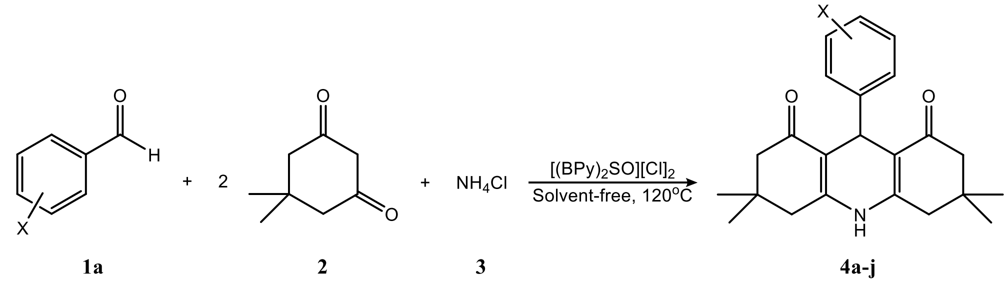
Table. 6. Optimization of reaction conditions on the model reaction [a].

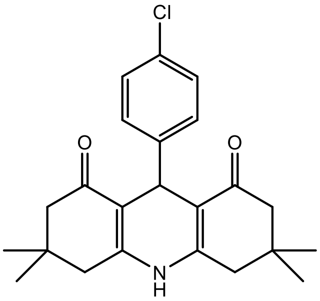
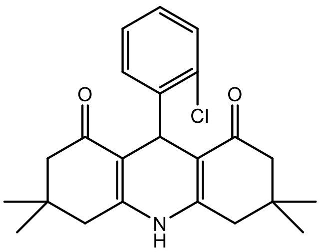
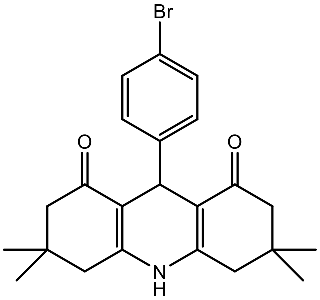
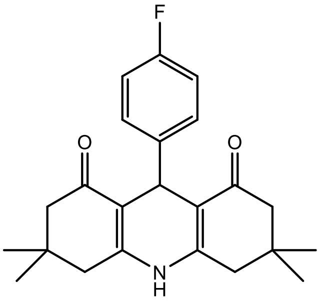
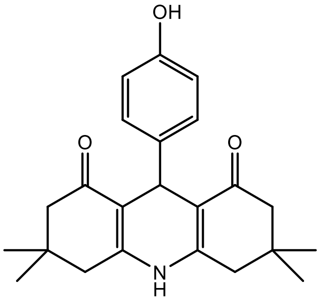
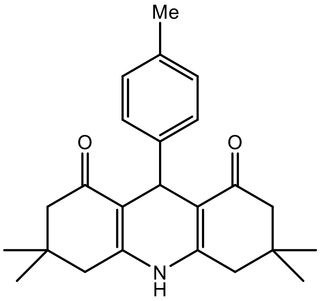
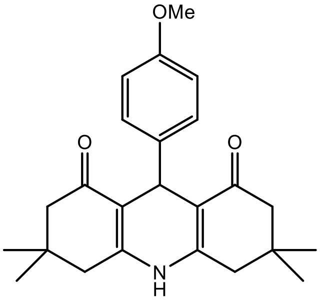
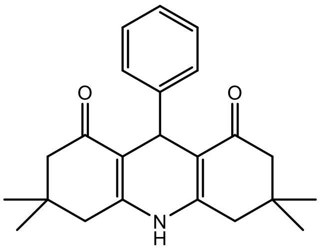
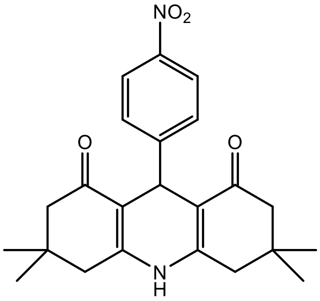
Entry	Amount of Catalyst (mol%)	Temp. (°C)	Yield (%)[b]
1	15	120	73
2	20	120	85
3	25	120	92
4	30	120	92
5	25	110	88
6	25	100	80

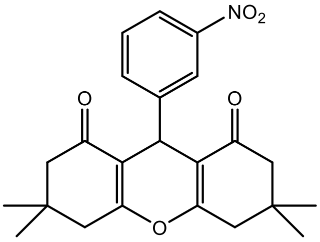
[a] Reaction conditions: 4-chlorobenzaldehyde (**1a**, 1 mmol), dimedone (**2**, 1 mmol), ammonium chloride (**3**, 1 mmol), solvent-free, reaction time: 60 min.
[b] Isolated yields of products.

