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RESEARCH ARTICLE

Synthesis of novel 1,5-benzothiazepine[7,6-*b*]-1, 8-naphthyridines under microwave irradiation *via* Mannich condensation

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A series of novel 1,5-benzothiazepine[7,6-*b*]-1,8-naphthyridines **3a–j** were synthesized by reaction of substituted 2-amino-thiophenol with different substituted 2-chloro-3-formyl-1,8-naphthyridines **1a–d** under microwave irradiation and solvent-free condition is described. A judicious choice of the reaction conditions under microwave allowed the final products **3a–j** in excellent one-step procedure, whereas experiments under thermal conditions led to **3a–j** in lower yields with tedious work-up. The new compounds have been characterized with the assistance of elemental analyses, IR, ^{1}H NMR and mass spectral studies.

Keywords: Microwave irradiation; 1,5-Benzothiazepine[7,6-*b*]-1,8-naphthyridine; 2-Amino-thiophenol

1. Introduction

The immense chemotherapeutic applications of 1,5-benzothiazepines especially that of *diltiazem* in the treatment of ailments of the cardiovascular system and a number of biological activities have been associated with it, such as inhibitors of antihypertensive [1–3], antiasthemic [4], analgesic [5], coronary vasodilation [6], cardiovascular [7] and platelate aggregation inhibitor [8]. Recently, Ahmed [9] patented 1,5-benzothiazepine derivatives as potential anticancer drugs.

In addition, 1,5-benzothiazepines are used as starting materials for the preparation of fused heterocyclic rings[10]. Despite their importance from a pharmacological and synthetic point of view, few methods have been reported for the preparation of 1,5-benzothiazepines by the reaction of 2-aminothiophenol with α,β-unsaturated ketones [11],

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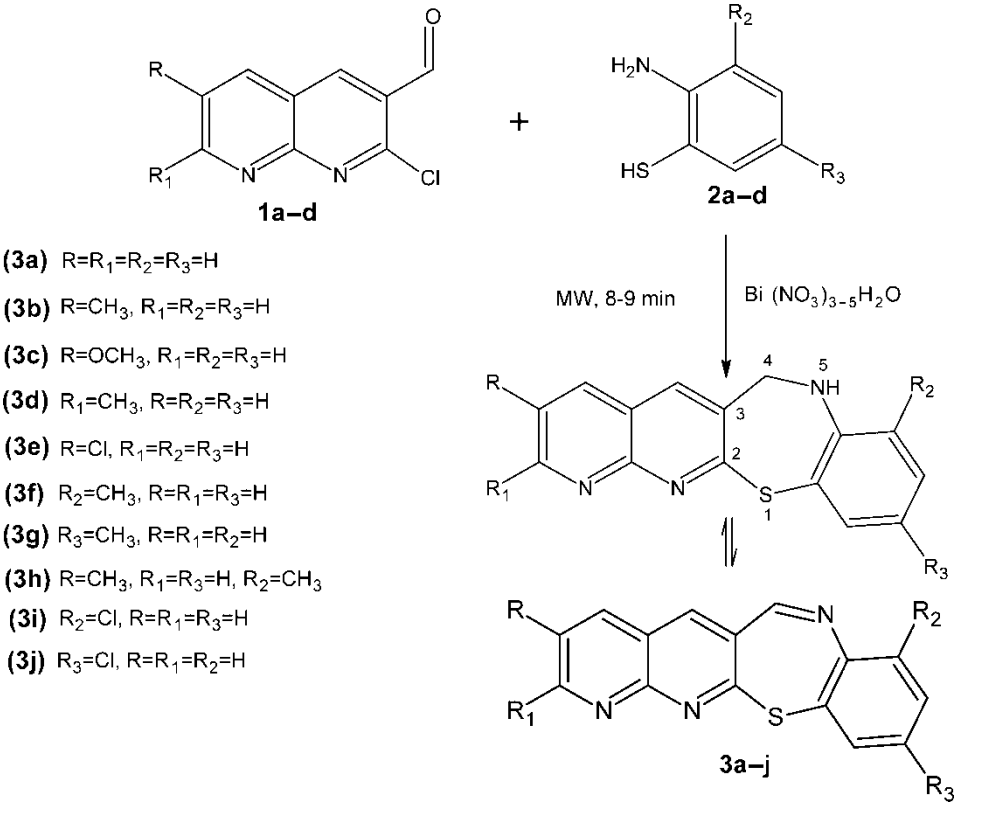
ω-bromoacetophenones, aromatic aldehydes [12] and by the reaction of α,β-unsaturated ketones with bis(2-nitrophenyl)disulfide use of $TiCl_4$/Sm catalyst [13].

