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Ionic liquid 1-butyl-3-methylimidazolium bromide ([bmim]Br): a green and neutral reaction media for the efficient, catalyst-free synthesis of quinoxaline derivatives

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Abstract: Quinoxaline derivatives were produced in excellent yields and short reaction times *via* the condensation of 1,2-diamines with 1,2-diketones in the neutral ionic liquid 1-butyl-3-methylimidazolium bromide ([bmim]Br) under catalyst-free and microwave irradiation conditions.

Keywords: quinoxaline; neutral ionic liquid; catalyst-free; 1,2-diamine; 1,2-di-ketone; green chemistry.

INTRODUCTION

Currently, catalyst-free reactions are the subject of considerable interest because of advantages such as ease of the experimental procedure as well as workup, low cost, possibility of using acid- or base-sensitive substrates and environmentally benign processes.^{1–21} This valuable technique has been applied in several chemical transformations, such as the self-assembly of carbon nanotubes, carbon nanowires and carbon filaments,¹ conjugate addition of thiols to electron deficient alkenes,² synthesis of dithiocarbamates,³ synthesis of 10-aryl-2,7-dimethyl-4,5-dioxo-3,6,9-trioxa-3,4,5,6,9,10-hexahydroanthracenes,⁴ synthesis of 2--substituted benzimidazoles and bis-benzimidazoles,⁵ synthesis of di- and tri-substituted thiazoles,⁶ synthesis of 2-aryl/alkyl-4(3*H*)-quinazolinones,⁷ regioselective conversion of epoxides to vicinal halohydrins,⁸ transesterification of triglycerides,⁹ conversion of aldehydes to acylals,¹⁰ synthesis of 2-aminothiazoles,¹¹ α -amination of nitrogen heterocycles,¹² synthesis of 2-aminothiazoles,¹³ *N*-Boc deprotection of *N*-Boc-amines,¹⁴ condensation of indoles with alde-

1315



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ZARE et al.

1316

hydes,¹⁵ synthesis of *N*-sulfonyl imines,¹⁶ dehydration of benzyl alcohols into (*E*)-arylalkenes,¹⁷ chemoselective oxidation of aryl alcohols/acetates,¹⁸ synthesis of furo[2,3-*d*]pyrimidine-2,4(1*H*,3*H*)-diones,¹⁹ synthesis of 3-acetoacetylcoumarin derivatives²⁰ and synthesis of bis-pyrazolo[3,4-*b*:4',3'-*e*]pyridines.²¹

The elimination of volatile organic solvents in organic synthesis is the most important goal in green chemistry. One of the most efficient protocols to reach this aim is the substitution of volatile solvents with ionic liquids.²² In recent years, ionic liquids have received increasing attention as benign reaction media in organic synthesis due to their unique properties, including non-volatility, non-flammability, high thermal stability, high polarity because of their ionic nature, recyclability, non-contaminating nature and ability to dissolve a wide range of polar and non-polar materials.²² The application of microwave irradiation in synthetic chemistry is a fast-growing research area since this method has opened up the possibility of realizing the fast synthesis of organic compounds.²³ From the perspective of microwave chemistry, the strong polar nature of ionic liquids makes them ideal reaction medium in microwave-assisted organic reactions.^{15,16,22,23} Many interesting studies have recently been published in which the advantages of microwave irradiation and ionic liquids were combined.^{15,16,22,23}

Quinoxalines have attracted great interest as they possess various biological activities, such as antimycobacterial,²⁴ antibacterial,²⁵ antifungal,²⁵ anthelmintic,²⁶ antidepressant²⁷ and antitumor.²⁸ These compounds have also been used for the preparation of various dyes.²⁹ The condensation of 1,2-diamines with 1,2--diketones has been used as a useful synthetic route toward quinoxalines. For this transformation, several catalysts and reagents have been reported, including o-iodoxybenzoic acid,³⁰ ceric(IV) ammonium nitrate,³¹ Yb(OTf)₃,³² zirconium tetrakis(dodecyl sulfate),³³ zeolite,³⁴ sulfamic acid/MeOH,³⁵ (NH₄)₆Mo₇O₂₄·4H₂O,³⁶ H₆P₂W₁₈O₆₂·24H₂O,³⁷ LiBr,³⁸ Zn[L-proline],³⁹ polyaniline-sulfate salt⁴⁰ and iodine in DMSO.⁴¹ The condensation of 1,2-diamines with 1,2-diketones has been also realized using the acidic ionic liquid 1-butylimidazolium tetrafluoroborate as the solvent and catalyst.⁴² However, in this work, a large amount of the ionic liquid was applied (2 mL of the ionic liquid per mmol of 1,2-diketone). Furthermore, the catalyst used in this study was similar to most employed catalysts, *i.e.*, an acidic catalyst.⁴² Quinoxalines have also been prepared from benzene-1,2-diamine and 2-oxo-N-phenylalkane-thioamides using the acidic ionic liquid N,N,N-trimethyl-3-sulfo-1-propanaminimum hydrogen sulfate bearing two acidic group. In this work, among the different 1,2-diamines, only benzene-1,2--diamine was employed. Moreover, the preparation of the ionic liquid required a difficult two-steps procedure.⁴³ Other methods which have been applied for the synthesis of guinoxalines are heteroannulation of the nitroketene N,S-aryliminoacetals with POCl₃,⁴⁴ Bi-catalyzed oxidative coupling of epoxides with ene-1,2--diamines,⁴⁵ cyclization of 1,2-arylimino oximes of 1,2-dicarbonyl compounds⁴⁶