The substrate scope of $[(BPy)_2SO][Cl]_2$-catalyzed was then examined under optimized reaction conditions. As shown in Table 7, various aromatic aldehydes, possessing electron-donating (-OH, -OMe, -Me) and electron-withdrawing (-NO_2, -F, -Cl, -Br) groups were reacted to prepare a wide range of 1, 8-dioxodecahydroacridine derivatives in high yields.

Table. 7. Scope of the developed catalytic method for the synthesis of 1,8-dioxodecahydroacridine derivatives under solvent-free conditions.



		
4a, 92%, 60 min 302°C (299°C-301°C [69]) TON/TOF = 3.68/3.68	**4b**, 89%, 70 min 220°C (222°C-224°C [70]) TON/TOF = 3.56/3.05	**4c**, 90%, 60 min 311°C (310°C-312°C [70]) TON/TOF = 3.60/3.60
		
4d, 90%, 60 min 291°C (289°C-291°C [70]) TON/TOF = 3.60/3.60	**4e**, 87%, 55 min 303°C (302°C-304°C [70]) TON/TOF = 3.48/2.98	**4f**, 84%, 70 min 271°C (269°C-270°C [69]) TON/TOF = 3.36/2.88
		

4g, 89%, 70 min 271°C (270°C-272°C [71]) TON/TOF = 3.56/3.06	**4h**, 85%, 60 min 193°C (190°C-192°C [71]) TON/TOF = 3.40/3.40	**4i**, 86%, 50 min 286°C (286°C-288°C [69]) TON/TOF = 3.44/5.16
		
4j, 83%, 65 min 291°C (288°C-290°C [69]) TON/TOF = 3.32/3.06		

2.4. Reusability of the catalyst

Efficient catalysts have acceptable reusability. Thus, the reusability of the catalyst was examined in the reaction of 4-chlorobenzaldehyde and dimedone under optimized reaction conditions. Next, as monitored by TLC (*n*-hexane/EtOAc:7/3), after completion of the reaction, H_2O (10 mL) was added to the reaction mixture, and the product was isolated by filtration. The catalyst was recovered from the filtrate by removing the water under reduced pressure. Subsequently, the recycled catalyst was dried for the next run. The experimental results displayed in Figure 10 show that $[(BPy)_2SO][Cl]_2$ can be reused at least six times without noticeable deterioration in catalytic activity. Furthermore, the FT-IR spectrum of the recycled catalyst exhibited no detectable structural alterations, confirming the stability and integrity of the catalyst under the applied reaction conditions (Figure 11).

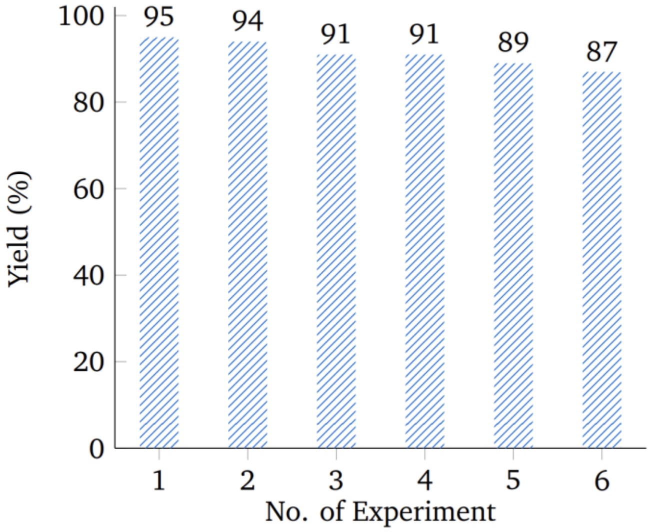


Fig. 10. Recovery and reusability of $[(BPy)_2SO][Cl]_2$ in the synthesis of **3a**.

