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One-Pot Three Component Synthesis of Symmetrical *N,N'*-Alkylidene Bisamides Catalyzed by $ZrOCl_2 \cdot 8H_2O$

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ABSTRACT

A highly efficient procedure for the preparation of *N,N'*-alkylidenebisamides in the presence of $ZrOCl_2 \cdot 8H_2O$ as a catalyst is described. *N,N'*-alkylidenebisamides have been prepared via one-pot three-component condensation reaction of various aldehydes and amides. It is the first successful report of $ZrOCl_2 \cdot 8H_2O$ has been used as Lewis acid catalyst for the preparation of symmetrical *N,N'*-alkylidenebisamides. All of the reactions proceeded in high yields, short reaction time, relatively non-toxic and easy workup are the advantages of the present catalyst.

Keywords: *N,N'*-alkylidenebisamides, aldehydes, amides, $ZrOCl_2 \cdot 8H_2O$.

1. INTRODUCTION

Amides and polyamides have been found as a key component of many biologically active and pharmaceutical compounds. In particular, symmetrical and unsymmetrical *N,N'*-alkylidenebisamides and their derivatives are found as key structural sub-units for the construction of peptidomimetic frameworks.^{1,2} Generally symmetrical alkylidenebisamides are synthesized by the direct reaction of aldehydes with the corresponding amides and similarly unsymmetrical alkylidenebisamides are prepared from aldehydes with different amides under suitable catalytic condition. In this topic, let us examine the various conditions of the catalysts³⁻⁵ such as sulfuric acid⁶, sulfonic acid⁷, triflic acid⁸, p-toluene sulfonic acid⁹, SiO_2 - $MgCl_2$ ¹⁰, hydrochloric acid¹¹, CC- or DCMT activated DMSO¹², silica coated magnetic $NiFe_2O_4$ nanoparticle supported

polyphosphoric acid¹³ and p-toluenesulfonic acid¹⁴ have been examined. However, each method has certain limitations with regards to scope and reaction conditions, such as, long reaction time, low yields, difficult work up and harsh reaction conditions.

We also reported reaction between aldehydes and amides under refluxing conditions giving symmetrical bisamides.¹⁵ These observations gave impetus to attempt reaction of aryl aldehydes and aryl amides using $ZrOCl_2 \cdot 8H_2O$ as a catalyst.

2. EXPERIMENTAL

Melting points were determined in open capillaries and are uncorrected. FT-IR spectra were recorded on a Thermo Mattson Satellite Fourier Transform-IR 3000 spectrophotometer using KBr tablets for solids and

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absorbencies are reported in cm^{-1} . NMR spectra were recorded at Bruker NMR spectrometer (300.13 and 75.47 MHz) or a Jeol-500 MHz spectrometer (500.13 and 125.77 MHz). Chemical shifts are reported in δ (ppm) relative to TMS (^1H) or DMSO- d_6 (^{13}C) as internal standards. Integrals are in accordance with assignments; coupling constants (J) are reported as values in Hz. Data for ^1H NMR are reported as follows: s = singlet, d = doublet, t = triplet, m = multiplet, br s = broad singlet. All ^{13}C NMR spectra were recorded with complete proton decoupling. Mass spectra were recorded on Q-TOF Micro Mass spectrometer. Yields refer to quantities obtained after chromatography.

2.1 General Procedure for the Preparation of N,N' -Alkylidenebisamides under Conventional Heating

A mixture of aldehyde **1** (2 mmol) and amide **2** (4 mmol), $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$ (0.05 g) was added in anhydrous toluene (5 mL). The mixture was heated in a water bath and the reaction was followed by TLC analysis (eluent: hexane/ethyl acetate, 3:2). After completion of the reaction, the mixture was cooled to room temperature and filtered to isolation of product and catalyst. The catalyst was separated from product by boiling ethanol. The crude solid product was purified by recrystallization in ethanol: water, 80:20, or ethanol.

