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Preparation, characterization and testing the catalytic activity of a new acidic ionic liquid in multicomponent reactions

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ABSTRACT

In this study, N^1,N^1,N^2,N^2-tetramethylethane-1,2-diamine was reacted with chlorosulfonic acid to afford N^1,N^1,N^2,N^2-tetramethyl-N^1,N^2-bis(sulfo)ethane-1,2-diaminium chloride ([TMBSED][Cl]$_2$) as a new acidic ionic liquid. [TMBSED][Cl]$_2$ was identified by ^{1}H and ^{13}C NMR, mass and FT-IR spectra. Then, its catalytic activity was examined to promote the following multicomponent reactions: (i) the production of 4,4′-(arylmethylene)-bis(3-methyl-1-phenyl-1*H*-pyrazol-5-ol)s from arylaldehydes and 3-methyl-1-phenyl-1*H*-pyrazol-5(4*H*)-one in ethanol, and (ii) the synthesis of 12-aryl-8,9,10,12-tetrahydrobenzo[*a*]xanthen-11-ones from arylaldehydes, dimedone and 2-naphthol in solvent-free conditions. The ionic liquid was highly efficient and general catalyst for these reactions.

***Keywords**: Acidic ionic liquid, [TMBSED][Cl]$_2$, 4,4′-(Arylmethylene)-bis(3-methyl-1-phenyl-1H-pyrazol-5-ol), 12-Aryl-8,9,10,12-tetrahydrobenzo[a]xanthen-11-one.*

1. Introduction

Recently, ionic liquids have attracted the attention of chemists due to their particular properties like undetectable vapor pressure, non-inflammability, high thermal and chemical stability, wide liquid range, and capability to use as solvent and catalyst for organic synthesis [1-7]. Among the various kinds of ionic liquids, Brønsted acidic ones, possessing both solid and liquid acid properties, have been designed to replace conventional mineral liquid acids like sulfuric acid and hydrochloric acid in catalytic chemical reactions [2-7]. Hence, development of a novel acidic ionic liquid to catalyze organic transformations would be desirable.

4,4′- (Arylmethylene)- bis (3- methyl- 1- phenyl- 1*H*-pyrazol-5-ol)s have a broad spectrum of approved biological activities; e.g. they have been used as antidepressant [8], antipyretic [9], antiviral [10], and antifilarial [11] agents. These compounds have been also applied as dyestuff [12]. The best method for the synthesis of 4,4′-(arylmethylene)-bis(3-methyl-1-phenyl-1*H*-pyrazol-5-ol)s is the condensation reaction of arylaldehydes with 3-methyl-1-phenyl-1*H*-pyrazol-5(4*H*)-one in the presence of a catalyst [13-19]. This class of compounds can be also prepared by the reaction between phenylhydrazine, ethyl acetoacetate and aldehydes [20].

Xanthenes and benzoxanthenes are of considerable interest in industry as well as in academia, because of their promising biological and pharmaceutical activities such as anti-inflammatory [21] and antiviral [22] actions. These active oxygen-containing heterocyclic compounds could also been employed as zoxazolamine [23] and dyes [24], and in photodynamic therapy [25] and laser technology [26]. 12-Aryl-8,9,10,12-tetrahydrobenzo[*a*]xanthen-11-ones are an important class of xanthene derivatives, which are prepared by the multicomponent reaction between arylaldehydes, dimedone and 2-naphthol using a catalyst [27-34].

Considering the above facts, we have reported here the preparation of a novel Brønsted acidic ionic liquid namely N^1,N^1,N^2,N^2-tetramethyl-N^1,N^2-bis(sulfo)ethane -1,2-diaminium chloride ([TMBSED][Cl]$_2$) from N^1,N^1,N^2,N^2-tetramethyl ethane-1,2-diamine and chlorosulfonic acid. This ionic liquid has been characterized by studying its ^{1}H and ^{13}CNMR, mass

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and FT-IR spectral data. Then, the catalytic activity of [TMBSED][Cl]$_2$ has been examined for the synthesis of 4,4′- (arylmethylene)- bis (3- methyl-1- phenyl- 1*H*-pyrazol-5-ol) and 12-aryl-8,9,10,12-tetrahydrobenzo [*a*]xanthen-11-one derivatives.

