Short communication

A Rapid Synthesis of Some 1,4-aryldiazines by the Use of Lithium Chloride as an Effective Catalyst

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Abstract

The synthesis of some 1,4-aryldiazines (novel and known dibenzo[*a,c*]phenazines and quinoxalines) based on the condensation of 1,2-aryldiamines with aromatic 1,2-dicarbonyl compounds in the presence of lithium chloride as a heterogeneous catalyst is presented as convenient and efficient strategy. This method has advantages such as excellent yields, short reaction times, and simple work-up procedure.

Keywords: Lithium chloride, Dibenzo[*a,c*]phenazine, Quinoxaline

1. Introduction

The chemistry of 1,4-aryldiazines such as phenazines and quinoxalines, has attracted wide interest because of the potential biological activity of this class of compounds.[1,2]

Some phenazine compounds are found in nature, and they are produced by bacteria such as *Pseudomonas spp*., *Streptomyces spp*., and *Pantoea agglomerans*. For example, some phenazine natural products have been implicated in the virulence and competitive fitness of producing organisms.[3,4] Quinoxaline and phenazine derivatives constitute the basis of many insecticides,[5] anti-tumors,[6] fungicides,[7] herbicides,[8] and receptor antagonists.[9] Moreover, they are used in dyes,[10] as the building blocks for the synthesis of organic semiconductors,[11] as well as for chemically controllable switches,[12] cavitands,[13] DNA cleaving agents,[14] dehydroannulenes,[15] electrical and photochemical materials,[16–18] and as an inhibitor for the corrosion of mild steel.[19] Synthesis of 1,4-aryldiazine moieties has remained the goal of many research groups over the years because of their wide range of applications. Recently, a number of synthetic strategies have been developed to prepare 1,4-aryl diazine derivatives.[20–29] The general method for the synthesis of quinoxalines is the condensation of 1,2-aryldiamines with 1,2-dicarbonyl compounds in refluxing ethanol in the presence of acetic acid.[31] However, the yields of products in this strategy were not good (2–12 h, 34–85% yields). Recently, the synthesis of quinoxaline derivatives via the condensation of 1,2-aryldiamines with 1,2-dicarbonyl compounds in MeOH/AcOH under microwave irradiation at 160 °C has been reported, but the process requires special instrumentation and procedure under harsh conditions.[32] It should be noted that improved methods have also been developed for synthesis of quinoxaline derivatives, using Fe/Al-MCM-41,[33] $Yb(OTf)_3$,[34] $H_6P_2W_{18}O_{62}.2H_2O$,[35] and Gallium (III) triflate.[36] However, some of the traditional processes suffer from several disadvantages such as pollution, the use of expensive catalyst, poor chemical yields, long reaction times, and tedious work-up procedures, which limit their use under the aspect of economically and environmentally benign processes.

In the present study, a new and simple route to quinoxalines and dibenzo[*a,c*]phenazines using lithium chloride as a heterogeneous catalyst is described.

2. Experimental

2. 1. General

Chemicals were purchased from Merck, Fluka, and Aldrich companies. The reactions were monitored by TLC (silica-gel 60 F_{254}, AcOEt/ hexane). IR spectra were

recorded on a FT-IR Shimadzu-470 spectrometer and the ^{1}H NMR spectra were obtained from a Bruker-Instrument DPX-400 and 500 MHz Avance 2 model. All products (except novel compounds) were characterized by comparison of their spectra and physical data with those reported in the literature.[20–30]

2. 2. General Procedure

A mixture of aromatic 1,2-dicarbonyl compound (1 mmol), *o*-phenylenediamine (1.1 mmol) and lithium chloride (10 mol %) in ethanol (5 mL) was stirred at room temperature. The progress of the reaction was monitored by TLC (AcOEt/ hexane: 3/7). After the completion of the reaction, the solid which separated, was filtered and recrystallized from ethanol to afford the pure product.