2.2 Spectral Data of Compounds

N,N' - (Phenylmethylene) dibenzamide (**3a**):

White solid, Mp 235 °C; FT-IR (KBr) ν_{max} : 3282, 1654, 1539, 1353, 1271, 1052, 706 cm^{-1} ; ^1H NMR (300.13 MHz, DMSO- d_6): δ 9.07 (d, $J = 7.8$ Hz, 2H, 2(NH)), 7.93 (d, $J = 7.5$ Hz, 4H, Ar-H), 7.33-7.60 (m, 11H, Ar-H), 7.08 (t, $J = 7.7$ Hz, 1H, CH); ^{13}C NMR (75.47 MHz, DMSO- d_6): δ 165.6, 140.2, 133.8, 131.6, 128.3, 127.7, 127.4, 126.4, 58.6; HRMS: Calcdfor [M+H] $\text{C}_{21}\text{H}_{18}\text{N}_2\text{O}_2$ m/z : 331.1447; Found 331.1460.

N,N' - (4-Fluorophenylmethylene) dibenzamide (**3b**):

Yellowish solid, Mp 226-228 °C; FT-IR (KBr) ν_{max} : 3271, 2915, 2855, 1644, 1507, 1348, 1271, 1222, 1052, 827, 707 cm^{-1} ; ^1H NMR (300.13 MHz, DMSO- d_6): δ 9.04 (d, $J = 7.8$ Hz, 2H, 2 (NH)), 7.91 (d, $J = 8.1$ Hz, 2H, Ar-H), 7.88 (d, $J = 8.1$ Hz, 2H, Ar-H), 7.32-7.58 (m, 10H, Ar-H), 7.06 (t, $J = 7.8$ Hz, 1H, CH); ^{13}C NMR

(75.47 MHz, DMSO- d_6): δ 167.9, 165.6, 140.2, 134.1, 133.7, 131.6, 131.2, 128.3, 128.2, 127.7, 127.4, 127.4, 126.4, 58.6; FAB Mass: Calcdfor [M+Na] $\text{C}_{21}\text{H}_{17}\text{FN}_2\text{O}_2$ m/z : 371; Found 371.

N,N' - (4-4-Chlorophenylmethylene) dibenzamide (**3c**):

White solid, Mp 230-232 °C; FT-IR (KBr) ν_{max} : 3277, 2926, 2855, 1644, 1540, 1490, 1348, 1266, 1068, 800, 701 cm^{-1} ; ^1H NMR (300.13 MHz, DMSO- d_6): δ 9.09 (d, $J = 7.8$ Hz, 2H, 2 (NH)), 7.90 (d, $J = 7.5$ Hz, 4H, Ar-H), 7.46-7.54 (m, 10H, Ar-H), 7.05 (t, $J = 7.5$ Hz, 1H, CH); ^{13}C NMR (75.47 MHz, DMSO- d_6): δ 165.7, 139.2, 133.6, 132.3, 131.7, 128.6, 128.4, 128.3, 128.2, 127.5, 58.3; FAB Mass: Calcdfor [M+Na] $\text{C}_{21}\text{H}_{17}\text{ClN}_2\text{O}_2$ m/z : 388; Found 388.

N,N' - (4-Cyanophenylmethylene) dibenzamide (**3d**):

White solid, Mp 230-234 °C; FT-IR (KBr) ν_{max} : 3276, 1654, 1556, 1480, 1331, 1282, 1073, 706 cm^{-1} ; ^1H NMR (300.13 MHz, DMSO- d_6): δ 9.19 (d, $J = 7.5$ Hz, 2H, 2 (NH)), 7.93 (d, $J = 7.8$ Hz, 4H, Ar-H), 7.88 (d, $J = 8.4$ Hz, 2H, Ar-H), 7.67 (d, $J = 8.1$ Hz, 2H, Ar-H), 7.48-7.61 (m, 6H, Ar-H), 7.05 (t, $J = 7.5$ Hz, 1H, CH); ^{13}C NMR (75.47 MHz, DMSO- d_6): δ 165.9, 145.5, 133.5, 132.3, 131.7, 128.3, 128.2, 127.6, 127.5, 127.4, 118.7, 110.4, 58.6; FAB Mass: Calcdfor [M+Na] $\text{C}_{22}\text{H}_{17}\text{N}_3\text{O}_2$ m/z : 378; Found 378.