2. Experimental

2.1. General

All chemicals were purchased from Fluka or Merck Chemical Companies. All known products were identified by comparison of their melting points and spectral data with those reported in the literature. Progress of the reactions was monitored by thin layer chromatography (TLC). The melting points were recorded on a Büchi B-545 apparatus in open capillary tubes. The ^{1}HNMR (250, 300 or 500 MHz) and ^{13}CNMR (62.5, 75 or 125 MHz) were run on a Bruker Avance DPX, FT-NMR spectrometers (δ in ppm). Mass spectra were obtained with Shimadzu GC-MS-QP 1100 EX model.

2.2. Production of [TMBSED][Cl]$_2$

A solution of N^1,N^1,N^2,N^2-tetramethylethane-1,2-diamine (5 mmol, 581 mg) in dry CH_2Cl_2 (30 mL) was added dropwise to a stirring solution of chlorosulfonic acid (10 mmol, 1165 mg) in dry CH_2Cl_2 (30 mL) over a period of 10 min, at 10 °C. After that, the reaction mixture was heated to room temperature (accompanied with stirring), and stirred for another 4 h. The solvent was evaporated under reduced pressure, and the liquid residue was triturated with dry petroleum ether (3×2 mL), and dried under powerful vacuum at 90 °C to give [TMBSED][Cl]$_2$ as a viscous pale yellow oil in 97% yield (Scheme 1).

IR (Nujol): $\bar{\nu}$ = 856, 1058, 1146, 1291, 2400-3300 cm^{-1}; ^{1}HNMR (250 MHz, DMSO-d_6): δ= 2.82 (s, 12H), 3.48 (s, 4H), 12.50 (br., 2H) ppm. ^{13}CNMR (62.5 MHz, DMSO-d_6): δ= 42.5, 50.5 ppm. MS: *m/z*= 349 (M^+), 332 (M^+-OH), 315 (M^+-2OH), 313 (M^+-Cl$^-$), 278 (M^+-2Cl$^-$), 268 (M^+-SO_3H).

2.3. Synthesis of 4,4′-(arylmethylene)-bis(3-methyl-1-phenyl-1H-pyrazol-5-ol)s

A mixture of arylaldehyde (1 mmol), 3-methyl-1-phenyl-1*H*-pyrazol-5(4*H*)-one (348 mg, 2 mmol) and [TMBSED][Cl]$_2$ (35 mg, 0.1 mmol) in ethanol (3 mL) was stirred at 60 °C. After completion of the reaction, as monitored by TLC, the ethanol was evaporated under vacuum, and the resulting precipitate was recrystallized from aqueous ethanol (90%) to give the pure product.

2.4. Production of 12-aryl-8,9,10,12-tetrahydrobenzo [a] xanthen- 11- ones

A mixture of compounds including 2-naphthol (144 mg, 1 mmol), arylaldehyde (1 mmol), dimedone (140 mg, 1 mmol) and [TMBSED][Cl]$_2$ (28 mg, 0.08 mmol) was firstly stirred magnetically at 100 °C, and after solidification of the reaction mixture, it was stirred with a small rod at same temperature. After completion of the reaction, as monitored by TLC, the mixture was cooled to room temperature, and the solid residue was recrystallized from aqueous ethanol (90 %) to afford the pure product.

3. Results and Discussion

3.1. The characterization of [TMBSED][Cl]$_2$

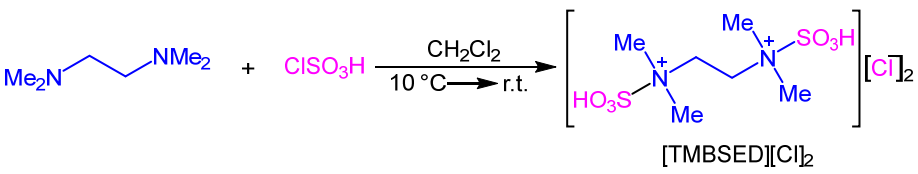
The novel acidic ionic liquid was characterized by studying its ^{1}H and ^{13}CNMR, mass and FT-IR spectral data.

The ^{1}HNMR spectrum of [TMBSED][Cl]$_2$ (Fig. 1) showed 3 peaks: 2.82 (s, 12H), 3.48 (s, 4H) and 12.50 (br., 2H) ppm. The corresponded hydrogens to each peak are shown in Fig. 2. As figures 1 and 2 indicate, the ^{1}HNMR data confirmed the ionic liquid structure.

In the ^{13}CNMR spectrum (Fig. 3), 2 peaks were observed (42.5 and 50.5 ppm). The related carbons to each peak are shown in Fig. 4. As it can be seen in Fig. 3 and 4, the ^{13}CNMR data verified the structure of [TMBSED][Cl]$_2$.