2. 3. Selected Spectral Data of SomeC

2,3-Diphenylquinoxaline (6a). ^{1}H NMR ($CDC1_3$, 400 MHz): δ 7.97–7.95 (m, 2H), 7.55–7.43 (m, 2H), 7.36 (d, 4H, *J* 6.00 Hz), 7.29–7.10 (m, 6H); ^{13}C NMR ($CDC1_3$, 100 MHz): δ 154.92, 142.39, 140.31, 131.54, 130.54, 130.00, 129.60, 129.05; IR (KBr): ν 3055, 1439, 1345, 768, 699 cm^{-1}.

6-Nitro-2,3-diphenylquinoxaline (6c). ^{1}H NMR ($CDC1_3$, 400 MHz): δ 8.83 (d, 1H, *J* 2.4 Hz), 8.27 (dd, 1H, *J* 9.2, 2.4 Hz), 8.08 (d, 1H, *J* 9.2 Hz), 7.38–7.35 (m, 4H), 7.22–7.15 (m, 6H); ^{13}C NMR ($CDC1_3$, 100 MHz): δ 157.43, 156.80, 148.96, 144.70, 141.07, 139.23, 139.17, 131.90, 131.07, 130.99, 130.93, 130.80, 129.61, 129.54, 126.74, 124.41; IR (KBr): ν 3010, 1610, 1519, 1327, 760, 690 cm^{-1}.

2,3-Bis(4-flouro-phenyl)quinoxaline (7a). ^{1}H-NMR (400 MHz, $CDC1_3$): δ 7.97 (dd, 2H, *J* 6.4, 3.6 Hz), 7.60 (dd, 2H, *J* 6.4, 3.2 Hz), 7.33–7.30 (m, 4H), 6.86 (t, 4H, *J* 8.8 Hz); ^{13}C-NMR (100 MHz, $CDC1_3$): δ 161.99, 152.20, 141.23, 135.02, 131.82, 131.74, 130.23, 129.16, 115.65, 115.43; IR (KBr): ν 3061, 1599, 1555, 1511, 1344, 1225, 839, 786 cm^{-1}.

2,3-Bis(4-flouro-phenyl)-6-methylquinoxaline (7b). ^{1}H-NMR (400 MHz, $CDC1_3$): δ 6.58 (t, 4H, *J* 8.8 Hz), 2.43 (s, 3H), 7.85 (d, 1H, *J* 8.8 Hz), 7.73 (s, 1H), 7.42 (d, 1H, *J* 8.8 Hz), 7.30 (dd, 4H, *J* 8.00, 5.2 Hz); ^{13}C-NMR (100 MHz, $CDC1_3$): δ 161.89, 152.05, 151.29, 141.28, 140.84, 139.69, 135.16, 135.13, 132.59, 131.77, 131.72, 131.69, 128.65, 127.96, 115.59, 115.37, 21.94 IR (KBr,): ν 2925, 2580, 1657, 1597, 1264, 1159, 833, 696 cm^{-1}.

2,3-Bis(4-chloro-phenyl)-6-methylquinoxaline (8b). ^{1}H NMR ($CDC1_3$, 500 MHz): δ 8.08 (d, 1H, *J* 6.8 Hz), 7.97 (s, 1H), 7.67 (dd, 1H, *J* 6.8, 1.6 Hz), 7.50 (dd, 4H, *J* 6.8, 0.8 Hz), 7.38 (d, 4H, *J* 6.4 Hz), 2.66 (s, 3H); ^{13}C NMR ($CDC1_3$, 125 MHz): δ 152.20, 151.43, 141.75, 141.43, 140.17, 137.85, 135.64, 135.56, 133.16, 131.6, 129.11, 128.42; IR (KBr): ν 3090, 2950, 1620, 1595, 1480, 1340, 1090, 840, 725 cm^{-1}.

Dibenzo[*a,c*]phenazine (9a). ^{1}H-NMR (400 MHz, $CDC1_3$): δ 9.18 (d, 2H, *J* 7.6 Hz), 8.34 (d, 2H, *J* 8 Hz), 8.12 (dd, 2H, *J* 6.4, 3.6 Hz), 7.66–7.51 (m, 6H); ^{13}C-NMR (100 MHz, $CDC1_3$): δ 143.54, 143.28, 133.15, 131.42, 130.88, 130.57, 129.04, 127.38, 124.03; IR (KBr): ν 3055, 1600, 1490, 1350, 760,720 cm^{-1}.