and from α -hydroxy ketones *via* a tandem oxidation process using Pd(OAc)₂ or RuCl₂(PPh₃)₃–TEMPO.⁴⁷ However, many of the synthetic protocols reported so far suffer from disadvantages such as the use of expensive reagents, the necessity of anhydrous conditions, prolonged reaction times, unsatisfactory yields, difficult experimental as well as workup procedures, the use of expensive and detrimental metal precursors and incompatibility with green chemistry protocols. Moreover, some methods involve multi-steps procedures. Therefore, development of an efficient, novel, safe, simple and catalyst-free method for the preparation of quino-xaline derivatives is desirable.

As a part of our ongoing program to develop the application of catalyst-free techniques in organic synthesis,^{15,16} herein, an efficient, green and simple catalyst-free method for the synthesis of quinoxaline derivatives from aromatic and aliphatic 1,2-diamines and 1,2-diketones in the neutral ionic liquid [bmim]Br under microwave irradiation is presented (Scheme 1). This synthesis represents a novelty in the synthesis of quinoxalines in a catalyst-free reaction in the neutral ionic liquid [bmim]Br, which is commercially available or easily prepared.



Scheme 1. The condensation of benzene-1,2-diamine with benzil.

RESULTS AND DISCUSSION

The condensation of benzene-1,2-diamine with benzil was selected as the model reaction to provide compound **1a**, for optimizing the reaction conditions (Scheme 1). At first, the model reaction was examined in [bmim]Br without a catalyst in an oil-bath (130 °C). However, under these conditions, the product was obtained in 69 % yield after 180 min. Changing the temperature or the reaction time did not increase the yield. Previously, the combination of microwave irradiation and ionic liquids was applied in several useful organic transformations.^{15,16,22,23} Thus, it was decided to use this technique in the catalyst-free synthesis of quinoxalines. For this purpose, a mixture of benzene-1,2-diamine and benzil in [bmim]Br was irradiated in a microwave oven at a maximum of 500 W (130 °C). Interestingly, the reaction yield increased to 95 % under these conditions (Table I). To determine the most suitable media for the reaction, the microwave-assisted synthesis of quinoxaline **1a** was checked in different ionic liquids, 1-methylimidazole (as a precursor of the used ionic liquids) as well as under solvent-free conditions at a maximum of 500 W of microwave power (130

ZARE et al.

°C). The results are summarized in Table I, from which it can be seen that the highest yield of **1a** was obtained in [bmim]Br. The results showed that Br[–] was the best anion part of the ionic liquid for the reaction, and *n*-butyl was the best alkyl chain of the cation part of the ionic liquid. Moreover, 1-methylimidazole cannot efficiently promote the reaction.

TABLE I. The condensation of benzene-1,2-diamine with benzil in some ionic liquids and 1-methylimidazole, as well as under solvent-free conditions

Entry	Solvent	Time, min	Yield ^a , %
1	[bmim]Br	30	95
2	[bmim]Cl	30	88
3	[bmim]I	30	84
4	[hmim]Br ^b	30	89
5	[omim]Br ^c	30	82
6	1-Methylimidazole (25 mol %) ^d	30	73
7	Solvent-free	30	67

^aIsolated yield; ^b1-hexyl-3-methylimidazolium bromide; ^c1-octyl-3-methylimidazolium bromide; ^dincrease in amount of 1-methylimidazole did not improve the yield

To assess the generality and efficiency of the catalyst-free protocol, different aromatic and aliphatic 1,2-diamines were reacted with structurally diverse 1,2-diketones. The results are given in Table II, from which it can be seen that all reactions proceeded efficiently and the desired products were obtained in good to excellent yields and in short reaction times. It was observed that electron-releasing substituents on the aromatic ring of the aromatic 1,2-diamines had no significant influence on the reaction results (Table II, entries 7–12); however, electron-withdrawing substituents slightly decreased the yields (Table II, entry 13). Moreover, aliphatic 1,2-diamines afforded the corresponding quinoxaline derivatives in high yields (Table II, entry 16).