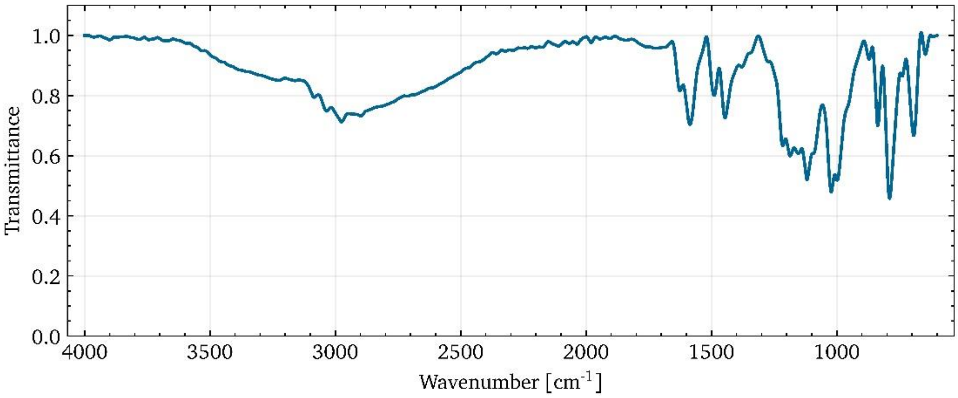
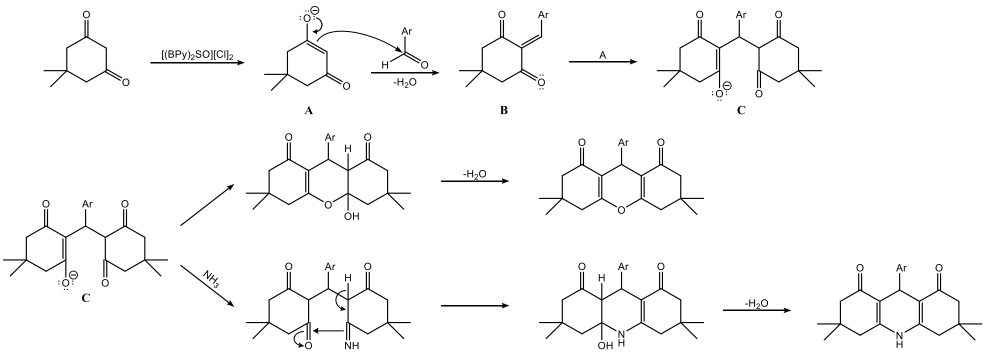


Fig. 11. FT-IR spectrum of the reused catalyst.

2.5. Mechanism

A plausible mechanism for base-catalyzed synthesis of 1, 8-dioxooctahydroxanthenes and 1, 8-dioxodecahydroacridines is suggested in Scheme 2. Initially, the basic catalyst removes an α proton of dimedone to form an enolate ion (**A**). The nucleophilic addition of **A** to aldehyde, after water removal to afford Michael acceptor **B**, which undergoes Michael addition with **A** to produce intermediate **C**. This intermediate can be converted to the xanthene product or in the presence of NH_3 to furnish the acridine product.



Scheme. 2. Plausible mechanistic route towards the synthesis of 1, 8-dioxooctahydroxanthenes and 1, 8-dioxooctahydroacridines in the presence of [(BPy)2SO][Cl]2.

A comparative study of some reported procedures in the literature to prepare **3a** is compared in Table \ref{tab:tab5} to show the efficacy of the prepared catalyst. The results indicate that this method is more effective than others regarding product yield and reaction time.

Table. 8. The comparison of catalytic performance of $[(BPy)_2SO][Cl]_2$ with some methods.

Entry	Catalyst	Reaction Conditions	Time	Yield (%) [Ref.]
		1, 8-dioxooctahydroxanthene derivatives		
1	SO_4^{2-}/10Fe-Zr-O	MW irradiation, 100°C	12 min	83 [71]
2	[bmim][HSO_4]	Solvent-free, 80°C, 100 mg	3.5 h	95 [72]
3	[Et_3NH][HSO_4]	Solvent-free, 100°C, 20 mol%	45 min	87 [65]
4	SmI_3	Solvent-free, 120°C, 20 mol%	9 h	97 [73]
5	$[(BPy)_2SO][Cl]_2$	Solvent-free, 100°C, 25 mol%	45 min	95 (this work)
		1, 8-dioxooctahydroacridine derivatives		
6	Fe_3O_4@SiO_2-MoO_3H	Solvent-free, 100°C, 20 mg	20 min	92 [74]
7	L-proline	H_2O, reflux, 35 mol%	(2-3) h	88 [69]
8	[ImSi][PF_6]@xanthan	Ethanol, reflux, 50 mg	32 min	95 [29]
9	Hbet[Lac]	Ethanol, reflux, 30 mol%	3.5 h	85 [32]
10	$[(BPy)_2SO][Cl]_2$	Solvent-free, 100°C, 25 mol%	45 min	92 (this work)

3. Experimental Section

The starting materials were commercially available and were used without further purification. Infrared spectra were recorded using Bruker-Equinox 55 (USA) FT-IR spectrophotometer using KBr disc. ^1H and ^{13}C NMR spectra were recorded on an INOVA 500

MHz using DMSO-d_6 as the solvent. The chemical shifts were recorded on the δ-scale (ppm) using residual solvents as internal standards. Thermo-gravimetric analysis (TGA/DTG) was performed using Linseis STA PT 1600. Mass spectra were recorded on Finnigza MAT-321 A, Germany. Scanning electron micrograph was collected using Tescan MIRA 3 LMU microscope. Melting points were determined on an electrothermal device Brönsted 9100 and were uncorrected. Progress of the reactions was monitored by TLC (*n*-hexane/EtOAc:7/3).

