Journal of Sulfur Chemistry
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713926081

One pot solvent-free synthesis of 2*H*-pyrano, 2*H*-thiopyrano, 2*H*-selenopyrano[2,3-*b*]-1,8-naphthyridin-2-ones on solid phase catalyst under microwave irradiation

T. R. Ravikumar Naik[a]; H. S. Bhojya Naik[a]; S. R. Gopala Krishna Naik[a]
[a] Department of P.G. Studies and Research in Industrial Chemistry, School of Chemical Sciences, Kuvempu University, Shankaraghatta, India

To cite this Article Naik, T. R. Ravikumar , Naik, H. S. Bhojya and Naik, S. R. Gopala Krishna(2007) 'One pot solvent-free synthesis of 2*H*-pyrano, 2*H*-thiopyrano, 2*H*-selenopyrano[2,3-*b*]-1,8-naphthyridin-2-ones on solid phase catalyst under microwave irradiation', Journal of Sulfur Chemistry, 28: 4, 393 — 400
To link to this Article: DOI: 10.1080/17415990701312279
URL: http://dx.doi.org/10.1080/17415990701312279

Journal of Sulfur Chemistry
Vol. 28, No. 4, August 2007, 393–400

Taylor & Francis
Taylor & Francis Group

RESEARCH ARTICLE

One pot solvent-free synthesis of 2*H*-pyrano, 2*H*-thiopyrano, 2*H*-selenopyrano[2,3-*b*]-1,8-naphthyridin-2-ones on solid phase catalyst under microwave irradiation

T. R. RAVIKUMAR NAIK, H. S. BHOJYA NAIK* and S. R. GOPALA KRISHNA NAIK

Department of P.G. Studies and Research in Industrial Chemistry, School of Chemical Sciences, Kuvempu University, Shankaraghatta-577 451, India

(Received 9 November 2006; in final form 20 February 2007)

A simple and efficient procedure has been developed for the synthesis of the 2*H*-pyrano (**3a**–**c**), 2*H*-thiopyrano (**5a**–**c**), 2*H*-selenopyrano[2,3-*b*]-1,8-naphthyridin-2-ones (**7a**–**c**) by cyclisation of 2-chloro-3-formyl[1,8]naphthyridines (**1a**–**c**) with acetic anhydride under microwave irradiation using solid phase catalyst anhydrous sodium acetate. These new compounds were characterized by elemental analysis, IR, ^{1}H NMR and mass spectral studies.

Keywords: Naphthyridines; 2*H*-Pyrano; 2*H*-Thiopyrano; 2*H*-Selenopyrano; Solid phase catalyst; Microwave irradiation

1. Introduction

1,8-Naphthyridine derivatives are of considerable interest because of their possible biological activities [1–3]. Antibiotics of this group are being widely used for the diagnostics and chemotherapy of infectious diseases of humans including AIDS. Some of the new 1,8-naphthyridines have recently been patented as growth regulators, fungicides, bactericides, herbicides, insecticides, and nemathocides of new generation [4–10]. In recent years, the number of publications devoted to various aspects of naphthyridine chemistry has sharply increased. More publications have appeared during the last two decades, 40% of them being patents.

Further, it is well known that a number of heterocyclic compounds containing N, S, O and Se exhibit a wide variety of biological activities [11–13]. Even though sulphur and selenium are considered to be isosteric as defined by Langmiur [14] and Erlenmeyer [15], the reports about selenium-containing heterocyclics are relatively few [16–18]. However, the medicinal application of isosterism has been reviewed by Klayman and Gunther [19].

*Corresponding author. Email: hsb_naik@rediffmail.com

Journal of Sulfur Chemistry
ISSN 1741-5993 print/ISSN 1741-6000 online © 2007 Taylor & Francis
http://www.tandf.co.uk/journals
DOI: 10.1080/17415990701312279

The antioxidant and anticancer activity of selenium containing compounds have been reported [20–22]. Selenium plays an important role in decreasing oxidative stress in HIV-infected cells and possibly suppressing the rate of HIV replication [23, 24]. Recent research indicates that HIV may be capable of incorporating host selenium into viral selenoproteins that have glutathione-peroxidase activity. Though the significance of these findings requires further clarification, they suggest that both the human immune system and the activity of the virus are affected by selenium nutritional status [25, 26].

