New Access to the One-Pot Solvent-Free Synthesis of 4,5-Dihydro-pyrido[2,3-*b*][1,4]diazepines and 2,3-Dihydro-benzo[*b*][1,4]diazepines by Microwave Irradiation

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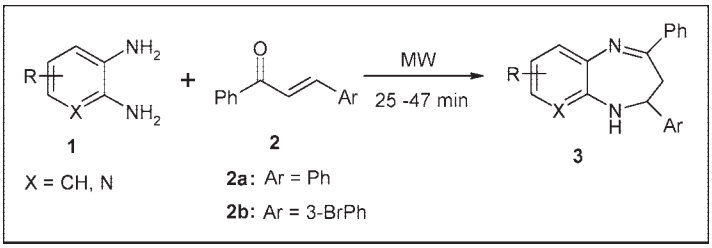

A series of diversely substituted pyridodiazepines and benzodiazepines have been synthesized in solvent and catalyst free microwave condition from aryl/heteroaryl diamines and β-aryl vinyl ketones. The yields of the reactions varied with the substituents attached to the aryl/heteroaryl diamines. This common protocol is equally effective for the synthesis of pyridodiazepines and benzodiazepines with pharmacological interest, though in case of pyridodiazepines required time is comparatively higher than benzodiazepines.

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INTRODUCTION

The pyridodiazepines and benzodiazepines are important class of compounds for their versatile use in biological [2] and therapeutic purpose [3]. Different substituted benzodiazepines are effective as tranquilizing, anti-inflammatory, and anticonvulsant agents. These compounds are also important synthons for the synthesis of oxadiazol, triazol [4] like various fused heterocyclic ring benzodiazepine derivatives. In industry, benzodiazepine derivatives are used as dyes in photography [5].

For the considerable applications in medicinal chemistry, scientific community has a continuous drive for efficient synthetic methods of such compounds. So far the different methods, which have been developed for the synthesis of benzodiazepines include the condensation of 1,2-diamines with α,β-unsaturated carbonyl compounds [6], ketones and β-haloketones [7] in the presence of Al_2O_3-P_2O_5 [8], $SbCl_3$-Al_2O_3 [9], polyphosphoroic acid-SiO_2 [10], $Yb(Otf)_3$ [11], BF_3-etherate [12], MgO-$POCl_3$ [13], Zn [L-proline]$_2$ [14], Amberlyst-15 [15], $NaBH_4$ [16], ionic liquid [17], and AcOH [18]. Recently, alum [$KAl(SO_4)_2 \cdot 12H_2O$] is also used as a catalyst for the synthesis of benzodiazepines [19]. However, all these reactions are carried out under hazardous reaction conditions and in the presence of a catalyst. Among all the available reports, there are very few examples for the synthesis of pyridodiazepines [20]. But according to our knowledge, there is no report for the synthesis of pyridodiazepines under microwave conditions.

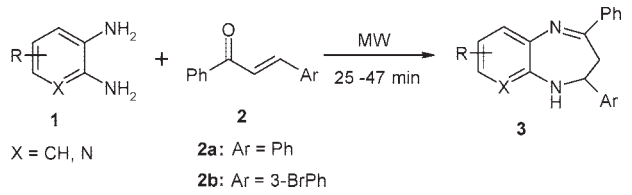
Nowadays, different organic synthesis has been carried out under microwave irradiation because of its very slight environmental impact and short reaction time [21]. This technique has been widely used in recent years for different organic transformations, including the synthesis of a wide range of heterocyclic compounds [22].

RESULTS AND DISCUSSION

In recent past, we have successfully exploited this microwave technique to develop new methodologies including the synthesis of different heterocyclic backbone [23]. Herein, we report the synthesis of pyridodiazepines and benzodiazepines by reacting heteroaryl/aryl diamines with β-aryl vinyl ketones in solid phase without using any catalyst and solvent under microwave irradiation (Scheme I). Previously microwave reactions for the synthesis of benzodiazepines from *o*-phenylenediamines with various ketones are carried out using Al_2O_3/P_2O_5 [8] or AcOH [18] catalyst. But in the present procedure benzodiazepines are synthesized in quite good yield without any catalyst.