3.1. Synthesis of basic dicationic molten salt

The synthesis of basic dicationic molten salt based on 4, 4'-bipyridine is shown in Figure 12. The synthesis proceeded as follows: To an ice-cooled flask (100 mL) containing CH_2Cl_2 (30 mL) and a magnetic stirring bar was added, while stirring, 4, 4'-bipyridine (0.624 g, 4 mmol) to become a clear solution. Then, with caution, a solution of thionyl chloride (0.237 g, 2 mmol) in CH_2Cl_2 (20 mL) was instilled to the flask at 0°C and stirring continued for 30 min. After that, the reaction mixture was stirred for 24 h at room temperature. Afterward, the solvent was removed by rotary evaporator, and the solid residue was washed with CH_2Cl_2 (3×15 mL) and dried under vacuum at 40°C by 6 h to afford $[(BPy)_2SO][Cl]_2$ as a white solid in 95% yield.

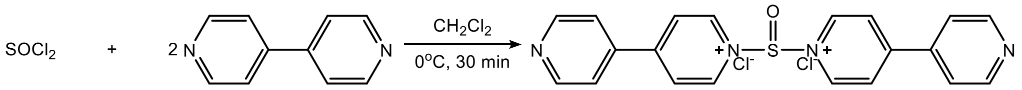


Fig. 12. Synthesis of the basic dicationic molten salt ($[(BPy)_2SO][Cl]_2$).

3.2. General procedure for the synthesis of 1, 8-dioxooctahydroxanthenes

To a mixture of aromatic aldehydes (1 mmol) and dimedone (2 mmol, 0.24 g), the catalyst (25 mol%, 0.11 g) was added and the resulting mixture was stirred in an oil-bath at 120°C for an appropriate time (Table 5). After completion of the reaction (as monitored by TLC), water (10 mL) was added while stirring for 5 min, and the solid residue was collected by filtration.

The crude product was recrystallized from hot ethanol to afford the pure product. The structure of pure products was confirmed by melting point, FT-IR, and ^{1}H NMR.

3.3. General procedure for the synthesis of 1, 8-dioxodecahydroacridines

To a mixture of aromatic aldehydes (1 mmol), dimedone (2 mmol, 0.28 g), and ammonium chloride (1 mmol, 0.05 g) in a small beaker, 25 mol% of the catalyst (0.11 g) was added. The resulting mixture was stirred in an oil-bath at 120°C for an appropriate time (Table 7). After completion of the reaction (as monitored by TLC), water (10 mL) was added while stirring for 5 min, and the solid residue was collected by filtration. The crude product was recrystallized from hot ethanol to afford the pure product. The structure of pure products was confirmed by melting point, FT-IR, and ^{1}H NMR.

5. Conclusion

In this work, a dicationic Brönsted-basic molten salt, $[(BPy)_2SO][Cl]_2$, was prepared through careful stoichiometric control and optimized reaction conditions enabling site-selective functionalization of 4, 4'-bipyridine. The catalyst combines the practical advantages of ionic-liquid-type reactivity with facile heterogenization, as it is readily separated from the reaction mixture and reused. Using $[(BPy)_2SO][Cl]_2$, the solvent-free synthesis of 1, 8-dioxooctahydroxanthenes and 1, 8-dioxodecahydroacridines was achieved in high yields and short reaction times with a simple work-up, avoiding the need for large amounts of ammonium salts commonly encountered in related protocols. The catalyst also exhibited good thermal stability (up to 250°C, as indicated by TGA) and maintained catalytic performance over six consecutive cycles; notably, the product yield decreased only slightly (from 95% in the first run to 87% in the sixth run), demonstrating practical recyclability. Overall, $[(BPy)_2SO][Cl]_2$ emerges as a robust, reusable, and operationally simple catalyst platform for efficient solvent-free synthesis of valuable heterocyclic and polycyclic scaffolds.

4. Conflict of interest

There is no conflict of interest.

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