In view of the biological importance of nitrogen, oxygen, sulphur and selenium containing condensed heterocycles and in continuation of our work on microwave assisted organic synthesis of condensed heterocycles [27–29], we have developed a simple, eco-friendly solvent free method for the synthesis of 2*H*-pyrano (**3a–c**), 2*H*-thiopyrano (**5a–c**), 2*H*-selenopyrano[2,3-*b*]-1,8-naphthyridin-2-ones (**7a–c**) from cyclisation of 2-chloro-3-formyl[1,8]naphthyrdines in the presence of solid phase catalyst anhydrous sodium acetate under microwave irradiation.

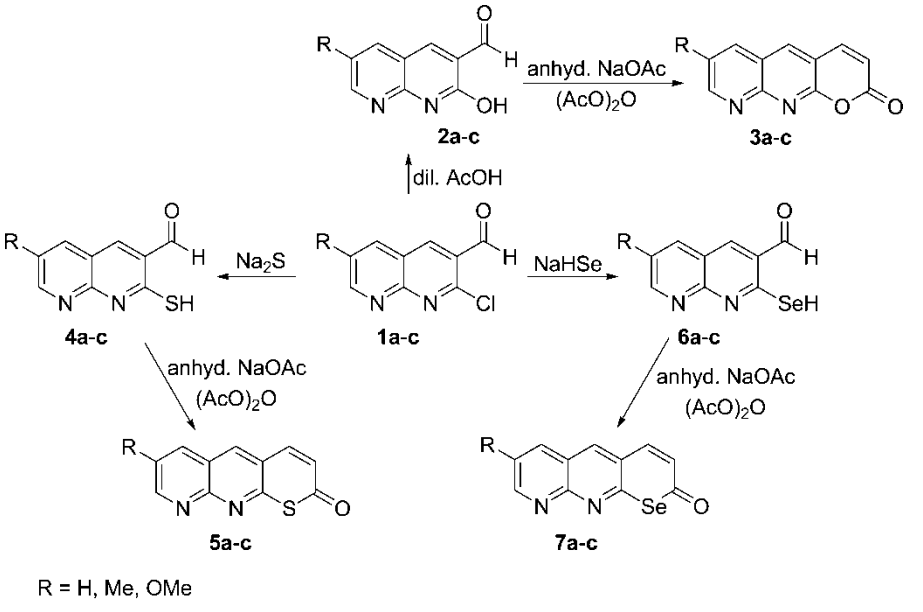
2. Results and discussion

General strategy for the synthesis of 2*H*-pyrano (**3a–c**), 2*H*-thiopyrano (**5a–c**), 2*H*-selenopyrano[2,3-*b*]-1,8-naphthyridin-2-ones (**7a–c**) compounds were summarized in scheme 1. Required starting material 2-chloro-3-formyl[1,8]naphthyridines (**1a–c**) have been prepared in quantitative yield by the cyclisation of N-pyridine-2-ylacetamide with $POCl_3$ to the substrate in DMF at 0–5 °C followed by microwave irradiation at 160 W. Then the microwave irradiation of (**1a–c**) with 70% dilute acetic acid that lead to the formation of substituted 2-hydroxy-1,8-naphthyridine-3-carbaldehydes (**2a–c**). The IR spectrum **2a** showed the peaks at 3440 cm^{-1} and 1655 cm^{-1} due to C-OH and C=N stretching frequency. The ^{1}H NMR spectrum of **2a** exhibited peaks at δ 11.0 for OH ($J = 9.5$ Hz), δ 10.59 ($J = 9$ Hz) for CHO and the observed multiplet in the region δ 7.00–8.02 ($J = 8.5$ Hz) for four aromatic protons.