There are few reports on the use of inorganic solid supports such as alumina, silica gel and clay as catalysts for the synthesis of 1,5-benzothiazepines under solvent-free condition. Recently, Danidia [14] reported a solvent-free synthesis of 1,5-benzothiazepines in the presence of a solid support under microwave irradiation [15–20].

Further, naphthyridines continue to be of great interest due to a wide range of biological activities, being widely used for the diagnosis and chemotherapy of infectious diseases including AIDS. Some of new 1,8-naphthyridines are recently been patented as growth regulators, fungicides, bactericides, herbicides, insecticides and nemathocides [21–24].

Microwave-assisted reactions are becoming popular for organic chemists [25] and have recently been reviewed [26]. More interest has been focused on dry media synthesis under microwave (MW) irradiation and especially by carrying out the experiments with supported reagents on mineral oxides [27]. This technology provides a promising alternative to environmentally unacceptable thermal procedures, which are usually time consuming, unsafe and cause solvent emission leading to pollution and waste disposal problems. In many cases, the use of solvent-free methodology or supported reagents under microwaves allows the preparation of products not accessible by the classical heating method.

Hence, an attempt towards a non-traditional approach to the experimental set up of organic reactions, and keeping in mind the biological importance of 1,5-benzothiazepines, our interest in devising solvent-free procedure in the synthesis of sulfur containing novel heterocycles



SCHEME 1

and their association with microwave activation [28–31], we describe herein, simple, enviro-friendly method for the synthesis of 1,5-benzothiazepine[7,6-*b*]-1,8-naphthyridine; 2-amino-thiophenol **(3a–j)** compounds using crystalline $Bi(NO_3)_3.5H_2O$ catalyst (scheme 1).

2. Results and discussion

The preparation of compounds **3a–j** were accomplished by the reaction sequence shown in scheme 1.

The Mannich condensation of substituted 1,8-naphthyridines [32] and substituted 2-aminothiophenol in presence of using crystalline $Bi(NO_3)_3.\ 5H_2O$ catalyst offered 1,5-benzothiazepine[7,6-*b*]-1,8-naphthyridines in good to excellent yields under MW irradiation. Molecules containing sulfur are known to exhibit various pharmacological activities. We tried to introduce sulfur atom by chlorine from C_2 of 1,8-naphthyridines with a SH group. The reaction proceeds through the condensation of amino group with aldehydic group of 1,8-naphthyridines, it is the sulfur atom that reacts with the carbon next to the chlorine at C_2 of 1,8-naphthyridines, which resulted in the title compounds **3a–j**.

For comparison, the reaction was also carried out in presence of different solid support including silica gel, acidic alumina, basic alumina, neutral alumina and molecular sieves (5A°) (table 1).

The reaction has also been carried out without adding any support under neat condition, which could be expected to be the most economical method. But unfortunately lower yields were obtained. From the results, it is obvious that $Bi(NO_3)_3.\ 5H_2O$ is the most effective solid for the synthesis of 1,5-benzothiazepine[7,6-*b*]-1,8-naphthyridines **(3a–j)**, where as acidic alumina, basic alumina, neutral alumina, molecular sieves (5A°) were ineffective in giving products in good yields.