The FT-IR spectrum of [TMBSED][Cl]$_2$ (supplementary information) was also assist us to prove the catalyst structure. The FT-IR data are summarized in Table 1.

In the mass spectrum of the ionic liquid (supplementary information), the molecular ion peak was observed at *m/z* = 349.



[TMBSED][Cl]$_2$

Scheme 1. The synthesis of [TMBSED][Cl]$_2$.

3.2. Catalytic activity testing

3.2.1. Examining the catalytic activity of $[TMBSED][Cl]_2$ for the synthesis of 4,4´-(arylmethylene)-bis(3-methyl-1-phenyl-1H-pyrazol-5-ol)s

At first, as a model reaction, the condensation of 3-nitrobenzaldehyde (1 mmol) with 3-methyl-1-phenyl-1*H*-pyrazol-5(4*H*)-one (2 mmol) (Scheme 2) was tested in the presence of $[TMBSED][Cl]_2$ (0.1 mmol) under solvent-free conditions and in some solvents (3 mL) at 60 °C; the results are displayed in Table 2. As Table 2 indicates, higher yield and shorter reaction time were obtained in EtOH (entry 2).

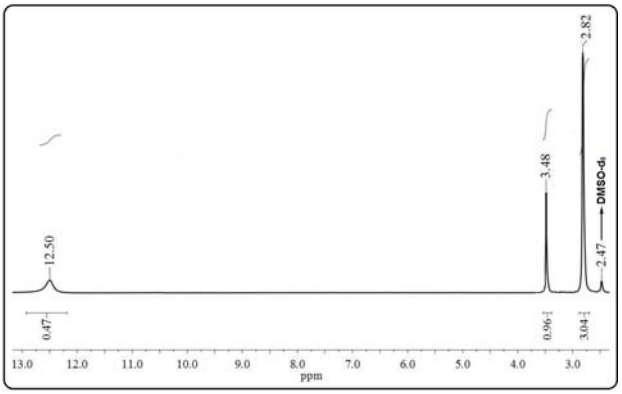


Fig. 1. The ^{1}HNMR spectrum of $[TMBSED][Cl]_2$.

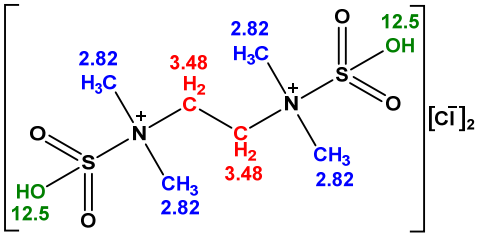


Fig. 2. The structure of $[TMBSED][Cl]_2$ for ^{1}HNMR data analyzing.

In second step, effect of the catalyst amount and temperature on the reaction was studied; for this purpose, the reaction was examined using various amounts of the ionic liquid at a range of 50-70 °C in ethanol. The best results were observed in the presence of 10 mol% of $[TMBSED][Cl]_2$ at 60 °C.

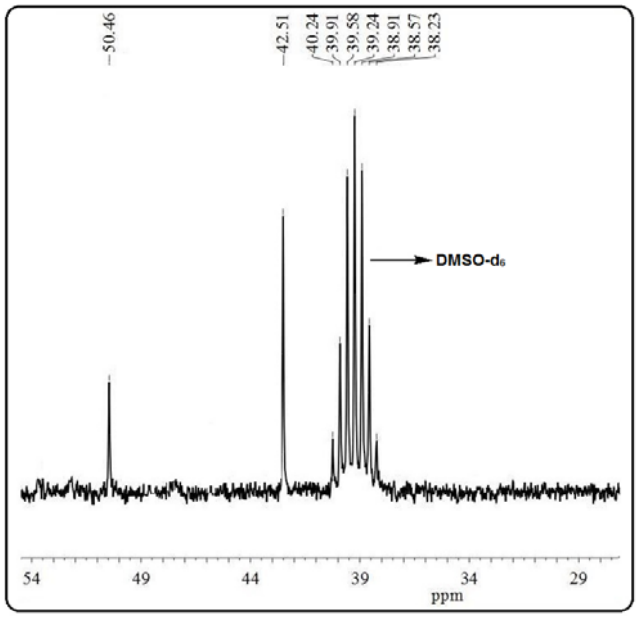


Fig. 3. The ^{13}CNMR spectrum of the catalyst.