11-Methyl-dibenzo[*a,c*]phenazine (9b). ^{1}H-NMR (400 MHz, $CDC1_3$): δ 9.14 (2H, dd, *J* 6.00, 1.6 Hz), 8.32 (d, 2H, *J* 8 Hz), 7.97 (d, 1H, *J* 8.4 Hz), 7.58 (s, 1H), 7.53–7.52 (m, 5H), 2.54 (s, 3H); ^{13}C-NMR (100 MHz, $CDC1_3$): δ 143.29, 143.27, 142.72, 141.81, 141.41, 133.45, 133.06, 132.87, 131.49, 131.45, 131.20, 131.07, 130.01, 129.10, 128.92, 127.29, 127.15, 123.95, 23.20; IR (KBr) ν 3055, 2910, 1620, 1500, 1350, 760, 720 cm^{-1}.

Acenaphtho[1,2-*b*]quinoxaline (10a). ^{1}H-NMR (400 MHz, $CDC1_3$): δ 8.21 (d, 2H, *J* 6.8 Hz), 8.02 (dd, 2H, *J* 6.2, 3.2 Hz), 7.90 (d, 2H, *J* 8.4 Hz), 7.65 (t, 2H, *J* 7 Hz), 7.57 (dd, 2H, *J* 6.4, 3.6 Hz); ^{13}C-NMR (100 MHz, $CDC1_3$): δ 155.19, 142.39, 137.60, 132.92, 131.10, 130.47, 130.59, 130.36, 129.78, 122.96; IR (KBr): ν 3050, 1610, 1430, 1300, 830, 760 cm^{-1}.

9-Methyl-acenaphtho[1,2-*b*]quinoxaline (10b). ^{1}H-NMR (400 MHz, $CDC1_3$): δ 8.21 (t, 2H, *J* 6.4 Hz), 7.90 (dd, 3H, *J* 8.2 Hz, 3.2 Hz), 7.79 (s, 1H), 7.64 (t, 2H, *J* 7.4 Hz), 7.40 (dd, 1H, *J* 8.4, 1.6 Hz), 2.43 (s, 3H); ^{13}C-NMR (100 MHz, $CDC1_3$): δ 155.15, 154.44, 142.38, 140.82, 140.71, 137.35, 133.08, 132.44, 131.06, 130.46, 130.31, 130.21, 129.89, 129.72, 122.83, 122.68, 22.94; IR (KBr): ν 3055, 2910, 1610, 1415, 1300, 810, 790 cm^{-1}.

11-Benzoyl-dibenzo[*a,c*]phenazine (12). ^{1}H-NMR ($CDCl_3$, 400MHz): δ 9.43 (dd, 1H, *J* 8 Hz, 1.2 Hz), 9.35 (dd, 1H, *J* 8 Hz, 1.2 Hz), 8.70 (d, 1H, *J* 1.6 Hz), 8.58 (d, 2H, *J* 8 Hz), 8.44 (d, 1H, *J* 8.8 Hz), 8.55 (dd, 1H, *J* 8.8 Hz, 2 Hz), 7.99–7.97 (m, 2H), 7.87–7.68 (m, 5H), 7.60 (t, 2H, *J* 8 Hz); ^{13}C-NMR ($CDCl_3$, 100MHz): δ 196.07, 184.81, 153.70, 143.74, 143.45, 141.05, 137.92, 137.38, 132.95, 132.84, 132.48, 132.18, 130.99, 130.75, 130.23, 129.94, 129.40, 128.58, 128.12, 126.69, 126.36, 123.01; IR (KBr): ν 3050, 1650, 1600, 1445, 1320 cm^{-1}; Anal. Calcd. for $C_{27}H_{16}N_2O$: C, 84.36; H, 4.20; N, 7.29. Found: C, 84.48, H, 4.183, N, 7.375.