N,N' - (4-Nitrophenylmethylene) dibenzamide (**3e**):

White solid, Mp 263-264 °C; FT-IR (KBr) ν_{max} : 3265, 2926, 1643, 1512, 1347, 1276, 1073, 712 cm^{-1} ; ^1H NMR (300.13 MHz, DMSO- d_6): δ 9.23 (d, $J = 7.5$ Hz, 2H), 8.25 (d, $J = 8.7$ Hz, 2H, Ar-H), 7.92 (d, $J = 7.2$ Hz, 4H, Ar-H), 7.74 (d, $J = 8.7$ Hz, 2H, Ar-H), 7.47-7.60 (m, 6H, Ar-H), 7.08 (t, $J = 7.5$ Hz, 1H, CH); ^{13}C NMR (75.47 MHz, DMSO- d_6): δ 165.9, 147.5, 147, 133.4, 131.7, 128.3, 127.9, 127.5, 123.5, 58.4; HRMS: Calcdfor [M+H] $\text{C}_{21}\text{H}_{17}\text{N}_3\text{O}_4$ m/z : 376.1297; Found 376.1296.

N,N' - (4-Methylphenylmethylene) dibenzamide (**3f**):

White solid, Mp 240 °C; FT-IR (KBr) ν_{max} : 3254, 2920, 2849, 1644, 1512, 1282, 1057, 701 cm^{-1} ; ^1H NMR (300.13 MHz, DMSO- d_6): δ 8.96 (d, $J = 7.5$ Hz, 2H, 2(NH)), 7.89 (d, $J = 8.1$ Hz, 4H, Ar-H), 7.46-7.58 (m, 6H, Ar-H), 7.35 (d, $J = 7.8$ Hz, 2H, Ar-H), 7.19 (d, $J = 7.8$ Hz, 2H, Ar-H), 7.00 (t, $J = 7.5$ Hz, 1H, CH), 2.29 (s, 3H, CH₃); ^{13}C NMR (75.47 MHz, DMSO- d_6): δ 165.5, 137.3, 136.8, 133.9, 131.6, 128.8, 128.3, 127.4, 126.3, 58.5, 20.6; FAB Mass: Calcdfor [M+Na] $\text{C}_{22}\text{H}_{20}\text{N}_2\text{O}_2$ m/z : 367; Found 367.

N,N'- (4-Methoxyphenylmethylene) dibenzamide (**3g**):

Yellowish solid, Mp 230-232 °C; FT-IR (KBr) ν_{\max} : 3259, 3083, 2925, 2854, 1652, 1543, 1482, 1340, 1273, 1140, 1059, 909, 697 cm^{-1} ; ^1H NMR (300.13 MHz, DMSO- d_6): δ 8.99 (d, $J = 7.8$ Hz, 2H, 2(NH)), 7.90 (d, $J = 8.1$ Hz, 4H, Ar-H), 7.38-7.56 (m, 7H, Ar-H), 6.93-7.02 (m, 4H, Ar-H, CH), 3.74 (s, 3H, OCH₃); ^{13}C NMR (75.47 MHz, DMSO- d_6): δ 165.5, 158.8, 133.8, 132.3, 131.6, 128.3, 127.7, 127.4, 113.6, 58.3; FAB Mass: Calcdfor [M+Na] C₂₂H₂₀N₂O₃ m/z : 383; Found 383.

N,N'- (3,4-Dimethoxyphenylmethylene) dibenzamide (**3h**):

Yellowish solid, Mp 216-218 °C; FT-IR (KBr) ν_{\max} : 3277, 3080, 2926, 2859, 1650, 1542, 1486, 1343, 1273, 1136, 1049, 801, 707 cm^{-1} ; ^1H NMR (500.13 MHz, DMSO- d_6): δ 9.02 (d, $J = 8.4$ Hz, 2H, 2(NH)), 7.87 (d, $J = 9.2$ Hz, 4H, Ar-H), 7.27-7.57 (m, 9H, Ar-H), 7.02 (t, $J = 7.8$ Hz, 1H, CH), 3.49 (s, 6H, 2(OCH₃)); ^{13}C NMR (125.77 MHz, DMSO- d_6): δ 165.9, 151.3, 141.5, 134.7, 132.2, 128.9, 128.0, 126.9, 100.1, 58.7; FAB Mass: Calcdfor [M+Na] C₂₃H₂₂N₂O₄ m/z : 413; Found 413.

