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Facile Method of Quinoxaline Synthesis Using Phenol as a New, Efficient and Cheap Catalyst at Room Temperature

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Abstarct A simple, highly efficient and green procedure for the condensation of aryl and alkyl 1,2-diamines with α-diketones in the presence of catalytic amount of phenol(20 mol%) at room temperature is described. Using this method, quinoxaline derivatives as biologically interesting compounds are produced in high to excellent yields and short reaction times.

Keywords Quinoxaline, Phenol, 1,2-Diamine, A-Diketone, Green Chemistry

1. Introduction

Quinoxaline derivatives are a very important class of nitrogen-containing compounds and have been widely used in dyes[1] pharmaceuticals[2-3] and electrical/photochemical materials[4-9]. Quinoxaline ring moiety constitute part of the chemical structures of various antibiotics such as Echinomycin, Levomycin and Actinoleutin[10-11] that are known to inhibit growth of gram positive bacteria and are active against various transplantable tumors. A number of synthetic strategies have been developed for the preparation of substituted quinoxalines[12-14]. By far, the most common method relies on the condensation of an aryl 1,2-diamine with a 1,2-dicarbonyl compound in refluxing ethanol or acetic acid for 2–12h giving 34–85% yields[15]. Recently, Heravi *et al.*[16] and More *et al.*[17] reported greener methods for the synthesis of quinoxaline derivatives in green solvents (EtOH/H_2O), using copper sulphate pentahydrate and cerium (IV) ammonium nitrate as catalysts, respectively. 2,3-Disubstituted quinoxalines have also been prepared by Suzuki–Miyaura coupling reaction[18], condensation of o-phenylenediamines and 1,2-dicarbonyl compounds in MeOH/AcOH under microwave irradiation[19], iodine catalyzed cyclocondensation of 1,2- dicarbonyl compounds and substituted o-phenylene diamines in DMSO[20], CH_3CN[21]. Different catalysts used for quinoxaline synthesis such as IBX[22], Oxalic Acid[23], SBSSA[24], Microwave/I_2[25], $SnCl_2/SiO_2$[26], I_2[27], Ultrasound Irradiaton[28], NH_4Cl[29], $(NH_4)_6Mo_7O_{24}.4H_2O$[30], Citric acid[31], ionic liquid[32], Bentonit Clay K-10[33], AcOH[34] and BSA[35]. We disclose herein our results for the synthesis of quinoxalines using catalytic amounts of phenol in water as an acidic solution at room temperature.

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2. Methods

2.1. General

IR spectra of the compounds were obtained on a Shimadzu IR-435 spectrometer using a KBr disk. The ^1H nuclear magnetic resonance (^1H NMR) spectra were recorded on a Bruker AQS 300 Avance instrument at 300 MHz in dimethyl sulfoxide (DMSO-d_6) using tetramethylsilane as an internal standard. The progress of reaction was followed with thin-layer chromatography (TLC) using silica gel SILG/UV 254 and 365 plates. All the products are known compounds and were characterized by comparing the IR, ^1H NMR, and ^{13}C NMR spectroscopic data and their melting points with the literature values.

2.2. General Procedure of Preparation of Quinox-Alines

A solution of aromatic o-diamine (1mmol) and a 1,2-dicarbonyl compound (1mmol) in ethanol:water (7:3, 10 mL) was stirred at room temperature in the presence of catalytic amount of phenol (20 mol%, 0.01 g). The progress of the reaction was monitored by TLC (n-hexan-ethylacetate 20:1). After completion of the reaction, water (20 mL) added to the mixture and was allowed to stand at room temperature for 30 min. During this time, crystals of the pure product were formed which were collected by filtration and dried. For further purification, the products recrystallized from hot ethanol.

2.3. Selected Specteral Data

2.3.1. 2,3-Diphenylquinoxaline(1Q)

white solid m.p 125-127[lit. 128-129][22], FT-IR (KBr): 1556 cm^{-1}; ^{1}H-NMR (FT-300 MHz, $CDCl_3$/TMS): dppm7.33977(bs, 6H, Ar-H) 7.54183(bs, 4H, Ar-H) 7.74584(bs, 2H, Ar-H) 8.20007(bs, 2H, Ar-H) ; ^{13}C NMR (300 MHz, $CDCl_3$): 128.290, 128.896, 129.121, 129.913, 130.066, 138.921, 141.115, 153.384; MS: *m/z* = 282 (M+).