9-Benzoylacenaphtho[1,2-*b*]quinoxaline (13). ^{1}H-NMR ($CDCl_3$, 400MHz): δ 8.61 (d, 1H, *J* 1.6 Hz), 8.50 (d, 1H, *J* 6.8 Hz), 8.44 (d, 1H, *J* 6.8 Hz), 8.34 (d, 1H, *J* 8.8 Hz), 8.28 (dd, 1H, *J* 8.6 Hz, 2 Hz), 8.18 (dd, 2H, *J* 8 Hz, 6 Hz), 7.96–786 (m, 4H), 7.67 (t, 1H, *J* 7.6 Hz), 7.57 (t, 2H, *J*

7.6 Hz); FT-IR (KBr): ν 3038, 1646, 1595, 1437, 1300 cm^{-1}; Anal. Calcd for $C_{25}H_{14}N_2O$: C, 83.78; H, 3.94; N, 7.82. Found: C, 83.46, H, 3.745, N, 7.607.

3-Naphthyl-7-benzoyl-quinoxaline (14). ^{1}H-NMR ($CDCl_3$, 400MHz): δ 9.31 (s, 1H), 8.59 (s, 1H), 8.35 (s, 2H), 8.23 (d, 1H, *J* 9.2 Hz), 8.06 (d, 1H, *J* 8.4 Hz), 8.01 (d, 1H, *J* 6.4 Hz), 7.94 (d, 2H, *J* 8 Hz), 7.48 (d, 1H, *J* 7.2 Hz), 7.68 (t, 2H, *J* 7.6 Hz), 7.60–7.53 (m, 4H); ^{13}C-NMR($CDCl_3$, 100MHz): δ195.65, 155.97, 147.77, 144, 140.41, 138.31, 137.08, 134.61, 134.07, 133.01, 132.38, 131.01, 130.67, 130.40, 130.23, 130.14, 128.90, 128.79, 128.60, 127.46, 126.89, 126.54, 125.46, 124.89; IR (KBr): ν 3050, 1697, 1651, 1446,1289 cm^{-1}; Anal. Calcd for $C_{25}H_{16}N_2O$: C, 83.31; H, 4.47; N, 7.77. Found: C, 82.63, H, 4.523, N, 6.903.