N,N'- (Dimethylaminophenylmethylene) dibenzamide (**3i**):

Brown solid, Mp 210-212 °C; FT-IR (KBr) ν_{\max} : 3279, 2923, 1650, 1533, 1273, 1139, 1051, 805, 705 cm^{-1} ; ^1H NMR (500.13 MHz, DMSO- d_6): δ 8.99 (d, $J = 8.4$ Hz, 2H, 2(NH)), 7.87 (d, $J = 6.9$ Hz, 4H, Ar-H), 7.34-7.53 (m, 10H, Ar-H), 7.01 (t, $J = 8.1$ Hz, 1H, CH), 2.5 (s, 6H, 2(CH₃)); ^{13}C NMR (125.77 MHz, DMSO- d_6): δ 162.3, 145.4, 131.9, 130.7, 128.9, 128.0, 127.5, 119.2, 59.5; FAB Mass: Calcdfor [M+Na] C₂₃H₂₃N₃O₂ m/z : 396; Found 396.

N,N'- (Furan-2-ylmethylene) dibenzamide (**3j**):

Yellowish solid, Mp 206-208 °C; FT-IR (KBr) ν_{\max} : 3277, 2928, 1650, 1489, 1340, 1273, 1137, 1051, 800, 706 cm^{-1} ; ^1H NMR (500.13 MHz, DMSO- d_6): δ 8.97 (d, $J = 6.9$ Hz, 2H, 2(NH)), 7.87 (d, $J = 7.7$ Hz, 4H, Ar-H), 7.27-7.51 (m, 9H, Ar-H), 7.03 (t, $J = 8.2$ Hz, 1H, CH); ^{13}C NMR (125.77 MHz, DMSO- d_6): δ 165.7, 152.7, 144.3, 140.7, 134.3, 130.1, 128.8, 127.9, 126.9, 119.2, 59.1; FAB Mass: Calcdfor [M+Na] C₁₉H₁₆N₂O₃ m/z : 343; Found 343.

N,N'- (Thiophen-2-ylmethylene) dibenzamide (**3k**):

Yellowish solid, Mp 208-210 °C; FT-IR (KBr) ν_{\max} : 3264, 3062, 2925, 2852, 1645, 1550, 1506, 1342,

1249, 1140, 1056, 931, 804, 702 cm^{-1} ; ^1H NMR (300.13 MHz, DMSO- d_6): δ 9.19 (d, $J = 7.8$ Hz, 2H, 2(NH)), 7.88-7.91 (m, 4H, Ar-H), 7.40-7.60 (m, 7H, Ar-H), 7.27 (t, $J = 7.8$ Hz, 1H, Ar-H), 7.121-7.131 (m, 1H, Ar-H), 7.00-7.035 (m, 1H, CH); ^{13}C NMR (75.47 MHz, DMSO- d_6): δ 170.8, 149.5, 138.8, 137.0, 133.7, 133.6, 132.7, 132.2, 131.7, 130.7, 130.1, 60.4; FAB Mass: Calcdfor [M+Na] C₁₉H₁₆N₂O₂ m/z : 359; Found 359.

N,N'- (Pyridin-4-ylmethylene) dibenzamide (**3l**):

Brown solid, Mp 208-212 °C; FT-IR (KBr) ν_{\max} : 3277, 2926, 2855, 1647, 1540, 1460, 1320, 1276, 1075, 970, 719 cm^{-1} ; ^1H NMR (300.13 MHz, DMSO- d_6): δ 9.05 (d, $J = 7.8$ Hz, 2H, 2(NH)), 7.90 (d, $J = 7.2$ Hz, 4H, Ar-H), 7.34-7.68 (m, 10H, Ar-H), 7.05 (t, $J = 7.8$ Hz, 1H, CH); ^{13}C NMR (75.47 MHz, DMSO- d_6): δ 165.6, 140.2, 133.7, 131.6, 128.3, 127.7, 127.4, 126.4, 58.6; FAB Mass: Calcdfor [M+Na] C₂₀H₁₇N₃O₂ m/z : 354; Found 354.