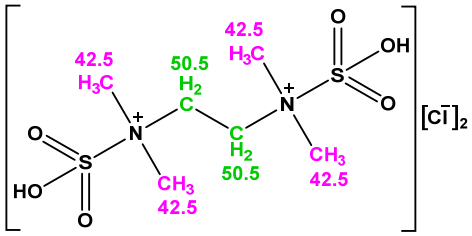
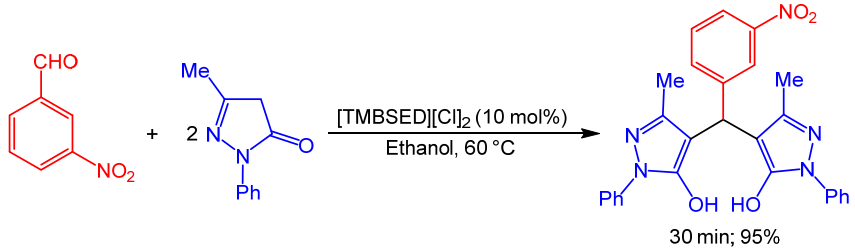


Fig. 4. The catalyst structure for analyzing the ^{13}CNMR data.

Table 1. The FT-IR data of $[TMBSED][Cl]_2$.

Peak (cm^{-1})	856	1058	1146 and 1291	2400-3300
Related bond	Symmetric N-S stretching vibration	S-OH bend	Asymmetric and symmetric stretching and bending of S-O vibrations	OH groups of the two SO_3H



Scheme 2. The condensation of 3-nitrobenzaldehyde with 3-methyl-1-phenyl-1*H*-pyrazol-5(4*H*)-one.

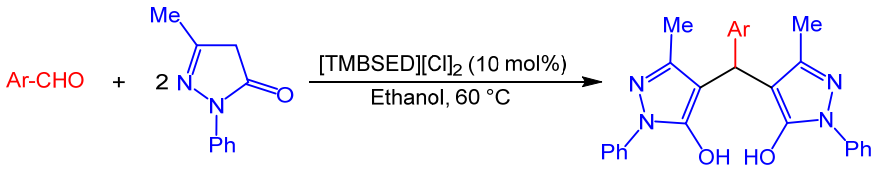
Table 2. Influence of solvent, catalyst amount and temperature on the reaction of 3-nitrobenzaldehyde with 3-methyl-1-phenyl-1*H*-pyrazol-5(4*H*)-one.

Entry	Solvent	The catalyst (mol%)	Temp. (ºC)	Time (min)	Yield (%)[a]
1	Solvent-free	10	60	50	69
2	EtOH	10	60	30	95
3	EtOAc	10	60	40	85
4	MeCN	10	60	40	89
5	EtOH	5	60	45	61
6	EtOH	8	60	30	72
7	EtOH	13	60	30	95
8	EtOH	10	50	30	82
9	EtOH	10	70	30	95

[a]Isolated yield.

To evaluate the generality and effectiveness of $[TMBSED][Cl]_2$ for the production of 4,4′- (arylmethylene)- bis (3-methyl- 1- phenyl- 1*H*-pyrazol-5-ol)s, a variety of arylaldehydes (possessing diverse substituents) were reacted with 3-methyl-1-phenyl-1*H*-pyrazol-5(4*H*)-one under the optimal reaction conditions; the respective results are depicted in Table 3. As the data in this Table show, the catalyst was general and highly efficient for the synthesis; all aromatic aldehydes (without substituent or containing electron-withdrawing, electron-donating and halogen substituents) furnished the related products in high yields within short reaction times. Nevertheless, the aldehydes with electron-withdrawing groups afforded the corresponding products in shorter reaction times than those with electron-donating groups; this can be attributed to activation of the carbonyl group of aldehyde (to accept a nucleophile) by electron-withdrawing substituents. Moreover, catalytic performance of the ionic liquid was checked for the reaction of 3-methyl-1-phenyl-1*H*-pyrazol-5(4*H*)-one with an aliphatic aldehyde (phenylacetic acid) in which the reaction wasn't successful.

Table 3. The production of 4,4′-(arylmethylene)-bis(3-methyl-1-phenyl-1*H*-pyrazol-5-ol)s promoted by $[TMBSED][Cl]_2$.