2.3.2. 6-Methyl-2,3-diphenylquinoxaline(2Q)

brown solid m.p 113-115[lit. 116-117][24], FT-IR (KBr): 1619 cm^{-1}(stretching C=N); ^{1}H-NMR (FT-300 MHz, $CDCl_3$/TMS): dppm 2.61(s, 3H, Ar-CH_3) 7.35(s, 6H, Ar-H) 7.55(d, J=6.48, 4H, Ar-H)7.60(s, 1H, Ar-H) 7.98(s, 1H, Ar-H) 8.09(d, J=8.4, 1H, Ar-H); ^{13}C NMR (300 MHz, $CDCl_3$): 21.948, 128.040, 128.244, 128.663, 128.731, 129.903, 129.915, 132.321, 139.246, 139.728, 140.486, 141.289, 152.552, 153.289; MS: *m/z* = 296 (M+).

2.3.3. 6-Nitro-2,3-diphenylquinoxaline(5Q)

red solid m.p 185-187[lit. 185-187][24], FT-IR (KBr): 1656 cm^{-1}(stretching C=N); ^{1}H-NMR (FT-300 MHz, $CDCl_3$/TMS): dppm 7.38(bs, 6H, Ar-H) 7.56(bs, 4H, Ar-H) 8.28(bs, 1H, Ar-H) 8.45(bs, 1H, Ar-H) 9.02(bs, 1H, Ar-H); ^{13}C NMR (300 MHz, $CDCl_3$):; MS: 123.269, 125.512, 128.450, 129.667, 129.854, 129.953, 130.666, 137..950, 139.870, 143.390, 147.801, 155.621, 156.176; MS: *m/z* = 327 (M+).

2.3.4. 2,3-bis(4-Methoxyphenyl)quinoxaline(8Q)

yellow solid m.p 134-136[lit. 148-150][36], FT-IR (KBr): 1615 cm^{-1}(stretching C=N); ^{1}H-NMR (FT-300 MHz, $CDCl_3$/TMS): dppm 3.85(s, 6H, 2×CH_3) 6.87(d, J=7.77, 1H, Ar-H) 6.94 (d, J=7.77, 4H, Ar-H) 7.50 (d, J=7.14, 1H, Ar-H) 7.71(s, 1H, Ar-H) 7.93(d, J=7.62, 4H, Ar-H) 8.13(s, 1H, Ar-H); ^{13}C NMR (300 MHz, $CDCl_3$): 55.291, 55.615, 113.786, 114.300, 126.271, 128.875, 129.645, 131.307, 131.449, 132.315, 140.867, 152.926, 160.267, 164.867, 193.506; MS: *m/z* = 342 (M+).

2.3.5. Dibenzo[a,c]phenazine(15Q)

yellow solid m.p 224-226[lit. 223-225][21], FT-IR (KBr): 1604 cm^{-1}(stretching C=N); ^{1}H-NMR (FT-300 MHz, $CDCl_3$/TMS): dppm 7.71(s, 4H, Ar-H) 7.85(s, 2H, Ar-H) 8.35(s, 2H, Ar-H) 8.43(s, 2H, Ar-H) 9.33(s, 2H, Ar-H); ^{13}C NMR (300 MHz, $CDCl_3$): 122.875, 126.448, 128.023, 128.925, 129.356, 130.205, 130.661, 132.019, 141.323, 141.876; MS: *m/z* = 280 (M+).

2.3.6. 11-Methyl-dibenzo[a,c]phenazine(16Q)

brown solid m.p 219-221[lit. 208-210][24], FT-IR (KBr): 1624 cm^{-1}(stretching C=N); ^{1}H-NMR (FT-300 MHz, $CDCl_3$/TMS): dppm 2.65(s, 3H, CH_3) 7.72(bs, 5H, Ar-H) 8.04(s, 1H, Ar-H) 8.15(s, 1H, Ar-H) 8.47(s, 2H, Ar-H) 9.32(bs, 2H, Ar-H); ^{13}C NMR (300 MHz, $CDCl_3$): 20.058, 122.795, 126.032, 126.178, 127.793, 128.797, 129.978, 130.127, 131.727, 131.922, 132.368, 140.393, 140.573, 141.508, 141.971; MS: *m/z* = 294 (M+).