N,N'- (2-ferrocenylmethylene) dibenzamide (**3m**):

Brown solid, Mp 240-242 °C; ^1H NMR (500.13 MHz, DMSO- d_6): δ 8.98 (d, $J = 8.4$ Hz, 2H, 2(NH)), 7.86 (d, $J = 6.9$ Hz, 4H, Ar-H), 7.51-7.32 (m, 10H, Ar-H), 7.02 (t, $J = 8.1$ Hz, 1H, CH), 5.02 (s, 2H), 4.48 (s, 2H), 4.13 (s, 5H).

3. RESULTS AND DISCUSSION

Our aim was to develop new synthetic methods using heterogeneous catalysts to reduce risks to humans and the environment. To prepare *N,N'*-alkylidenebisamides and find the best reaction conditions, the reaction of benzamide and benzaldehyde was examined under various conditions and different quantities of initially, for the synthesis of bisamides, a reaction of benzaldehyde **1a** (1 mmol) with benzamide **2a** (2 mmol) in toluene (5 mL), under refluxed condition in the absence of ZrOCl₂ · 8H₂O catalyst gave only a trace of desired product was obtained due to equilibrium and the reaction takes longer time to complete (Table 1, entry 1). Similar results were obtained when, Amberlite IR-120, ZnCl₂, and ZrCl₄ were applied in this reaction (Table 1, entries 2–4). Ceric ammonium nitrate gave better yield of the product under the same reaction conditions. When Zirconium oxychloride were used as catalyst, much greater yield of the product **3a** in 80% yield was obtained (Table 1, entry 6) (Scheme 1) and found as an optimum conditions.

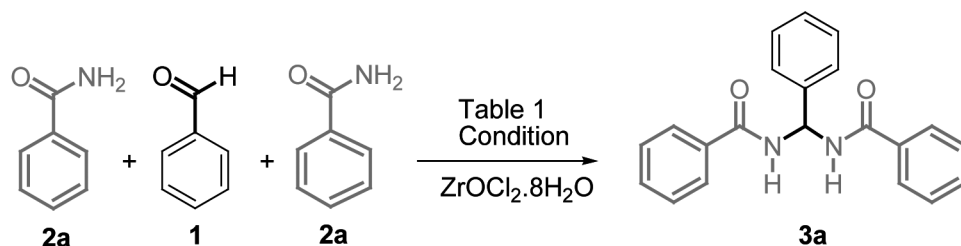
Scheme 1. Optimization of *N,N'*-alkylidenebisamides Synthesis

Table 1. One-pot Condensation Reaction between Benzaldehyde and Benzamide in the Presence of different Catalysts

Entry	Catalyst	Catalyst loading (g)	Time (h)	Yield %
1		No catalyst	6	10
2	Amberrlite IR-120	0.5	16	55
3	ZnCl ₂	0.05	12	60
4	ZrCl ₄	0.05	12	35
5	Cerium (IV) ammonium nitrate (CAN)	0.05	10	62
6	ZrOCl ₂ ·8H ₂ O	0.05	4	80

To understand the effect of solvent, the reaction has been carried out with various solvents (Table 2, entries 1–6). The results indicated that different solvents affected the efficiency of the reaction. No product was observed in CH₃CN and DCM as solvents (Table 2, entries 1 and 5). However, a considerable amount of desired products were formed in EtOH, EtOAc and THF (Table 1, entries 2, 3 and 6). Best yield was obtained when the reaction was performed in refluxing toluene with lesser reaction time and found as optimum condition (Table 2, entry 4).

Table 2. Solvent effect on the reaction between benzaldehyde (1 mmol) and benzamide (2 mmol) catalyzed by ZrOCl₂·8H₂O^a

Entry	Solvent	Temp (°C)	Time (h)	Yield %
1	CH ₃ CN	80	16	0
2	EtOH	80	8	35
3	EtOAc	80	8	45
4	Toluene	115	4	80
5	CH ₂ Cl ₂	45	16	0
6	THF	65	12	30

[a] The reaction was carried out in 5mL of solvent at reflux. [b] Isolated yield.