Finally, in order to check the possible intervention of specific (non-thermal) MW effects, the reaction was performed also under thermal conditions in the presence of a solvent. It was observed that, the yields were significantly lower than those obtained using the MW method (experimental section). The synthesized compounds were characterized by elemental analysis, IR and ^{1}H NMR spectroscopy and mass spectrometry.

The IR spectra of the products **3a–j** did not reveal the presence of primary amino group in the region 3218 cm^{-1}. On the other hand, the absorption bands were observed in the region 1596–1663 cm^{-1} for (C=N). The N-H bending vibrations were observed as a sharp medium to strong band at 1500–1558 cm^{-1} in compounds **3a–j**. The C-S-C linkage of the seven membered ring caused a weak and sharp absorption band at 685–748 cm^{-1} in all the compounds confirming the formation of **3a–j**.

Table 1. Yield of 1,5-benzothiazepine[7,6-*b*]-1,8-naphthyridines in the presence of various supports.

Catalyst in mol (%)	Yield (%)
$Bi(NO_3)_3.\ 5H_2O$ (20)	78
Silica gel (30)	56
Acidic alumina (30)	25
Basic alumina (35)	5
Neutral alumina (40)	18
Molecular sieves (5 A°) (45)	27
None	20

The ^{1}H NMR spectra showed a singlet at δ 8.53 ppm due to NH of benzothiazepine. A multiplet due to aromatic protons appeared at δ 7.30–8.05 ppm. Absence of NH_2 protons also confirmed the formation of **3a–j**. A proton of the methoxy group was observed at δ 4.02 ppm. The methyl protons, whenever present, were observed at their usual position (δ 2.26–2.33 ppm) as a singlet. The structures of compounds **3a–j** were further confirmed by their mass spectrum, as an example compound **3a** exhibit the molecular ion peak [M^+] at m/z 265.

3. Experimental section

Melting points were determined in an open capillary tube and uncorrected. Elemental analyses were carried out using Perkin-Elmer 240C CHN-analyser. IR spectra were recorded on FT-IR spectrophotometer. ^{1}H-NMR spectra were run on a NMR spectrometer (chemical shifts in δ ppm). Mass spectra were recorded on a LC MS mass spectrometer. The purity of the compounds was checked by thin layer chromatography (TLC).

3.1 *General MW procedure for the synthesis of 1,5-benzothiazepine [7,6-b]-1,8-naphthyridines (3a–f)*

The substituted 1,8-naphthyridine **2a** (1.91 g, 1 mmol), 2-aminothiophenol (1.25 g, 1 mmol) were transformed into a 100 mL beaker, and 20 mol% $Bi(NO_3)_3$. $5H_2O$ were mixed thoroughly with a glass rod; the color changes to yellow. Then the contents were irradiated in a MW oven for about 8–9 min at an interval of 1 min at 160 W. The completion of reaction was monitored by TLC. Once complete, the mixtures were cooled, treated with (50 mL) water and ethyl acetate (25 mL). The organic layer was separated from the aqueous one and organic phase was washed with saturated solution of sodium bicarbonate (2 × 20 mL) and water, dried over anhydrous Na_2SO_4,filtered. The filtrates were concentrated to yield a yellow colour product. It was recrystalised from MeOH to give pure **3** as a yellow solid. The same procedure was used for the synthesis of **3b–j**. The physico-chemical data for the synthesized compounds are as shown below.

3.2 *Conventional method*

To a mixture of substituted, 18-naphthyridine **2a** (1.91 g, 1 mmol), 2-aminothiophenol (1.25 g, 1 mmol) and 20 mol% $Bi(NO_3)_3$. $5H_2O$ and 30 mL methanol were taken in 100 mL round bottom flask, kept reflux for about 10–12 h after the completion of the reaction confirmed by TLC, reaction mixture was concentrated then poured into ice cold water (50 mL) and ethyl acetate (25 mL). The organic layer was separated from the aqueous one and organic phase was washed with saturated solution of sodium bicarbonate (2 × 20 mL) and water, dried over anhydrous Na_2SO_4 and filtered. The filtrates were concentrated to yield a yellow color product. It was recrystalized from MeOH to give pure **3** as an yellow solid. The same procedure was used for the synthesis of **3b–j**.