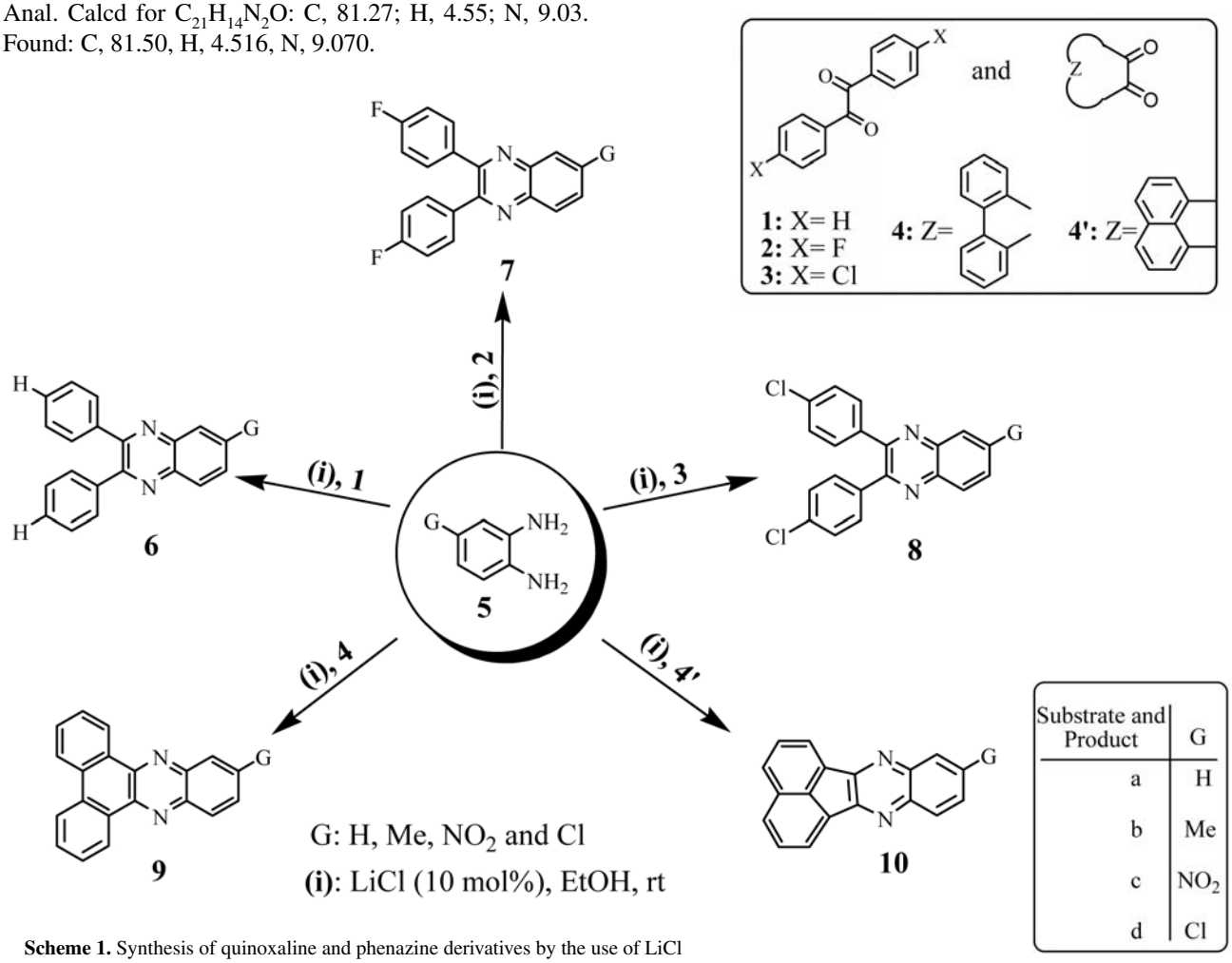
3-Phenyl-7-benzoyl-quinoxaline (15). ^{1}H-NMR ($CDCl_3$, 400MHz): δ 9.43 (s, 1H), 8.51 (s, 1H), 8.28–8.25 (m, 4H), 7.29 (s, 1H), 7.91 (d, 1H, *J* 1.2 Hz), 7.68–7.53 (m, 6H); ^{13}C-NMR ($CDCl_3$,100MHz): δ 195.63, 153.31, 144.41, 144.16, 140.63, 137.88, 137.12, 136.24, 132.89, 132.33, 130.82, 130.26, 130.16, 130.09, 129.30, 128.54, 127.77, 127.60; IR (KBr): ν 3053, 1650, 1595, 1454, 1294 cm^{-1}; Anal. Calcd for $C_{21}H_{14}N_2O$: C, 81.27; H, 4.55; N, 9.03. Found: C, 81.50, H, 4.516, N, 9.070.

3. Results and Discussion

In continuation of our previous studies on synthesis of organic compounds,[37–39] we found that lithium chloride (LiCl) can be used as an efficient and very cheap catalyst for the rapid condensation of aromatic 1,2-dicarbonyl compounds **1-4'** with *o*-phenylenediamines **5** at room temperature to afford dibenzo[*a,c*]phenazine and quinoxaline derivatives **6–10** in good to excellent yields (Scheme 1).

To determine simple and suitable conditions for the preparation of 1,4-aryldiazine derivatives using LiCl as a Lewis acid catalyst, the treatment of benzil **1** with *o*-phenylenediamine **5a** was chosen as a model reaction (Table 3, entry 1).

In order to compare the catalytic efficiency of LiCl with other lithium salts, a variety of lithium salts were first investigated (Table 1). The data of this study reveal that LiBr under conventional conditions afford the product **6a** with longer reaction time in moderate yield (50%, Table 1, entry 2). The use of LiCl also promoted the reaction to a reasonable extent (Table 1, entry 3), but the other catalysts such as Li_2SO_4 and LiI did not work well.