2.3.7. 11-Benzoil-dibenzo[a,c]phenazine(17Q)

yellow solid m.p 245-247, FT-IR (KBr): 1653, 1606 cm^{-1}(stretching C=N); ^{1}H-NMR (FT-300 MHz, $CDCl_3$/TMS): dppm 7.27(s, 1H, Ar-H) 7.60(s, 2H, Ar-H) 7.71(s, 2H, Ar-H) 7.79(s, 2H, Ar-H) 7.98(s, 2H, Ar-H) 8.35(s, 1H, Ar-H) 8.52(s, 3H, Ar-H) 8.69(s, 1H, Ar-H) 9.31(s, 1H, Ar-H) 9.44(s, 1H, Ar-H); ^{13}C NMR (300 MHz, $CDCl_3$): 123.048, 128.254, 128.601, 129.450, 130.220, 130.754, 131.056, 132, 916, 140.540, 140.980, 141.323, 141.876, 154.046; MS: *m/z* = 384 (M+).

2.3.8.2-Bromopyrido-[2,3-b]dibenzo[5,6-7,8]quinoxaline(18Q)

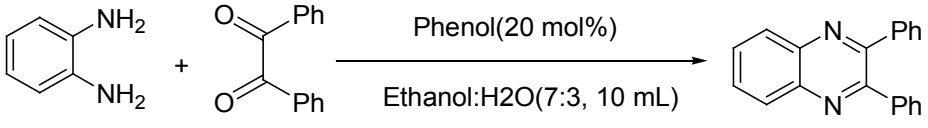
yellow solid m.p 216-218 ^{o}C, FT-IR (KBr): 1603 cm^{-1}(stretching C=N); ^{1}H-NMR (FT-300 MHz, $CDCl_3$/TMS): dppm 7.72(bs, 4H, Ar-H) 8.41(bs, 2H, Ar-H) 8.66(s, 1H, Ar-H) 9.06(s, 1H, Ar-H) 9.12(s, 1H, Ar-H) 9.30(s, 1H, Ar-H); ^{13}C NMR (300 MHz, $CDCl_3$): 122.858, 126.519, 127.292, 128.048, 129.026, 131.393, 139.507, 143.815, 155.170; MS: *m/z* = 360 (MH+).

2.3.9. 9-Methylacenaphto[1,2-b]quinoxaline(23Q)