In one representative procedure, pyridine-2,3-diamine (2 mmol) and β aryl vinyl ketone (2 mmol) are mixed thoroughly in solid phase and irradiated at 400 W for

Scheme 1. Synthesis of pyridodiazepines and benzodiazepines from 2,3-diaminopyridine and 1,2-diaminobenzene with β-aryl vinyl ketones (for R, X and Ar see Table 1).



27 min (Scheme 1, Table 1) in a microwave oven to afford 2,4-diphenyl-4,5-dihydro-3*H*-pyrido[2,3-b][1,4] diazepine (**3a**) in 80% yield, and no isomerisation product is obtained. The pyridodiazepines [**3**(**a**, **c**, **f** and **h**)] and benzodiazepines [24] [**3**(**b**, **d**, **e**, **g**, **i** and **j**)] are synthesized using this procedure with variable time, and power for each set of reaction and are also characterized by spectroscopic studies (Table 1).

In case of diaminopiridines the overall time for the microwave irradiation is higher than the unsubstituted 1,2-phenylenediamine to afford the respective diazepines. Similar trend is also observed in case of substituted 1,2-phenylenediamine. Here, both bromo (entries c, d, h and i) and nitro (entries e and j) groups may reduce the nucleophilicity of the amine group attached with the aromatic ring. This effect is very much pronounced in case of nitro group, which also affects the yield of the compounds **3e** and **3j**. The overall yield of the other diazepine compounds are varied from moderate to good, depending upon the reactivity of the starting diamines. The ^{1}H NMR spectra of the nitro group containing benzodiazepines (**3e** and **3j**) may tautomerise to the quinoid form of the compounds resulting the disappearance of N—*H* peak.

All these reactions probably proceed through Michael addition of amine to the β position of α,β-unsaturated ketone followed by condensation and dehydration (Scheme 2). In the case of 2,3 diaminopyridine, probably nucleophilic attack takes place through comparatively less reactive 2-amino group to the double bond of α,β-unsaturated ketone followed by cyclization of the more reactive amino group with the keto group to achieve only the substituted 4,5-dihydropyridodiazepine [23a,25]. The other isomer like substituted 2,3-dihydropyridodiazepine is not obtained under this reaction condition. The appearance of N*H* proton at δ 5.14 ppm in pyridodiazepine (**3a**) (more downfield because of amidine system *i.e* conjugated with pyridine ring ''N'') comparing with benzdiazepine (**3g**) with N*H* proton at 3.77 ppm also support the formation of 4,5 dihydropyridodiazepine. Formation of minor amount (5%) of aryl substituted benzimidazole (**4**) [26] by thermally induced ring contraction also favors the suggested mechanism in case of 1,2-phenylenediamine (**1a**) and β aryl vinyl ketone (**2b**) [27].

In conclusion we have developed a general straightforward method under microwave irradiation for the efficient synthesis of 4,5-dihydro-3*H*-pyrido[2,3-b][1,4] diazepines and 2,3-dihydro-1*H*-benzo[b][1,4]diazepines. Major advantage of this reaction is that same solvent and catalyst free condition is effective for both aryl and heteroaryl diamines. By this method different diazepines of pharmacological interest can be synthesized by varying the substituents at diamines, as well as the other substituted phenyl rings.

EXPERIMENTAL

General reaction procedure for the synthesis of 2,4 substituted-4,5-dihydro-3*H*-pyrido[2,3-*b*][1,4]diazepine/2,4 substituted-2,3-dihydro-1*H*-benzo[*b*][1,4]diazepine using β aryl vinyl ketone. A mixture of pyridine-2,3-diamine (218 mg, 2.0 mmol) (1a) and β aryl vinyl ketone (416 mg, 2.0 mmol) (2a) was thoroughly grinded and taken in an open-mouthed conical flask and then irradiated at 400 W for 27 min (Scheme 1, Table 1) in a domestic microwave oven (SANYO 800G). The residue was dissolved in water and then extracted with $CHCl_3$. The organic layer was evaporated under reduced pressure. The crude product was then purified by column chromatography (silica gel, 100–200 mesh) to afford a pure yellow colored 2,4-diphenyl-4,5-dihydro-3*H*-pyrido[2,3-b][1,4]diazepine (3a) in 80% yield using 5% ethyl acetate-petroleum ether as eluent.