Scheme 1. Synthesis of quinoxaline and phenazine derivatives by the use of LiCl

Table 1. Comparison of efficiency of various lithium salts (10 mol %) in synthesis of **6a**

Entry	Catalyst	Time (min)	Yield (%)[a]
1	Li_2SO_4	120[b]	30
2	LiBr	100	50
3	LiCl	42	92
4	LiI	120[b]	30

[a] Refers to isolated yields. [b] Not completed.

However, according to the above results (Table) and from the economic point of view (because the LiCl is inexpensive), it can be concluded that the LiCl is better than the other ones. Furthermore, the effect of several solvents on reaction rates and product yields was also investigated (Table 2).

Table 2. Comparison of several solvents in synthesis of **6a** using LiCl (10 mol%) at room temperature

Time (min)	Yield (%)[a]	Solvent	Entry
42	92	EtOH	1
60	87	MeOH	2
300	30	H_2O	3
135	80	THF	4
110	85	CH_2Cl_2	5
115	85	$CHCl_3$	6
135	80	CH_3CN	7

[a] Refers to isolated yields.

According to the above results, it was observed that the condensation reaction can be efficiently carried out in ethanol by adding 10 mol % of the catalyst in a short time span of 42 min. The use of excessive amounts of the catalyst did not have a marked influence on the product yield. The probable reason for this is the coordination of excessive catalyst to the diamine.

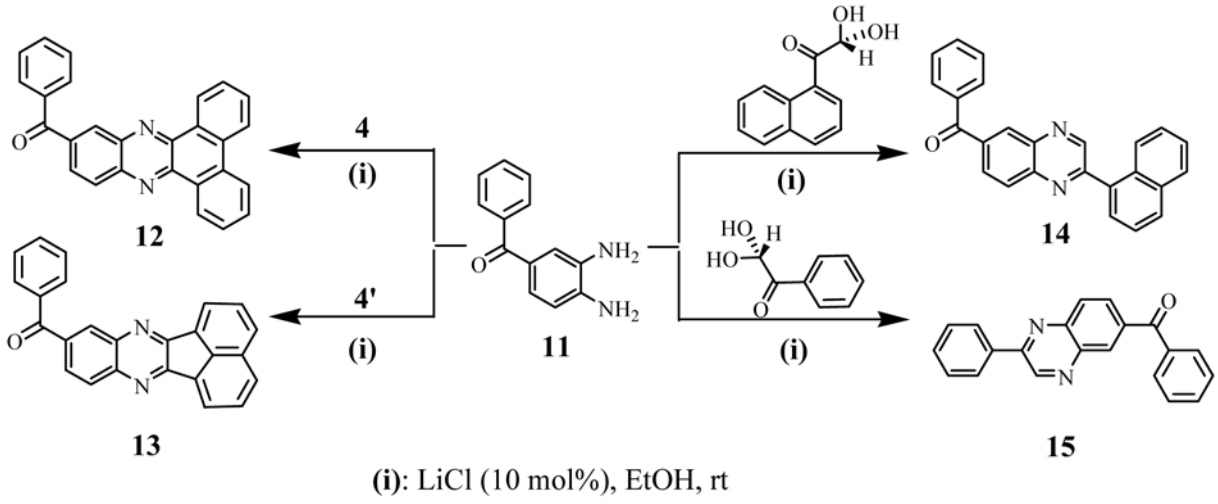
In order to show the versatility of this method, after optimizing the reaction conditions, we have examined different aromatic 1,2-dicarbonyls with *o*-phenylenediamines at room temperature in ethanol. The results are summarized in Table 3.

Table 3. Synthesis of phenazine and quinoxaline derivatives using LiCl (10 mol%) at room temperature