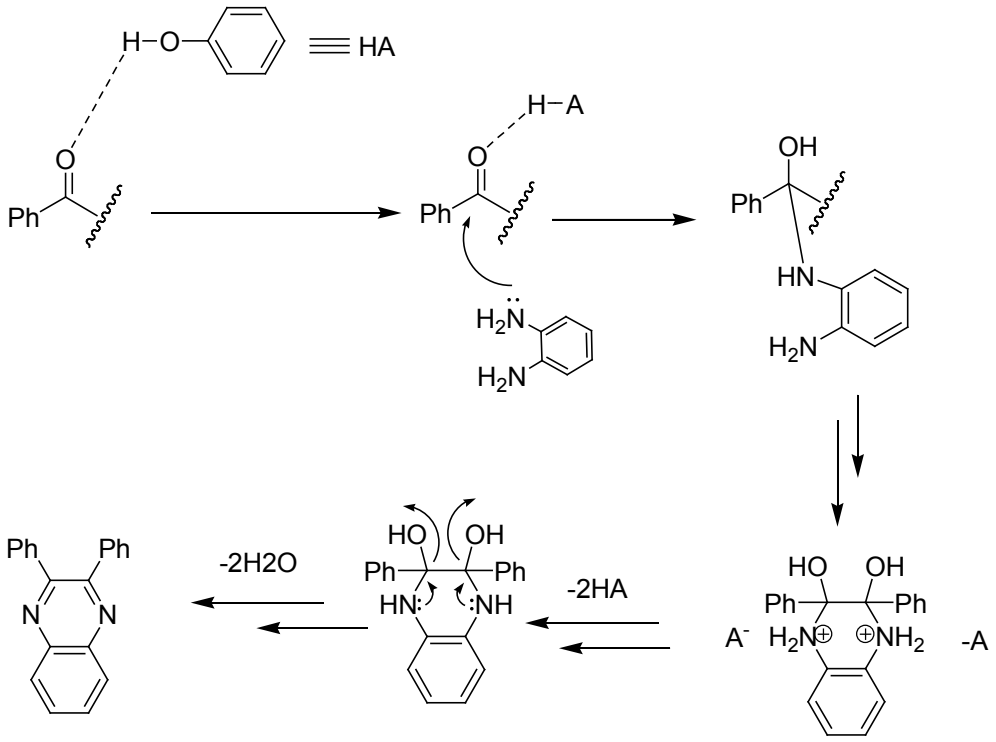
brown solid m.p 231-242[lit. >300 ^{o}C][24], FT-IR (KBr): 1626 cm^{-1}(stretching C=N); ^{1}H-NMR (FT-300 MHz, $CDCl_3$/TMS): dppm 2.56(s, 3H, CH_3) 7.49(d, J=8.31, 1H, Ar-H) 7.70(t, J=7.5, 2H, Ar-H) 7.88(s, 1H, Ar-H) 7.94(d, J=8.07, 2H, Ar-H) 7.99(d, J=8.61, 1H, Ar-H) 8.27(t, J=6.40, 2H, Ar-H); ^{13}C NMR (300 MHz, $CDCl_3$): 21.735, 121.583, 121.785, 128.484, 128.554, 128.938, 129.113, 129.284, 129.799, 131.250, 131.645, 136.097, 139.359, 139.666, 140.915, 153.035, 153.661; MS: *m/z* = 268 (M+).

2.3.10. Acenaphto[1,2-b]quinoxaline(25Q)

yellow solid m.p 241-242[lit. 238-240][24], FT-IR (KBr): 1614 cm^{-1}(stretching C=N); ^{1}H-NMR (FT-300 MHz, $CDCl_3$/TMS): dppm 7.74-7.81(m, 4H, Ar-H) 8.05(d, J=8.16, 2H, Ar-H) 8.20-8.23(m, 2H, Ar-H) 8.4(d, J=6.87, 2H, Ar-H); ^{13}C NMR (300 MHz, $CDCl_3$): 122.328, 128.679, 129.285, 129.442, 129.694, 129.900, 131.248, 136.479, 140.707, 153.595; MS: *m/z* = 254 (M+).

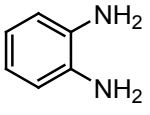
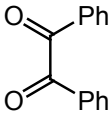
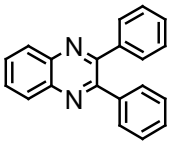
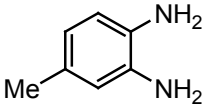
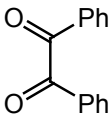
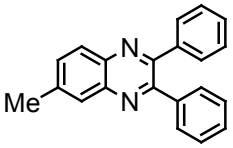
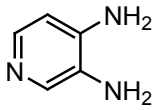
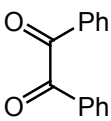
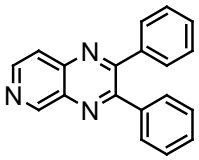
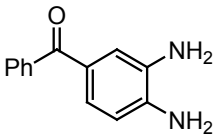
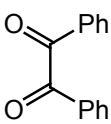
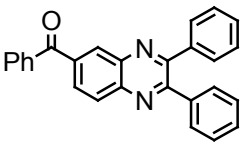