Representative spectral data of all compounds.

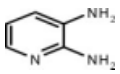
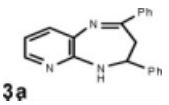
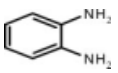
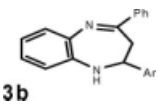
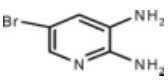
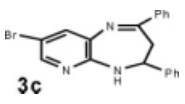
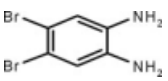
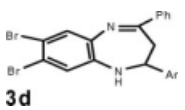
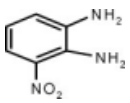
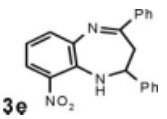
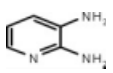
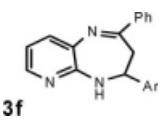
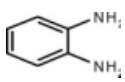
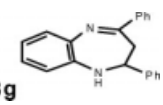
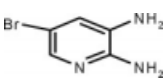
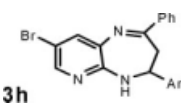
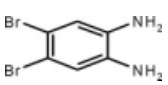
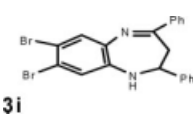
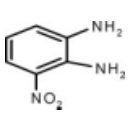
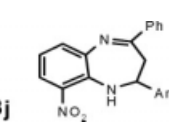
2,4-Diphenyl-4,5-dihydro-3H-pyrido[2,3-b][1,4]diazepine (3a). Yield: 80%; mp 126–128°C; IR: 3230, 3058, 2917, 2849, 1610, 1579, 1445, 1363, 1234, 1101, 915 cm^{-1}; ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm): 8.03 (d, 1H, J = 4.7 Hz), 7.78 (d, 2H, J = 7.2 Hz), 7.66 (d, 1H, J = 7.7 Hz), 7.39 (m, 7H), 7.29 (m, 1H), 6.89 (q, 1H, J = 7.7 Hz), 5.16 (m, 1H), 5.14 (s, 1H), 3.37 (d, 1H, J = 13.9 Hz), 3.09 (q, 1H, J = 13.9 Hz); ms (HRMS-ESI): m/z (%): Calculated for $C_{20}H_{18}N_3$ is 300.1484, found 300.1477 [$(M+H)^+$, 100], 301.1532 [$(M+2H)^+$, 23]. *Anal*. Calcd. for $C_{20}H_{17}N_3$: C, 80.24; H, 5.72; N, 14.04. Found: C, 80.29; H, 5.77; N, 13.98.

2-(3-Bromo-phenyl)-4-phenyl-2,3-dihydro-1H-benzo[b]-[1,4]diazepine (3b). Yield: 82%; mp 90–92°C; IR: 3359, 3057, 2916, 1679, 1608, 1569, 1473, 1428, 1296, 1070, 996 cm^{-1}; ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm): 7.81 (d, 2H, J = 8.1 Hz), 7.56 (s, 1H), 7.39 (m, 6H), 7.17 (t, 1H, J = 7.8 Hz), 7.05 (m, 2H), 6.82 (d, 1H, J = 7.5 Hz), 5.17 (q, 1H, J = 8.4 Hz), 3.73 (bs, 1H), 3.21 (dd, 1H, J = 4.0, 13.5 Hz), 3.02 (dd, 1H, J = 8.5, 13.5 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz): δc (ppm): 167.4, 147.4, 139.8, 139.4, 138.3, 131.5, 130.8, 130.7, 129.6, 129.3, 129, 128.8, 128.5, 127.4, 126.9, 125, 123.2, 122.1, 121.1, 70.6, 37.7; ms (HRMS-ESI): m/z (%): Calculated for $C_{21}H_{17}BrN_2$ is 377.0625, found 377.0617 [M^+, 100], 379.0558 [$(M+2H)^+$, 80]. *Anal*. Calcd. for $C_{21}H_{17}BrN_2$: C, 66.85; H, 4.54; N, 7.42. Found: C, 66.79; H, 4.69; N, 7.47.