TABLE II. The catalyst-free preparation of quinoxaline derivatives from 1,2-diamines and 1,2-diketones

Entry	1,2-Diamine	Product ^a	Time, min	Yield ^b , %	M.p., °C
1	NH ₂ NH ₂		30	95	130–131 (128–129) ³⁰
2		F (1 b)	30	96	132–134 (135–137) ³⁰

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1318

TABLE II. Continued						
Entry	1,2-Diamine	Product ^a	Time, min	Yield ^b , %	M.p., °C	
3	NH ₂ NH ₂		30	94	191–193 (195–196) ³⁹	
4			32	87	188–190	
5		(1d) Br	35	90	75–77 (78) ³⁵	
6			30	83	228–230	
7	Me NH ₂ NH ₂		30	96	116–117 (117–118) ³⁰	
8		(1g) Me N	35	87	139–140 (139–140) ³⁶	
9		(1h) Me F Me N	30	95	164–166 (163–165) ³³	
10		(1i) Me N N (1j) F	30	86	233–235 (233–235) ³⁸	

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1320

TABLE II. Continued					
Entry	1,2-Diamine	Product ^a	Time, min	Yield ^b , %	M.p., °C
11	Me NH ₂ Me NH ₂		25	96	176–178 (172) ³⁵
12		Me N Me N (11)	30	91	301–303 (302–304) ³³
13	O ₂ N NH ₂ NH ₂		30	78	190–192 (193–194) ³⁰
14	NH ₂ NH ₂		30	92	184–186 (187–188) ³¹
15		(1o)	40	85	132–134 (134) ³¹
16	NH ₂ NH ₂		40	84	168–170 (168–170) ³³
		(1p)			

^aThe structures of known compounds were identified by comparison of their melting points and spectral data with those in the authentic samples; ^bisolated yield

The interesting behavior of ionic liquids lies in the fact that they can be reused after simple washing with a suitable solvent, thus rendering the process more economical. The yields of compounds **1a** (model compound) in the 2^{nd} , 3^{rd} and 4^{th} uses of [bmim]Br were almost as high as in the first use.

Selected spectral data of the products

2,3-Diphenylquinoxaline (1a). White solid; yield: 95 %; m.p. 131–132 °C (lit.³⁰ m.p. 128–129 °C). Anal. Calcd. for $C_{20}H_{14}N_2$: C, 85.08; H, 5.00; N, 9.92 %. Found: C, 85.31; H, 5.16; N, 9.79 %. ¹H-NMR (250 MHz, CDCl₃, δ / ppm): 7.29–7.33 (6H, *m*), 7.48–7.54 (4H, *m*), 7.76–7.79 (2H, *m*), 8.20–8.23 (2H, *m*).



¹³C-NMR (62.5 MHz, CDCl₃, *δ* / ppm): 128.1, 128.7, 129.1, 129.9, 131.0, 139.6, 141.7, 153.2.

2,3-Bis(4-bromophenyl)quinoxaline (1d). Pale yellow solid; yield: 87 %; m.p. 188–190 °C. Anal. Calcd. for C₂₀H₁₂Br₂N₂: C, 54.58; H, 2.75; N, 6.36 %. Found: C, 54.79; H, 2.65; N, 6.25 %. ¹H-NMR (500 MHz, CDCl₃, δ / ppm): 7.45 (4H, dd, J = 8.5, 2.0 Hz), 7.62 (4H, d, J = 8.5 Hz), 7.88–7.92 (2H, m), 8.15–8.18 (2H, m). ¹³C-NMR (125 MHz, CDCl₃, δ / ppm): 124.2, 129.7, 130.9, 131.6, 132.2, 138.0, 141.5, 152.9.

Dibenzo[a,c]*phenayine* (*If*). Pale yellow solid; yield: 83 %; m.p. 228–230 °C. Anal. Calcd. for C₂₀H₁₂N₂: C, 85.69; H, 4.31; N, 9.99 %. Found: C, 85.43; H, 4.19; N, 10.14 %. ¹H-NMR (500 MHz, CDCl₃, δ / ppm): 7.75–7.7.83 (4H, *m*), 7.86–7.89 (2H, *m*), 8.33–8.37 (2H, *m*), 8.54–8.57 (2H, *m*), 9.41 (2H, *dd*, *J* = 7.9, 1.4 Hz). ¹³C-NMR (125 MHz, CDCl₃, δ / ppm): 123.3, 126.7, 128.3, 129.9, 130.2, 130.7, 130.7, 132.4, 142.6, 142.8.