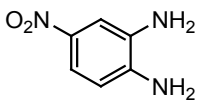
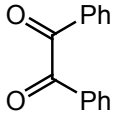
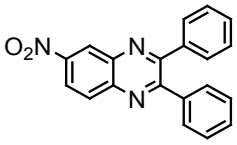
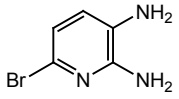
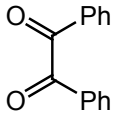
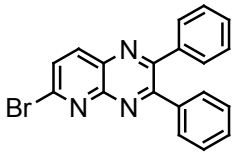
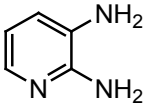
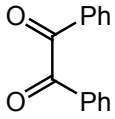
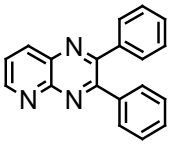
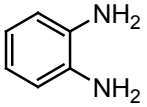
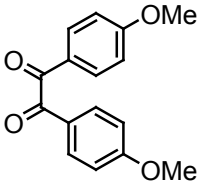
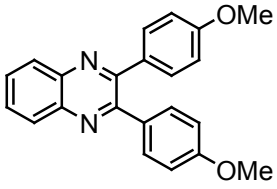
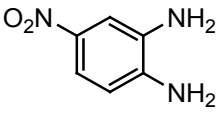
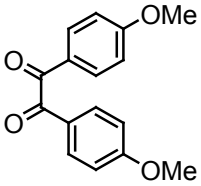
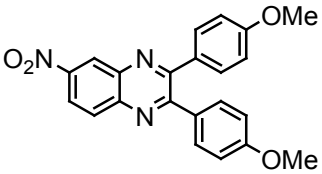
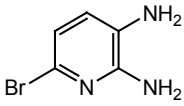
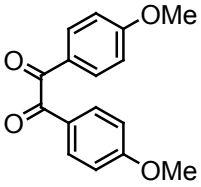
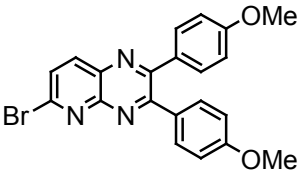
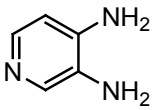
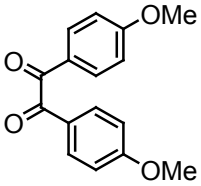
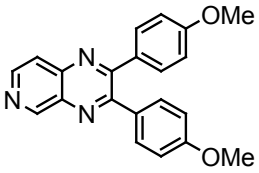
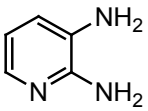
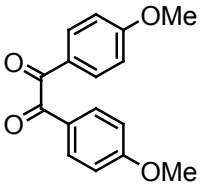
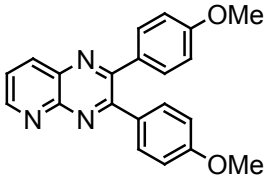
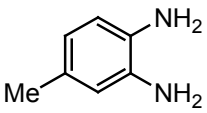
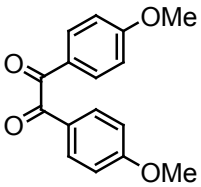
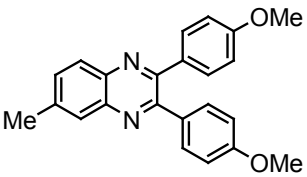
Scheme 1. 2,3-diphenylquinoxaline synthesis as a model by using phenol

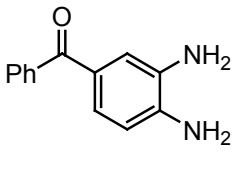
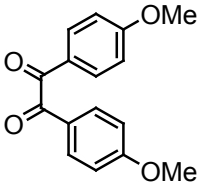
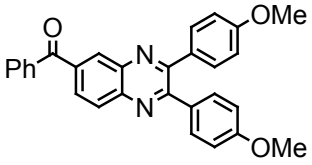
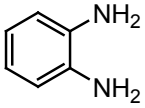
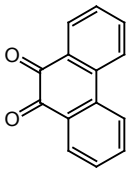
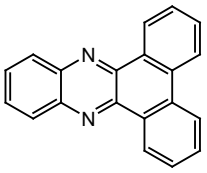
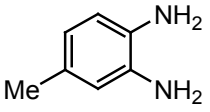
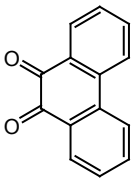
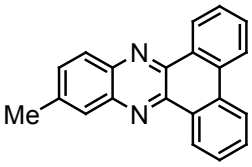
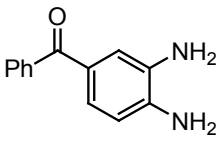
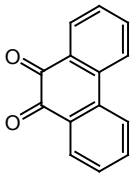
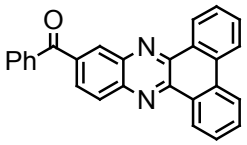
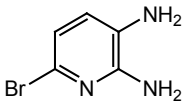
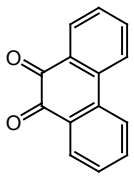
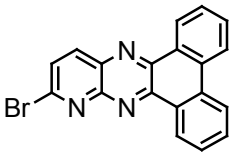
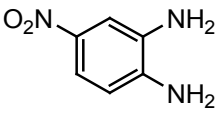
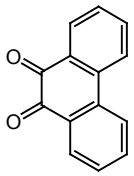
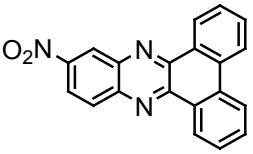
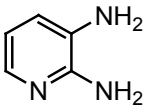
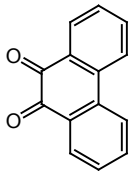
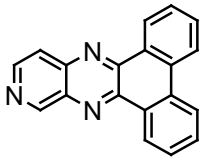
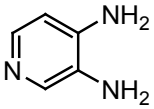
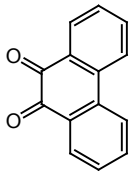
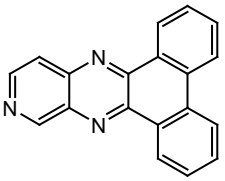