Encouraged by these results, we explored a variety of aromatic aldehydes **1b–m**, benzamide **2a**, using the above catalysts afforded symmetrical *N,N'*-alkylidenebisamides **3b–m** in moderate to good yields. The reaction is outlined in Scheme 2 and the results are summarized in Table 2. Various aromatic aldehydes and benzamide used in the reaction for the synthesis of symmetrical *N,N'*-alkylidenebisamides are shown in Table 2. It has been observed that substitution at the 4-position of aromatic aldehyde produced no significant effect in yields; aldehydes possessing electron-donating groups or electron-withdrawing groups caused slightly lower yields. To further investigate the scope of the reaction, the reaction of heteroaryl aldehyde such as 2-thiophenealdehyde and furfural have also been explored and took part in the reaction to provide corresponding bisamides products **3j** and **3k**, respectively (Table 2, entries 10 and 11). Significantly, the reaction of pyridine-4-aldehyde under optimized condition afforded excellent yield of pyridine substituted *N,N'*-alkylidenebisamide **3l** (Table 2, entry 12). Interestingly, to diversify the reaction organometallic aldehyde such as ferrocenealdehyde with benzamide proceeded well and gave the desired product **3m** in good yield (Table 2, entry 13).

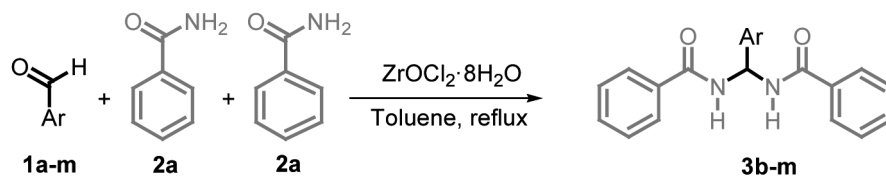
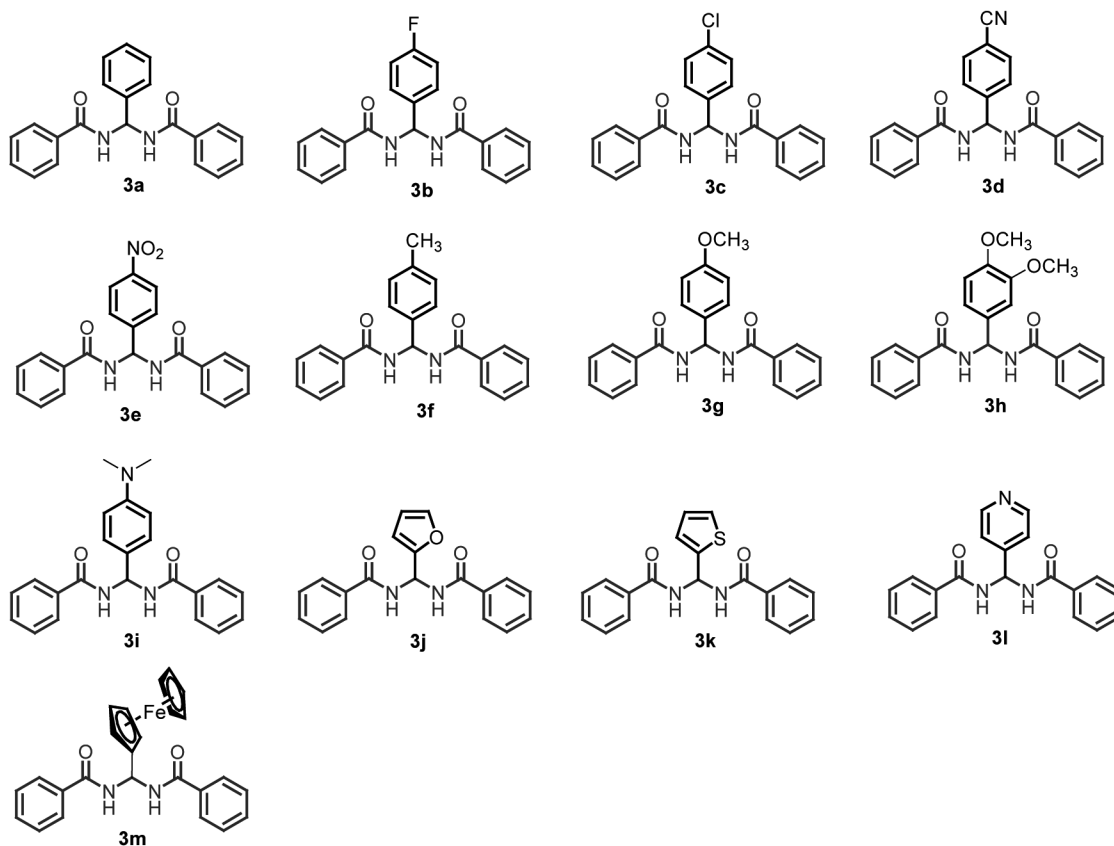
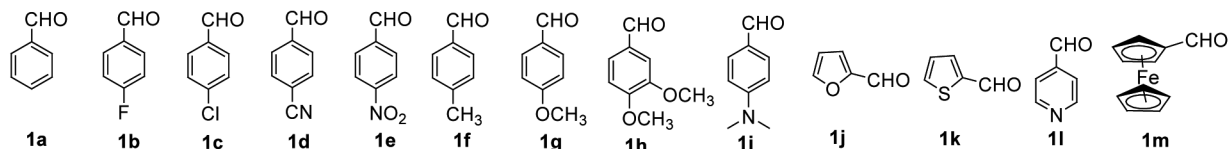
Scheme 2. Synthesis of Various *N,N'*-alkylidenebisamides