9-Methylacenaphtho[1,2-b]quinoxaline (1j). Pale yellow solid; yield: 86 %; m.p. 233–235 (lit.: 233–235 °C³⁸). Anal. Calcd. for C₁₉H₁₂N₂: C, 85.05; H, 4.51; N, 10.44 %. Found: C, 85.23; H, 4.65; N, 10.32 %. ¹H-NMR (500 MHz, CDCl₃, δ / ppm): 2.60 (3H, *s*), 7.69 (1H, *d*, *J* = 7.8 Hz), 7.93–7.95 (2H, *m*), 8.00 (1H, *s*), 8.09–7.12 (1H, *m*), 8.27–8.31 (2H, *m*), 8.39–8.42 (2H, *m*). ¹³C-NMR (125 MHz, CDCl₃, δ / ppm): 22.3, 121.8, 122.3, 129.0, 129.1, 129.2, 129.5, 129.7, 129.7, 130.4, 131.8, 132.4, 136.7, 140.0, 140.1, 141.8, 153.7, 154.3.

9,10-Dimethylacenaphtho[1,2-b]quinoxaline (**1**l). Yellow solid; yield: 91 %; m.p. 301–303 °C (lit.³³ m.p. 302–304 °C). Anal. Calcd. for C₂₀H₁₄N₂: C, 85.08; H, 5.00; N, 9.92 %. Found: C, 84.81; H, 5.13; N, 10.09 %; ¹H-NMR (250 MHz, CDCl₃, δ / ppm): 2.51 (6H, *s*), 7.76–7.80 (2H, *m*), 7.89 (2H, *s*), 8.01–8.05 (2H, *m*), 8.32–8.36 (2H, *m*); ¹³C-NMR (62.5 MHz, CDCl₃, δ / ppm): 20.3, 121.5, 127.8, 128.0, 128.6, 128.9, 129.1, 139.5, 140.00, 148.5, 153.3.

2,3-Diphenylbenzo[g]quinoxaline (1n). Yellow solid; yield: 92 %; m.p. 184–186 °C (lit.³¹ m.p. 187–188 °C). Anal. Calcd. for C₂₄H₁₆N₂: C, 86.72; H, 4.85; N, 8.43 %. Found: C, 86.52; H, 4.94; N, 8.31 %. ¹H-NMR (250 MHz, CDC1₃, δ / ppm): 7.30–7.34 (6H, *m*), 7.48–7.53 (6H, *m*), 8.01–8.05 (2H, *m*), 8.59 (2H, *s*). ¹³C-NMR (62.5 MHz, CDCl₃, δ / ppm): 126.5, 127.4, 128.0, 128.3, 129.0, 129.7, 133.8, 136.9, 139.4, 153.7.

EXPERIMENTAL

All chemicals were purchased from Merck or Fluka Chemical Companies. The ionic liquids were prepared according to reported methods.⁴⁸ All reactions were performed using a laboratory microwave oven (MicroSYNTH, Milestone Company, Italy). The ¹H-NMR (250 or 500 MHz) and ¹³C-NMR (62.5 or 125 MHz) measurements were run on a Bruker Avance DPX-250 FT-NMR spectrometer. The microanalyses were realized on a Perkin-Elmer 240-B microanalyzer. The melting points were recorded on a Büchi B-545 apparatus in open capillary tubes.

1322

ZARE et al

General procedure for the catalyst-free preparation of quinoxalines

To a mixture of 1,2-diamine (6 mmol) and 1,2-diketone (6 mmol) in a microwave vessel was added [bmim]Br (2 g) and mixed carefully with a small rod. The resulting mixture was irradiated and stirred in a microwave oven at maximum of 500 W. The microwave oven was programmed to enhance the internal reaction temperature to 130 °C within 5 min, and then continue suitable irradiation (0–500 W) at this temperature, for the appropriate time. The combined times are showed in Table II. Subsequently, the reaction mixture was cooled to room temperature and extracted with Et₂O (3×50 mL). The combined organic extracts were then washed with brine (2×50 mL) and dried over MgSO₄. The solvent was recrystallized from EtOH (96 %) when required to give the pure product. After isolation of the product, the remaining Et₂O in [bmim]Br was evaporated and the ionic liquid was successfully employed for the next run under identical reaction conditions.

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извод

ЈОНСКА ТЕЧНОСТ 1-БУТИЛ-3-МЕТИЛИМИДАЗОЛИЈУМ-БРОМИД ([bmim]Br): ЕКОЛОШКИ И НЕУТРАЛАН РАСТВАРАЧ ЗА ЕФИКАСНУ СИНТЕЗУ ДЕРИВАТА ХИНОКСАЛИНА БЕЗ КАТАЛИЗАТОРА

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Деривати хиноксалина добијени су у кратком реакционом времену и у високом приносу кондензацијом 1,2-диамина и 1,2-дикетона у неутралној јонској течности, без катализатора под условима озрачивања микроталасима.

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ZARE et al.

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