Entry	Product[a]	Time (min)	Yield (%)[b]	mp (°C) (Literature)
1	**6a**	42	92	128–130 (130–131)[20]
2	**6b**	48	86	115–117 (116–117)[23]
3	**6c**	97	88	190–192 (192–193)[22]
4	**6d**	39	90	115–117 (115–116)[22]
5	**7a**	40	88	134–136 (135–137)[23]
6	**7b**	43	85	163–165 (165–167)[23]
7	**8a**	45	92	190–192 (195–196)[23]
8	**8b**	45	90	175–177 (178–180)[24]
9	**8c**	90	95	174–176 (175–176)[24]
10	**9a**	26	95	224–226 (223–225)[22]
11	**9b**	42	95	217–219 (208–210)[22]
12	**9c**	365	81	256–258 (253–255)[25]
13	**9d**	8	84	243–244 (241–242)[25]
14	**10a**	32	90	237–239 (238–240)[22]
15	**10b**	42	95	230–232 (>300)[22]
16	**10c**	370	84	320–321 (>300)[24]
17	**10d**	8	82	227–228 (224–225)[25]

[a] Identified by comparison with authentic samples. [b] Refers to isolated yields.

In additional investigation three novel 1,4-aryldiazines such as phenazine **12** and qunioxalines **14** and **15** were prepared by the use of 4-benzoyl-1,2-phenylenediamine **11**, under the same conditions (Scheme 2). The results are summarized in Table 4.



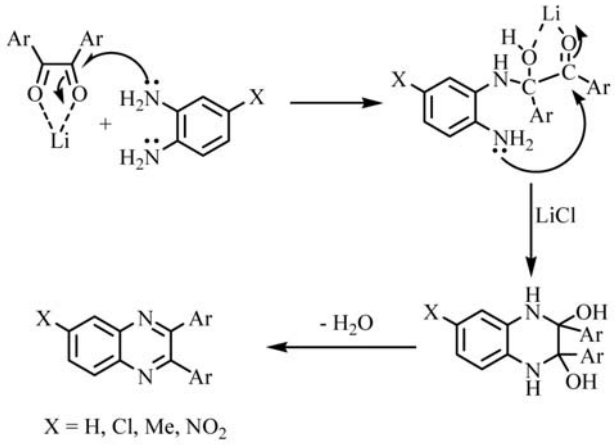
Scheme 2. Synthesis of new 1,4-aryldiazine derivatives

Table 4. Synthesis of new 1,4-aryldiazine derivatives using LiCl (10 mol %) at room temperature

Entry	Product	Time (min)	Yield (%)[a]	mp °C (Literature)
1	**12**	40	97	245–247
2	**13**	45	95	250–251 (255–256)[26]
3	**14**	45	93	166–167
4	**15**	40	95	144–145

[a] Refers to isolated yields.

Although the general mechanistic details are not fully understood, a plausible mechanism can be envisioned based on the previous studies. As indicated in Scheme 3, the first step is the activation of carbonyl groups by complexation of lithium with the dicarbonyl. This complexation facilitates the cleavage of the C=O bond. Thereafter, the nucleophilic *o*-phenylenediamine species reacts with the dicarbonyl in a substitution reaction. Moreover, LiCl plays a key role in promoting the dehydration steps to afford the product.



Scheme 3. Plausible mechanism for the synthesis of 1,4-aryldiazines using LiCl

It should be mentioned that our efforts for the synthesis of ,4-aryldiazines by using aliphatic 1,2-dicarbonyls through this method were unsuccessful.

4. Conclusion

In summary, we have presented a new application of lithium chloride (LiCl) as a heterogeneous catalyst for the synthesis of many phenazines and quinoxalines based on the condensation of aromatic 1,2-dicarbonyl compounds with *o*-phenylenediamines under mild reaction conditions. The main advantage of the present method is the elimination of expensive catalysts, corrosive liquid acids, and special equipment. Moreover, other advantages, such as using the inexpensive, available, and stable catalyst, simple reaction conditions, high product yields, and short reaction times, make this method a valid contribution to the existing methodologies.

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Povzetek

V prispevku je predstavljena enostavna in učinkovita priprava nekaterih 1,4-arildiazinov s kondenzacijo 1,2-arildiaminov z aromatskimi 1,2-dikarbonilmimi spojinami v prisotnosti litijevega klorida kot heterogenega katalizatorja. Na ta način so pripravili nekatere nove in tudi že znane dibenzo[*a,c*]fenazine in kinoksaline. Prednosti predstavljene metode so dobri izkoristki reakcij, kratki reakcijski časi in enostavna izolacija produktov.