Product	Ar	Time (min)	Yield (%)[a]	m.p. (°C)		Ref.
				Found	Reported	
1A	C_6H_5	30	91	167-169	168-170	[16]
2A	3-$O_2NC_6H_4$	30	95	147-149	151-154	[15]
3A	4-$O_2NC_6H_4$	30	95	228-230	228-230	[16]
4A	2-$O_2NC_6H_4$	30	90	222-224	221-223	[18]
5A	4-MeC_6H_4	40	93	202-204	203-205	[17]
6A	4-$MeOC_6H_4$	45	90	174-176	176-179	[15]
7A	4-HOC_6H_4	40	94	151-153	154-157	[15]
8A	4-ClC_6H_4	15	90	213-215	213-215	[18]
9A	2-ClC_6H_4	25	90	234-236	236-237	[17]
10A	3-BrC_6H_4	30	90	172-174	172-174	[16]
11A	2-BrC_6H_4	30	88	247-249	248-250	[16]
12A	4-Cl-3-$O_2NC_6H_3$	25	95	234-236	237-238	[15]

[a]Isolated yield.

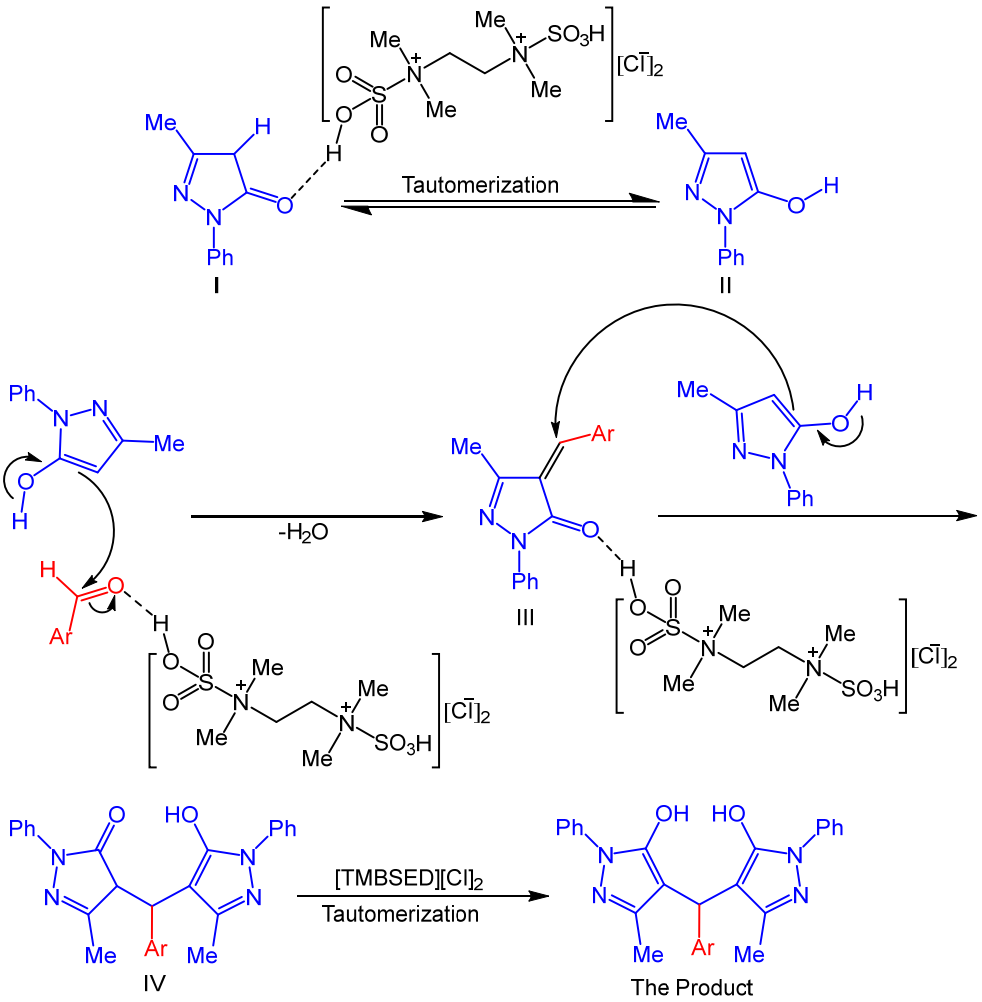
We proposed a plausible mechanism for the synthesis of 4,4′-(arylmethylene)-bis(3-methyl-1-phenyl-1*H*-pyrazol-5-ol)s catalyzed by [TMBSED][Cl]$_2$ (Scheme 3). This mechanism is supported by the literature [16].