The compound (**1a–c**) reacts with Na_2S in presence of DMF gave desired products 2-mercapto-1,8-naphthyridine-3-carboldehyde (**4a–c**) under microwave irradiation. In a similar way, 2-chloro-3-formyl[1,8]naphthyridines (**1a–c**) react with sodium hydrogen selenide (which was prepared by selenium powder and sodium borohydride, stirring at freezing temperature for about one hour) in ethanol under microwave irradiation at 160 W gave 2-seleno-1,8-naphthyridine-3-carbaldehydes (**6a–c**). The IR spectrum of (**4a–c**) and (**6a–c**) showed the peaks in the region 1626 cm^{-1}, 1634 cm^{-1} and 1658 cm^{-1} pertaining to C−SH, C−SeH and C=N stretching frequency. The ^{1}H NMR spectrum of **4a** and **6a** shows peaks at δ 11.03 for OH ($J = 9.5$ Hz), δ 10.58 ($J = 9$ Hz) and δ 11.05 for OH ($J = 9.5$ Hz), δ 10.61 ($J = 9$ Hz) for CHO singlet. The multiplet peaks observed in the region δ 7.03–8.10 ($J = 8.5$ Hz) due to the four aromatic protons of these compounds.

Further, the cyclisation of 2-hydroxy-1,8-naphthyridine-3-carbaldehydes (**2a–c**) with acetic anhydride in the presence of anhydrous sodium acetate catalyst under microwave irradiation conditions afforded 2H-pyrano[2,3-*b*]-1,8-naphthyridin-2-ones (**3a–c**) in 88–90% yields (scheme 1).

Under similar conditions, the cyclisation of 2-mercapto-1,8-naphthyridine-3-carbaldehyde (**4a–c**) and 2-seleno-1,8-naphthyridine-3-carbaldehydes (**6a–c**) with acetic anhydride in the presence of anhydrous sodium acetate catalyst under microwave irradiation conditions afforded 88–90% yields of corresponding 2*H*-thiopyrano (**5a–c**) and 2*H*-selenopyrano[2,3-*b*]-1,8-naphthyridin-2-ones (**7a–c**) (scheme 1). All the reactions afforded high yields of target products in a shorter reaction time with high purity. The structure of compounds



SCHEME 1

were confirmed on the basis of elemental analysis and spectral data (Experimental section). As an example the IR (KBr) spectra of 2*H*-pyrano (**3a–c**), 2*H*-thiopyrano (**5a–c**), 2*H*-selenopyrano[2,3-*b*]-1,8-naphthyridin-2-ones (**7a–c**) exhibited an absorption band in the region 1666–1700 cm^{-1}, 1222–1230 cm^{-1} and 1226–1234 cm^{-1} due to C=O, C−S−C and C−Se−C groups, respectively.

When the same reaction is carried out in classical heating it took 10–12 hrs for completion. And also, it has been found that the reaction yield 55–60% with incomplete consumption of the starting material even took a longer reaction time. From these results we found that the microwave-assisted method is more convenient for the synthesis of title compounds.

3. Conclusions

In conclusion, we have developed a versatile and useful new access to different classes of biological importance 2*H*-pyrano (**3a–c**), 2*H*-thiopyrano (**5a–c**), 2*H*-selenopyrano[2,3-*b*]-1,8-naphthyridin-2-ones (**7a–c**) using an efficient and simple methodology based on microwave irradiation. The most important result of this approach, is the optimization of yields and reaction times. The efficiency of the employed methodology can be explained by the fact that microwave energy is probably much higher than the activation energy necessary for each reaction so that reaction speeds are increased and yields are higher.

4. Experimental section

Melting points were determined in an open capillary tube and are uncorrected. Elemental analyses were carried out using Perkin-Elmer 240C CHNS-analyzer and the selenium content was analysed by reference method [30]. IR spectra were recorded on a FT−IR spectrophotometer. ^{1}H NMR spectra was run in (DMSO−d_6) solvent at 300 MHz 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).