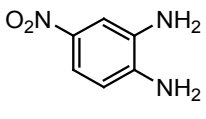
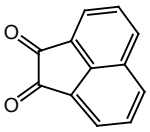
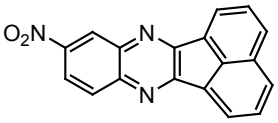
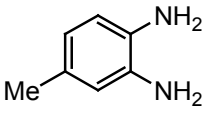
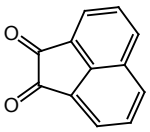
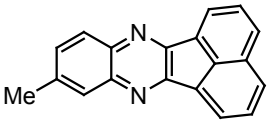
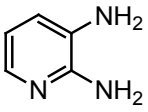
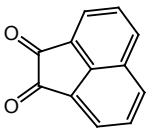
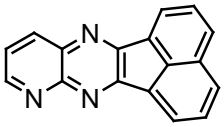
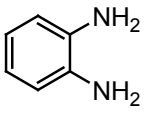
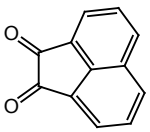
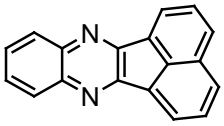
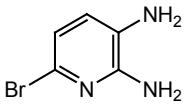
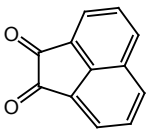
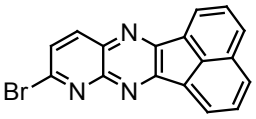
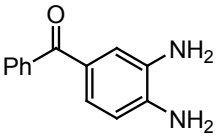
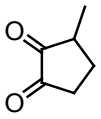
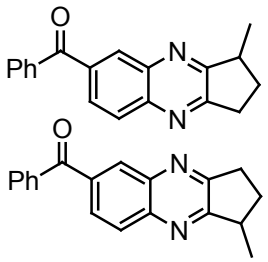
Scheme 2. Proposed mechanism of catalyst effect

Table 1. Quinoxalines synthesis from 1,2-diamines and α-diketones by using phenol (20 mol%)

Entry	Diamine (DA)	Diketone (DK)	Product (Q)	Time(h:min) Yield% m.p(Found) m.p[lit.]
1				00:02 98 125 -126 128 -129[20]
2				00:02 94 113 -115 116 -117[21]
3				08:13 93 127 -128 ------
4				02:30 94 140 -142 139-140[28]

5				03:45 92 185 -186 185 -187[21]
6				04:45 89 143 -145 ------
7				03:45 91 134 -137 141 -142[37]
8				03:20 91 134 -136 148 -150[22]
9				03:00 90 188 -189 ----[26]
10				05:45 88 124 -126 -----
11				07:15 90 120 -122 -----
12				02:30 00:02 131 -134 -----
13				00:05 90 123 -125 129 -131[21]