3.3 *Physical and spectral data of the products*

3.3.1 1,5-benzothiazepine [7,6-b]-1,8-naphthyridines (3a). Solid. Yellow 78%. Time; 8 min (MW), 10 h (CM), mp: 240–242 °C. FT-IR (KBr): 1608 (C=N) cm^{-1}. ^{1}H NMR (DMSO-d_6, 300 MHz): δ (ppm) = 7.32 (d, 1H, J = 8.0), 7.35 (d, 1H, J = 8.0), 7.37 (d, 1H, J = 8.0), 7.40 (d, 1H, J = 8.0), 7.48 (d, 1H, J = 8.0), 7.55 (d, 1H, J = 8.5), 7.58 (d, 1H, J = 8.5),

7.64 (d, 1H, $J = 8.5$), 7.78 (d, 1H, $J = 8.6$). Mass, m/z (relative intensity): 263 (M^+). Calcd. (%) for $C_{15}H_9N_3S$: C; 68.42, H; 3.45, N; 15.96, S; 12.18. Found: C; 68.41, H; 3.43, N; 15.94, S; 12.15.

3.3.2 12-Methyl-1,5-benzothiazepine [7,6-b]-1,8-naphthyridines (3b). Solid. Yellow 78%. Time; 8 min (MW), 10 h (CM), mp: 228–231 °C. FT-IR (KBr): 1615 (C=N) cm^{-1}. ^1H NMR (DMSO-d_6, 300 MHz): δ (ppm) = 2.36 (s, 3H, CH_3), 7.23 (d, 1H, $J = 8.0$), 7.27 (d, 1H, $J = 8.0$), 7.35 (d, 1H, $J = 8.0$), 7.45 (d, 1H, $J = 8.0$), 7.61 (d, 1H, $J = 8.5$), 7.67 (d, 1H, $J = 8.5$), 7.72 (d, 1H, $J = 8.6$), 7.89 (d, 1H, $J = 8.6$). Mass, m/z (relative intensity): 277 (M^+). Calcd. (%) for $C_{16}H_{11}N_3S$: C; 69.29, H; 4.00, N; 15.15, S; 11.56. Found: C; 69.26, H; 3.98, N; 15.14, S; 11.52.

3.3.3 12-Methoxy-1,5-benzothiazepine [7,6-b]-1,8-naphthyridines (3c). Solid. Yellow 76%. Time; 8 min (MW), 11 h (CM), mp: 254–256 °C. FT-IR (KBr): 1605 (C=N) cm^{-1}. ^1H NMR (DMSO-d_6, 300 MHz): δ (ppm) = 4.02 (s, 3H, OCH_3), 7.33 (d, 1H, $J = 8.0$), 7.36 (d, 1H, $J = 8.0$), 7.58 (d, 1H, $J = 8.5$), 7.61 (d, 1H, $J = 8.5$), 7.65 (d, 1H, $J = 8.5$), 7.68 (d, 1H, $J = 8.5$), 7.73 (d, 1H, $J = 8.6$), 7.91 (d, 1H, $J = 8.6$). Mass, m/z (relative intensity): 291 (M^+). Calcd. (%) for $C_{16}H_{11}N_3OS$: C; 65.51, H; 3.78, N; 14.32, S; 10.93. Found: C; 65.52, H; 3.75, N; 14.30, S; 10.90.