8-Bromo-2,4-diphenyl-4,5-dihydro-3H-pyrido[2,3-b][1,4]-diazepine (3c). Yield: 72%; mp 152–153°C; IR: 3235, 3062, 2928, 2844, 1606, 1578, 1432, 1224, 912 cm^{-1}; ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm): 8.00 (s, 1H), 7.78 (d, 3H, J = 6.8 Hz), 7.42 (t, 1H, J = 7.2 Hz), 7.38 (d, 2H, J = 7.2 Hz),

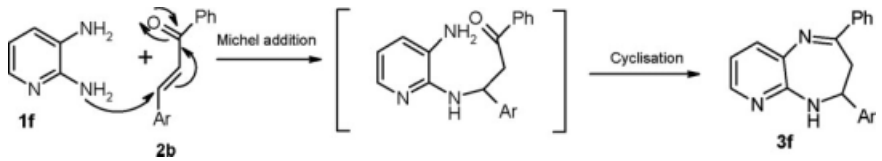
Table 1

Pyridodiazepines and benzodiazepines synthesis under microwave irradiation.

Entry	Substrate (**1**)	β-aryl vinyl ketones (**2**)	MW condition (Watt. min)	Products [24] (**3**)	Yield (%)[a]
a		**2a**	400, 35	 **3a**	80
b		**2b**	400, 27	 **3b**	82
c		**2a**	400, 37	 **3c**	72
d		**2b**	400, 35	 **3d**	65
e		**2a**	400, 47	 **3e**	35
f		**2b**	400, 40	 **3f**	70
g		**2a**	400, 25	 **3g**	85
h		**2b**	400, 38	 **3h**	65
i		**2a**	400, 32	 **3i**	68
j		**2b**	400, 45	 **3j**	32

[a] Chromatographically isolated pure materials.

Scheme 2. Probable mechanism for the synthesis of pyridodiazepines and benzodiazepines.



7.34 (t, 4H, J = 7.1 Hz), 7.29 (t, 1H, J = 7.1 Hz), 5.22 (bs, 1H), 5.11 (m, 1H), 3.38 (d, 1H, J = 14.0 Hz), 3.07 (dd, 1H, J = 8.7, 13.9 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz): δc (ppm): 168.2, 150.0, 146.6, 144.0, 140.3, 139.2, 132.2, 131.0, 129.4, 128.9, 128.7, 127.5, 126.3, 109.8, 66.1, 39.8; ms (HRMS-ESI): m/z (%): Calculated for $C_{20}H_{16}BrN_3$ is 378.0625, found 378.0921 [M^+, 100], 380.0891 [$(M+2H)^+$, 93]. *Anal*. Calcd. for $C_{20}H_{16}BrN_3$: C, 63.50; H, 4.26; N, 11.11. Found: C, 63.41; H, 4.35; N, 11.21.

7,8-Dibromo-2-(3-bromo-phenyl)-4-phenyl-2,3-dihydro-1H-benzo[b][1,4]diazepine (3d). Yield: 65%; mp 88–89°C; IR: 3356, 2922, 2863, 1658, 1568, 1248, 1021, 910 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz): δ (ppm): 7.77 (d, 2H, J = 7.1 Hz), 7.52 (s, 1H), 7.37 (m, 6H), 7.19 (m, 2H), 5.13 (m, 1H), 3.80 (bs, 1H), 3.21 (dd, 1H, J = 3.7, 16.6 Hz), 3.03 (q, 1H, J = 13.3 Hz); ms (HRMS-ESI): m/z (%): Calculated for $C_{21}H_{15}Br_3N_2Na$ is 558.8683, found 558.8696 [$(M+Na)^+$, 50], 560.8768 [$(M+2H+Na)^+$, 10], 556.8659 [$(M-2H+Na)^+$, 100], 554.8785 [$(M-4H+Na)^+$, 50]. *Anal*. Calcd. for $C_{21}H_{15}Br_3N_2$: C, 47.14; H, 2.83; N, 5.24. Found: C, 47.22; H, 2.69; N, 5.32.