To compare the efficiency of our catalyst with the reported catalysts for the preparation of 4,4′-(arylmethylene)-bis(3-methyl-1-phenyl-1*H*-pyrazol-5-ol)s, we have tabulated the reaction results and conditions for the synthesis of compound **3A** using these catalysts, in Table 4. As it can be seen in this Table, [TMBSED][Cl]$_2$ is superior related to the reported catalysts in terms of reaction time, yield, temperature and/or conditions.

3.2.2. Testing the catalytic activity of [TMBSED][Cl]$_2$ for the production of 12-aryl-8,9,10,12-tetrahydrobenzo[a]xanthen-11-ones

Catalytic performance of [TMBSED][Cl]$_2$ was also tested for the production of 12-aryl-8,9,10,12-tetrahydrobenzo[*a*]xanthen-11-ones. To reach this aim, the condensation between 3-nitrobenzaldehyde (1 mmol), dimedone (1 mol) and 2-naphthol (1 mmol) was selected as a model reaction (Scheme 4), and studied in the presence of different mol% of the catalyst at a range of 90-110 °C in solvent-free conditions, and also in some solvents; the results are summarized in Table 5. As this Table shows, the reasonable results were obtained when the reaction was performed using 8 mol% of [TMBSED][Cl]$_2$ at 100 °C in the absence of solvent (entry 1).

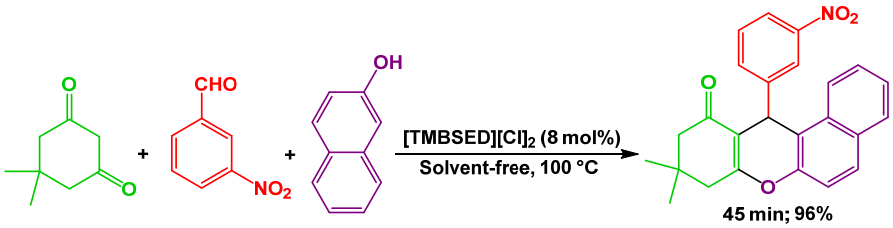
After that, various arylaldehydes (bearing electron-attracting, electron-releasing and halogen substituents) were reacted with dimedone and 2-naphthol in the optimized conditions (Table 6). As it can be seen in this Table, [TMBSED][Cl]$_2$ has successfully catalyzed the reactions, and was general and highly effective for the preparation of these xanthenes derivatives.



Scheme 3. The proposed mechanism for the production of 4,4′-(arylmethylene)-bis(3-methyl-1-phenyl-1*H*-pyrazol-5-ol)s using [TMBSED][Cl]$_2$.

Table 4. Comparison of the results and conditions for the synthesis of compound **3A** using our catalyst with those using the reported catalysts.

Catalyst	Conditions	Time (min)	Yield (%)	Ref.
$[TMBSED][Cl]_2$	EtOH, 60 °C	30	95	This work
Biosurfactant obtained from balanites roxburghii fruit	H_2O, 60 °C	25	83	[14]
1,3,5-Tris(hydrogensulfato) benzene	EtOH, 75 °C	3	96	[15]
Nanomagnetite-Fe_3O_4	Solvent-free, 70 °C	4	95	[16]
Nano-SiO_2/$HClO_4$	H_2O, Reflux	20	96	[17]
Catalyst-free	Poly(ethylene glycol)-400, 110 °C	60	94	[18]
[Amberlite]-*L*-prolinate	EtOH, Reflux	8	96	[19]
Silica-bonded *N*-propyl piperazine sulfamic acid	Solvent-free, 80 °C	35	91	[20]



Scheme 4. The model reaction for the synthesis of 12-aryl-8,9,10,12-tetrahydrobenzo[*a*]xanthen-11-ones.