3.3.4 13-Methyl-1,5-benzothiazepine [7,6-b]-1,8-naphthyridines (3d). Solid. Yellow 75%. Time; 9 min (MW), 12 h (CM), mp: 226–227 °C. FT-IR (KBr): 1630 (C=N) cm^{-1}. ^1H NMR (DMSO-d_6, 300 MHz): δ (ppm) = 2.30 (s, 3H, CH_3), 7.19 (d, 1H, $J = 8.0$), 7.25 (d, 1H, $J = 8.0$), 7.38 (d, 1H, $J = 8.5$), 7.46 (d, 1H, $J = 8.5$), 7.55 (d, 1H, $J = 8.5$), 7.61 (d, 1H, $J = 8.5$), 7.68 (d, 1H, $J = 8.6$), 7.75 (d, 1H, $J = 8.6$). Mass, m/z (relative intensity): 277 (M^+). Calcd. (%) for $C_{16}H_{11}N_3S$: C; 69.29, H; 4.00, N; 15.15, S; 11.56. Found: C; 69.28, H; 4.01, N; 15.12, S; 11.52.

3.3.5 12-Chloro-1,5-benzothiazepine [7,6-b]-1,8-naphthyridines (3e). Solid. Yellow 76%. Time; 8 min (MW), 11 h (CM), mp: 253–256 °C. FT-IR (KBr): 1645 (C=N) cm^{-1}. ^1H NMR (DMSO-d_6, 300 MHz): δ (ppm) = 7.24 (d, 1H, $J = 8.0$), 7.29 (d, 1H, $J = 8.0$), 7.47 (d, 1H, $J = 8.5$), 7.56 (d, 1H, $J = 8.5$), 7.59 (d, 1H, $J = 8.5$), 7.65 (d, 1H, $J = 8.5$), 7.70 (d, 1H, $J = 8.6$), 7.78 (d, 1H, $J = 8.6$). Mass, m/z (relative intensity): 297 (M^+). Calcd. (%) for $C_{15}H_8ClN_3S$: C; 60.50, H; 2.71, N; 14.11, S; 10.77. Found: C; 60.48, H; 2.73, N; 14.08, S; 10.75.

3.3.6 6-Methyl-1,5-benzothiazepine [7,6-b]-1,8-naphthyridines (3f). Solid. Yellow 75%. Time; 8 min (MW), 10 h (CM), mp: 226–229 °C. FT-IR (KBr): 1659 (C=N) cm^{-1}. ^1H NMR (DMSO-d_6, 300 MHz): δ (ppm) = 2.28 (s, 3H, CH_3), 7.30 (d, 1H, $J = 8.0$), 7.37 (d, 1H, $J = 8.0$), 7.49 (d, 1H, $J = 8.5$), 7.56 (d, 1H, $J = 8.5$), 7.63 (d, 1H, $J = 8.5$), 7.72 (d, 1H, $J = 8.5$), 7.79 (d, 1H, $J = 8.6$), 7.94 (d, 1H, $J = 8.6$). Mass, m/z (relative intensity): 277 (M^+). Calcd. (%) for $C_{16}H_{11}N_3S$: C; 69.29, H; 4.00, N; 15.15, S; 11.56. Found: C; 69.27, H; 3.97, N; 15.12, S; 11.52.

3.3.7 8-Methyl-1,5-benzothiazepine [7,6-b]-1,8-naphthyridines (3g). Solid. Yellow 77%. Time; 9 min (MW), 11 h (CM), mp: 228–230 °C. FT-IR (KBr): 1647 (C=N) cm^{-1}.

^{1}H NMR (DMSO-d_6, 300 MHz): δ (ppm) = 2.33 (s, 3H, CH_3), 7.26 (d, 1H, $J = 8.0$), 7.35 (d, 1H, $J = 8.0$), 7.54 (d, 1H, $J = 8.5$), 7.60 (d, 1H, $J = 8.5$), 7.68 (d, 1H, $J = 8.5$), 7.72 (d, 1H, $J = 8.5$), 7.76 (d, 1H, $J = 8.6$), 7.88 (d, 1H, $J = 8.6$). Mass, m/z (relative intensity): 277 (M^+). Calcd. (%) for $C_{16}H_{11}N_3S$: C; 69.29, H; 4.00, N; 15.15, S; 11.56. Found: C; 69.26, H; 4.02, N; 15.13, S; 11.54.