14				00:25 85 145 -147 ----[27]
15				00:02 99 225 -226 223 -225[21]
16				00:10 95 218 -220 208 -210[21]
17				03:00 98 245 -246 -----
18				00:45 86 215 -216 -----
19				00:20 93 259 -260 ------
20				02:00 91 221 -223 ------
21				03:00 94 159 -162 ------

22				04:30 93 321-323 ------
23				00:02 97 228 -229 >300[21]
24				03:00 93 229 -231 ------
25				00:25 91 235-236 238-240[21]
26				04:00 94 224-226 ------
27				20:00 85 243-245 -----

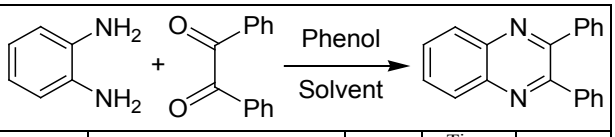
3. Result and Discussion

Table 2. The condensation of benzene-1,2-diamine (1 mmol) with benzil (1 mmol) in the presence of different catalysts (0.2 mmol, 20 mol%) in EtOH:H2O (7:3) at room tempera

Number	Catalyst name(cat%)	Time (mine)	Yield% [lit]
1	-----	600	----
2	**Phenol(20 mol%)**	**10**	**98**
3	Oxalic acid(20 mol%)	10	93[23]
4	$ZnCl_2$(20 mol%)	240	70
5	$Mn(OAc)_2$(20 mol%)	240	78
6	$CoCl_2$(20 mol%)	240	81
7	$CuCl_2$((20 mol%))	240	69
8	$Ni(OAc)_2$(20 mol%)	240	68
9	BSA(3 mol%)	5	98[35]

In order to find a suitable catalyst for the synthesis of quinoxalines from 1,2-diamines and α-diketones, the condensation of benzene-1,2-diamine with benzil was chosen as a model to provide compound 1Q (Scheme 1), and its behavior was studied in the presence of various catalysts in EtOH/H_2O at room temperature. The results are displayed in Table 1. As it can be seen from Table 2, phenol as an organic catalyst afforded the good results with respect to the inorganic catalysts. Total reactions are summarized in Table 1. Water is a desirable solvent for chemical reactions for reasons of cost, safety and environmental concerns, use of water in this reaction gave only moderate yields of products (67% after 3h). For chosen better solvent, different solvents were examined (Table 3). H_2O:Ethanol (3:7, 10 mL) was better solvent. The optimum yields of the products are obtained when 20 mol% of phenol is used.o-Phenylenediam ines and 1,2-dicarbonyl compounds with electron-donating or electron-withdrawing groups were used. As indicated in the Table 1 both electron rich and electron deficient 1,2-dicarbonyl compounds worked pretty well, mostly leading to high yields of products but withdrawing groups had lower yield.

Table 3. Solvent and catalyst optimize in synthesis 2,3-diphenylquinoxaline as a model



Number	Solvent	Cat%	Time (min)	Yield%
1	H2O(10 mL)	3	240	67
2	H2O(10 mL)	5	180	76
3	H2O(10 mL)	10	80	76
4	EtOH:H2O (1:1, 10 mL)	1	200	70
5	EtOH:H2O (1:1, 10 mL)	5	150	75
6	EtOH:H2O (1:1, 10 mL)	10	55	78
7	EtOH:H2O (4:1, 10 mL)	10	40	80
8	EtOH:H2O (4:1, 10 mL)	20	10	90
9	EtOH:H2O(7:3, 10mL)	20	5	98
10	EtOH:H2O(7:3, 10mL)	30	25	92
11	EtOH:H2O (9:1, 10 mL)	5	100	79
12	EtOH(10 mL)	10	60	81
13	Ethanol(10 mL)	15	35	78
14	Ethanol(10 mL)	20	20	85

4. Conclusions

In summary, we have developed an efficient method for the synthesis of quinoxaline derivatives via the condensation of 1,2-diamines with α-diketones. This new strategy has several advantages, such as excellent yield, mild reaction conditions, short duration of reaction time, low cost, simple experimental as well as isolation procedures, and finally, it is in agreement with the green chemistry protocols. These advantages will make this method become an attractive greener technique for the construction of quinoxalines and notably similar molecules, compared to the existing methods.

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