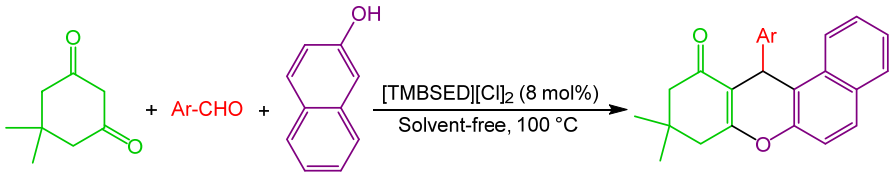
We proposed a plausible mechanism for the preparation of 12-aryl-8,9,10,12-tetrahydrobenzo[*a*] xanthen-11-ones using $[TMBSED][Cl]_2$ which is supported by the literature (Scheme 5) [30]. In another study, efficacy of our catalyst was compared with the reported catalysts for the preparation of 12-aryl-8,9,10,12-tetrahydrobenzo[*a*]xanthen-11-ones. The reaction results and conditions for the synthesis of compound **2B** were tabulated in Table 7. As it is shown in this Table, $[TMBSED][Cl]_2$ is superior with respect to the reported catalysts in terms of reaction time, yield, temperature and/or conditions.

Table 5. Influence of solvent, catalyst amount and temperature on the reaction of dimedone with 3-nitrobenzaldehyde and 2-naphthol.

Entry	Solvent	The catalyst (mol%)	Temp. (ºC)	Time (min)	Yield (%)[a]
1	Solvent-free	8	100	45	96
2	EtOH	8	Reflux	130	79
3	EtOAc	8	Reflux	100	65
4	THF	8	Reflux	90	74
5	H_2O	8	Reflux	150	21
6	Solvent-free	5	100	70	85
7	Solvent-free	10	100	45	94
8	Solvent-free	8	90	60	80
9	Solvent-free	8	110	45	93

[a]Isolated yield.

Table 6. The synthesis of 12-aryl-8,9,10,12-tetrahydrobenzo[*a*]xanthen-11-ones catalyzed by [TMBSED][Cl]$_2$.



Product	Ar	Time (min)	Yield (%)[a]	m.p. (°C)		Ref.
				Found	Reported	
1B	C_6H_5	55	95	148-150	150-152	[28]
2B	3-$O_2NC_6H_4$	45	96	168-170	169-171	[29]
3B	4-$O_2NC_6H_4$	40	90	176-178	176-179	[30]
4B	4-MeC_6H_4	35	85	172-174	175-177	[34]
5B	4-$MeOC_6H_4$	35	90	198-200	199-201	[29]
6B	4-ClC_6H_4	50	88	181-183	180-182	[28]
7B	2-ClC_6H_4	35	92	179-181	178-180	[28]
8B	2,4-$Cl_2C_6H_3$	55	88	182-184	183-185	[29]
9B	3-BrC_6H_4	60	96	158-160	159-162	[29]
10B	4-BrC_6H_4	45	94	184-186	186-187	[30]
11B	2-BrC_6H_4	50	96	171-173	168-170	[28]

[a]Isolated yield.

4. Conclusion

In summary, we have introduced a novel acidic ionic-liquid catalyst namely N^1,N^1,N^2,N^2-tetramethyl-N^1,N^2-bis(sulfo)ethane-1,2-diaminium chloride ([TMBSED][Cl]$_2$) for organic synthesis. In this research, it was successfully applied as catalyst for the production of two important class of organic compounds, i.e. 4,4′-(arylmethylene)-bis(3-methyl-1-phenyl-1*H*-pyrazol-5-ol)s and 12-aryl-8,9,10,12-tetrahydrobenzo[*a*]xanthen-11-ones, with the following benefits: (i) efficiency, (ii) generality, (iii) short reaction times, (iv) high yields, (v) easy preparation of the catalyst from relatively inexpensive reactants, (vi) capability of the catalyst to act in solvent-free and in solution conditions, (vii) clean reaction profile, and (viii) good compliance with the green chemistry protocols (in solvent-free conditions).