9-Nitro-2,4-diphenyl-2,3-dihydro-1H-benzo[b][1,4]-diazepine (3e). Yield: 35%; mp 86–88°C; IR: 3062, 2934, 2846, 1680, 1586, 1445, 1218, 1056, 982 cm^{-1}; ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm): 7.98 (m, 4H), 7.59 (m, 1H), 7.52 (d, 1H, J = 8.4 Hz), 7.44 (m, 2H), 7.42 (m, 1H), 7.40 (m, 2H), 7.32 (t, 2H, J = 7.9 Hz), 6.57 (q, 1H, J = 8.9 Hz), 4.87 (dd, 1H, J = 8.7, 18.9 Hz), 3.87 (dd, 1H, J = 4.9, 17.9 Hz); ms (FIA-MS): m/z (%): 347 $[(M+4H)^+$, 10], 328.2 $[(M+3H-H_2O)^+$, 100], 209.2 (70); *Anal*. Calcd. For $C_{21}H_{17}N_3O_2$: C, 73.45; H, 4.99; N, 12.24. Found: C, 72.95; H, 5.65; N, 12.23.

4-(3-Bromo-phenyl)-2-phenyl-4,5-dihydro-3H-pyrido[2,3-b][1,4] diazepine (3f). Yield: 70%; mp 138–140°C; IR: 3242, 3068, 2910, 1665, 1575, 1448, 1226, 1120, 924 cm^{-1}; ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm): 8.03 (s, 1H), 7.77 (d, 2H, J = 6.8 Hz), 7.66 (d, 1H, J = 7.7 Hz), 7.54 (s, 1H), 7.39 (m, 5H), 7.18 (t, 1H, J = 7.8 Hz), 6.92 (dd, 1H, J = 7.7 Hz), 5.17 (m, 1H), 5.12 (bs, 1H), 3.33 (dd, 1H, J = 2.8, 13.8 Hz), 3.10 (dd, 1H, J = 8.2, 13.9 Hz); ms (FIA-MS): m/z (%): 378.1 [M^+, 100], 380.1 $[(M+2H)^+$, 80], 196.2. *Anal*. Calcd. for $C_{20}H_{16}BrN_3$: C, 63.50; H, 4.26; N, 11.11. Found: C, 63.57; H, 4.30; N, 11.02.

2,4-Diphenyl-2,3-dihydro-1H-benzo[b][1,4]diazepine (3g). Yield: 85%; mp 112–114°C; IR: 3336, 3020, 2925, 1610, 1580, 1470, 1238, 1125, 940 cm^{-1}; ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm): 7.84 (d, 2H, J = 7.9 Hz), 7.35 (m, 9H), 7.04 (m, 2H), 6.82 (d, 1H, J = 7.6 Hz), 5.18 (q, 1H, J = 9.1 Hz), 3.77 (bs, 1H), 3.24 (dd, 1H, J = 3.8, 13.5 Hz), 3.05 (q, 1H, J = 13.5 Hz); ms (FIA-MS): m/z (%): 299.3 $[(M+H)^+$, 100], 284.2, 195.2. *Anal*. Calcd. for $C_{21}H_{18}N_2$: C, 84.53; H, 6.08; N, 9.39. Found: C, 84.58, H, 6.17; N, 9.33.