Table 3. Synthesis of Symmetrical Alkylidenebisamides

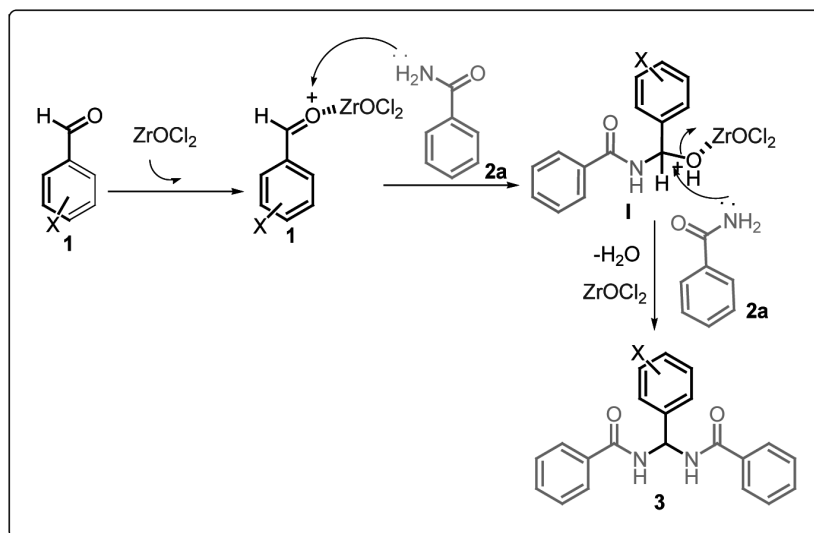


Entry	Ar-CHO	Amide	Products	Conventional heating by $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$	
				Time (h)	Yield %
1	1a	2a	3a	2	80
2	1b	2a	3b	5	52
3	1c	2a	3c	5	62
4	1d	2a	3d	4	65
5	1e	2a	3e	4	64
6	1f	2a	3f	4	78
7	1g	2a	3g	5	76

8	1h	2a	3h	5	55
9	1i	2a	3i	5.5	45
10	1j	2a	3j	4.5	55
11	1k	2a	3k	4.5	75
12	1l	2a	3l	5	68
13	1m	2a	3m	6	60

A plausible reaction mechanism of the reaction is shown in Scheme 3. Symmetrical *N,N'*-alkylidenebisamides derivative **3** was believed to be formed *via* an initial condensation of benzamide **2a** with aldehyde **1** (activated by the catalyst) to afford

intermediate **I**, which subsequently intermediate **I** was finally condenses with another molecule of benzamide **2a** to produce the *N,N'*-alkylidenebisamide **3**.



Scheme 3. A plausible reaction mechanism for the synthesis of symmetrical *N,N'*-alkylidenebisamides

4. CONCLUSION

We have demonstrated that $ZrOCl_2 \cdot 8H_2O$ has been successfully used as effective catalyst for the synthesis of symmetrical *N,N'*-alkylidenebisamides for the first time. This procedure has advantages in competition with the previously reported methods, in terms of yield, green catalyst, mild reaction condition, low cost, simple procedure, the use of a commercially available catalyst and simplicity of work-up. Further work using the catalyst for the synthesis of novel heterocycles is in progress in this laboratory.

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