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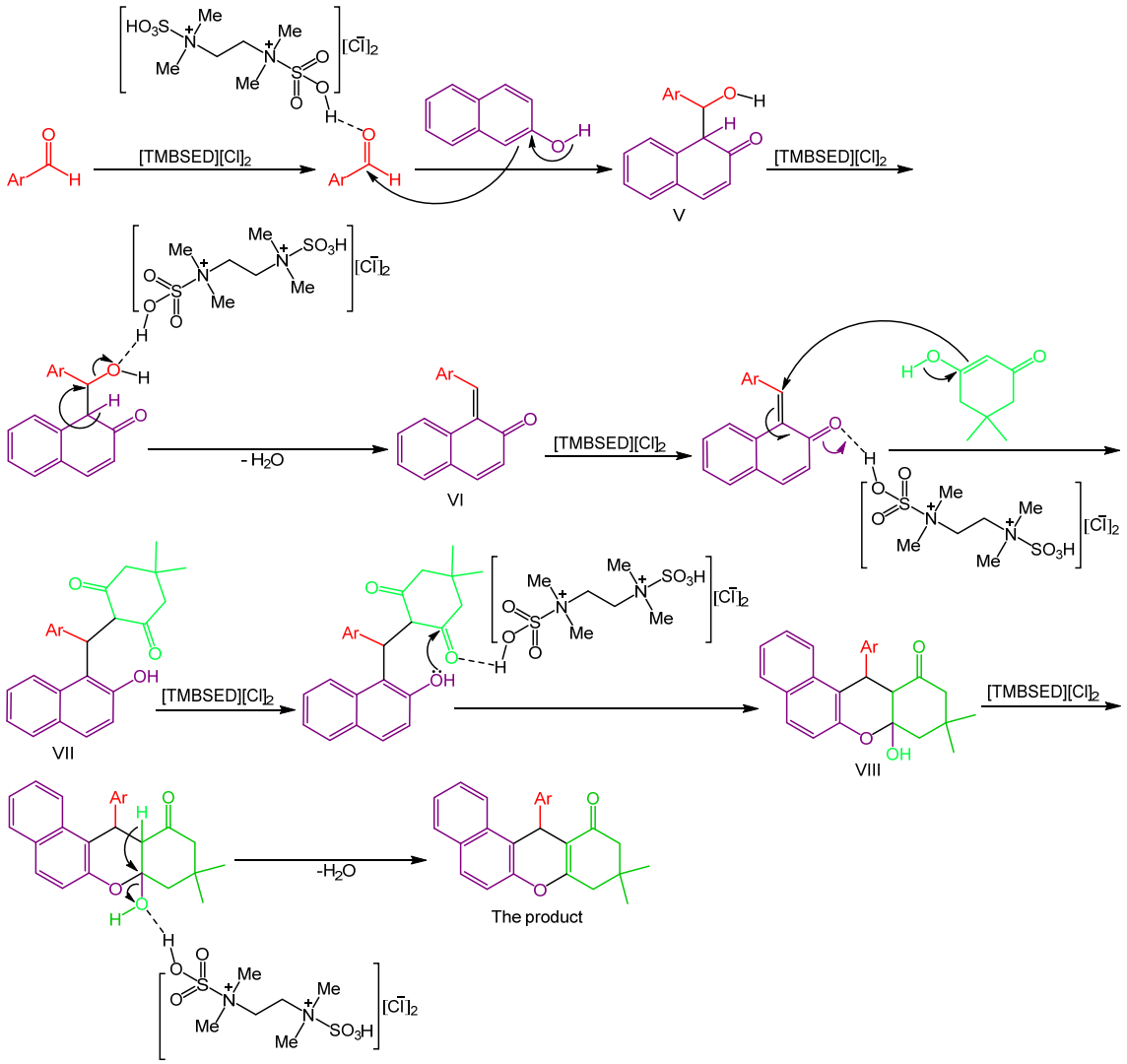

Scheme 5. The proposed mechanism for the production of 12-aryl-8,9,10,12-tetrahydrobenzo[*a*]xanthen-11-ones using $[TMBSED][Cl]_2$.

Table 7. Comparison of the results and conditions for the synthesis of compound **2B** using our catalyst with those using the reported catalysts.

Catalyst	Conditions	Time (min)	Yield (%)	Ref.
$[TMBSED][Cl]_2$	Solvent-free, 100 °C	45	96	This work
Orange peel	Solvent-free, 120 °C	40	90	[27]
Polyvinylpolypyrrolidoniume triflate	Toluene, 110 °C	300	85	[28]
$[Et_3N\text{-}SO_3H]Cl$	Solvent-free, 120 °C	60	85	[29]
silica-bonded imidazolium-sulfonic acid chloride	Solvent-free, 100 °C	8	93	[30]
Glucose sulfonic acid	H_2O, 90 °C	240[a]	84[a]	[31]
$RuCl_3.nH_2O$	EtOH, Reflux	35	86	[32]
$KAl(SO_4)_2.12H_2O$	Poly(ethylene glycol)-400, 60 °C	50[b]	87[b]	[33]
Fe_3O_4/chitosan-Ag nanoparticles	H_2O, 80 °C	30	91	[34]

[a]The results of the preparation of compound **9B** (In this work, compound **2B** hasn't been synthesized).
[b]The results of the reaction between dimedone, 4-nitrobenzaldehyde and 2,7-dihydroxynaphthalene.

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