8-Bromo-4-(3-bromo-phenyl)-2-phenyl-4,5-dihydro-3H-pyrido[2,3-b][1,4]diazepine (3h). Yield: 65%; mp 130–132°C; IR: 3239, 3056, 2922, 2852, 1669, 1573, 1472, 1214, 1071, 900 cm^{-1}; ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm): 8.04 (s, 1H), 7.79 (s, 1H), 7.76 (d, 2H, J = 7.1 Hz), 7.52 (s, 1H), 7.39 (m, 4H), 7.32 (d, 1H, J = 7.8 Hz), 7.19 (t, 1H, J = 7.8 Hz), 5.13 (m, 1H), 5.12 (bs, 1H), 3.34 (d, 1H, J = 13 Hz), 3.09 (dd, 1H, J = 8.9, 13.9 Hz); ms (FIA-MS): m/z (%): 458.1 $[(M+H)^+$, 97], 458.9 $[(M+2H)^+$, 25], 459.8 $[(M+3H)^+$, 35], 456.0 $[(M-H)^+$, 100], 454.1 $[(M-3H)^+$, 50], 356.1 (45), 276.1 (15), 188.1 (40); *Anal*. Calcd. for $C_{20}H_{15}Br_2N_3$: C, 52.55; H, 3.31; N, 9.19. Found: C, 52.49; H, 3.36; N, 9.23.

7,8-Dibromo-2,4-diphenyl-2,3-dihydro-1H-benzo[b][1,4]-diazepine (3i). Yield: 68%; mp 71–72°C; IR: 3360, 2918, 2850, 1608, 1574, 1451, 1272, 1112, 1025, 885 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz): δ (ppm): 7.80 (d, 2H, J = 6.9 Hz), 7.58 (s, 1H), 7.36 (m, 8H), 7.06 (s, 1H), 5.12 (m, 1H), 3.83 (bs, 1H), 3.26 (dd, 1H, J = 3.6, 13.6 Hz), 3.03 (dd, 1H, J = 8.9, 13.66 Hz); ms (HRMS): m/z (%): Calcd for $C_{21}H_{16}Br_2N_2$ is 456.9766, found 456.9735 [M^+, 100], 458.9732 $[(M+2H)^+$, 50], 454.9669 $[(M-2H)^+$, 50]. *Anal*. Calcd. for $C_{21}H_{16}Br_2N_2$: C, 55.29; H, 3.54; N, 6.14. Found: C, 55.23; H, 3.62, N, 6.21.

2-(3-Bromophenyl)-9-nitro-4-phenyl-2,3-dihydro-1H-benzo[b][1,4]diazepine (3j). Yield: 32%; mp 94–96°C; IR: 3068, 2922, 2851, 1685, 1595, 1529, 1473, 1207, 1073 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz): δ (ppm): 8.03 (d, 2H, J = 8.3 Hz), 7.97 (t, 1H, J = 8.4 Hz), 7.59 (d, 1H, J = 9.2 Hz), 7.56 (m, 1H), 7.49 (m, 3H), 7.42 (d, 2H, J = 7.0 Hz), 7.34 (m, 1H), 7.18 (t, 1H, J = 7.8 Hz), 6.52 (q, 1H, J = 8.5 Hz), 4.83 (dd, 1H, J = 9.1, 16.9 Hz), 3.87 (dd, 1H, J = 5.1, 17.9 Hz); ms (FIA-MS): m/z (%): 421.2 $[(M-H)^+$, 20], 408.3 (30), 406.1 (20), 279.2 (100), 262.3 (80). *Anal*. Calcd. for $C_{21}H_{16}BrN_3O_2$: C, 59.73; H, 3.82; N, 9.95. Found: C, 59.37; H, 4.37; N, 9.81.

2-(3-Bromo-phenyl)-1H-benzoimidazole (4). Yield: 5%; mp 245–247°C; IR: 3342, 3121, 1621, 1534, 1012 cm^{-1}; ^{1}H NMR ($CDCl_3$ + d_6-DMSO, 400 MHz): δ (ppm): 8.41 (s, 1H), 8.17 (d, 1H, J = 7.8 Hz), 7.76 (d, 1H, J = 8.1 Hz), 7.56 (d, 1H, J = 7.9 Hz), 7.50 (d, 1H, J = 7.45 Hz), 7.38 (t, 1H, J = 7.9 Hz), 7.23 (m, 2H); ms (FIA): m/z (%): 273.0 (M^+, 98), 275.0 $[(M+2)^+$, 100].

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