3.3.8 6,12-Dimethyl-1,5-benzothiazepine [7,6-b]-1,8-naphthyridines (3h). Solid. Yellow 74%. Time; 9 min (MW), 12 h (CM), mp: 231–234 °C. FT-IR (KBr): 1663 (C=N) cm^{-1}. ^{1}H NMR (DMSO-d_6, 300 MHz): δ (ppm) = 2.26 (s, 3H, CH_3), 2.43 (s, 3H, CH_3), 7.34 (d, 1H, $J = 8.0$), 7.47 (d, 1H, $J = 8.5$), 7.55 (d, 1H, $J = 8.5$), 7.67 (d, 1H, $J = 8.5$), 7.70 (d, 1H, $J = 8.5$), 7.74 (d, 1H, $J = 8.6$), 7.82 (d, 1H, $J = 8.6$). Mass, m/z (relative intensity): 291 (M^+). Calcd. (%) for $C_{17}H_{13}N_3S$: C; 70.08, H; 4.50, N; 14.42, S; 11.01. Found: C; 70.05, H; 4.52, N; 14.40, S; 10.98.

3.3.9 6-Chloro-1,5-benzothiazepine [7,6-b]-1,8-naphthyridines (3i). Solid. Yellow 70%. Time; 9 min (MW), 12 h (CM), mp: 256–258 °C. FT-IR (KBr): 1596 (C=N) cm^{-1}. ^{1}H NMR (DMSO-d_6, 300 MHz): δ (ppm) = 7.20 (d, 1H, $J = 8.0$), 7.35 (d, 1H, $J = 8.0$), 7.47 (d, 1H, $J = 8.5$), 7.52 (d, 1H, $J = 8.5$), 7.66 (d, 1H, $J = 8.5$), 7.74 (d, 1H, $J = 8.5$), 7.78 (d, 1H, $J = 8.6$), 7.94 (d, 1H, $J = 8.6$). Mass, m/z (relative intensity): 297 (M^+). Calcd. (%) for $C_{15}H_8ClN_3S$: C; 60.50, H; 2.71, N; 14.11, S; 10.77. Found: C; 60.48, H; 2.70, N; 14.13, S; 10.78.

3.3.10 8-Chloro-1,5-benzothiazepine [7,6-b]-1,8-naphthyridines (3j). Solid. Yellow 72%. Time; 8 min (MW), 10 h (CM), mp: 263–265 °C. FT-IR (KBr): 1580 (C=N) cm^{-1}. ^{1}H NMR (DMSO-d_6, 300 MHz): δ (ppm) = 7.27 (d, 1H, $J = 8.0$), 7.32 (d, 1H, $J = 8.0$), 7.54 (d, 1H, $J = 8.5$), 7.67 (d, 1H, $J = 8.5$), 7.71 (d, 1H, $J = 8.5$), 7.78 (d, 1H, $J = 8.5$), 7.82 (d, 1H, $J = 8.6$), 7.96 (d, 1H, $J = 8.6$). Mass, m/z (relative intensity): 297 ($M^{+\cdot}$). Calcd. (%) for $C_{15}H_8ClN_3S$: C; 60.50, H; 2.71, N; 14.11, S; 10.77. Found: C; 60.49, H; 2.67, N; 14.08, S; 10.75.

4. Conclusion

In conclusion, we have developed a simple, practical and very region-selective method for the synthesis of 1,5-benzothiazepine[7,6-*b*]-1,8-naphthyridines derivatives using the inexpensive, oxygen and moisture-tolerant, and easily available $Bi(NO_3)_3.\ 5H_2O$ catalyst. The reaction offers several advantages including high yields of the products and it makes the present method as a useful addition to the present methodologies.

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