4.1 *General procedure for the synthesis of 2-chloro-3-formyl-1,8-naphthyridines (1a–c)*

A mixture of N-pyridine-2-ylacetamide (5 mmol), dry DMF (13 mmol) and $POCl_3$ (45 mmol) was transferred into a beaker and irradiated in a microwave oven at 160 W for 5–6 min. On completion of the reaction as indicated in the TLC, the reaction mixture was treated with ice-cold water. The precipitated solid **1a** was filtered, washed with water and recrystallized from methanol or acetonitrile solvent. The same procedure was used for the synthesis of (**1b**,**c**) compounds. The physicochemical data for the synthesized compounds are as shown below.

4.1.1 2-Chloro-3-formyl-1,8-naphthyridines (1a). Solid. Yellow 90%. mp: 146 °C. FT−IR (KBr): 1585 (C=N), 1685 (C=O) cm^{-1}. ^1H NMR (DMSO-d_6, 300 MHz): δ (ppm) = 10.59 (CHO, s, 1H), 8.00 (d, 1H, $J = 8.5$ Hz), 7.86 (d, 1H, $J = 8.4$ Hz), 7.56 (d, 1H, $J = 8.4$ Hz), 6.90 (d, 1H, $J = 6.8$ Hz. Mass, m/z (relative intensity): 194 ($M^{+.2}$). Calcd. (%) for $C_9H_5ClN_2O$: C; 56.12, H; 2.62, N; 14.54. Found: C; 56.10, H; 2.61, N; 14.53.

4.1.2 6-Methyl-2-chloro-3-formyl-1,8-naphthyridines (1b). Solid. Yellow 93%. mp: 139 °C. FT−IR (KBr): 1580 (C=N), 1700 (C=O) cm^{-1}. ^1H NMR (DMSO−d_6, 300 MHz): δ (ppm) = 10.60 (CHO, s, 1H), 8.02 (d, 1H, $J = 8.4$ Hz), 7.58 (d, 1H, $J = 8.4$ Hz), 6.92 (d, 1H, $J = 6.8$ Hz), 2.66 (3H, s, $-CH_3$). Mass, m/z (relative intensity): 208 ($M^{+.2}$). Calcd. (%) for $C_{10}H_7ClN_2O$: C; 58.13, H; 3.41, N; 13.56. Found: C; 58.11, H; 3.40, N; 13.54.

4.1.3 6-Methoxy-2-chloro-3-formyl-1,8-naphthyridines (1c). Solid. Yellow 95%. mp: 141 °C. FT−IR (KBr): 1580 (C=N), 1703 (C=O) cm^{-1}. ^1H NMR (DMSO−d_6, 300 MHz): δ (ppm) = 10.62 (CHO, s, 1H), 8.02 (d, 1H, $J = 8.4$ Hz), 7.47 (d, 1H, $J = 6.9$ Hz), 7.02 (d, 1H, $J = 6.9$ Hz), 3.60 (3H, s, $-OCH_3$). Mass, m/z (relative intensity): 224 ($M^{+.2}$). Calcd. (%) for $C_{10}H_7ClN_2O_2$: C; 53.95, H; 3.17, N; 12.58. Found: C; 53.94, H; 3.18, N; 12.59.

4.2 *Synthesis of 2-hydroxy-1,8-naphthyridine-3-carbaldehyde (2a)*

Solution of 2-chloro-3-formyl[1,8]naphthyridine (1 mmol) in 70% (10 ml) dilute acetic acid was placed in a beaker and irradiated in MW at 160 W for 5 min. On completion of the reaction monitored by TLC eluting the phase ethyl acetate: carbon tetrachloride (70:30), the reaction mixture was poured in to crushed ice. The product was filtered, washed with water, dried and was pure enough for further use. The same procedure was used for the synthesis of (**2b**,**c**).

4.2.1 General procedure for the synthesis of substituted 2H-pyrano[2,3-b]-1,8-naphthyridin-2-one (3a). To a mixture of 2-hydroxy-1,8-naphthyridine-3-carbaldehyde (1 mmol), and acetic anhydride (5 mmol) and catalytic amount of anhydrous sodium acetate was taken in a beaker and irradiated in MW at 160 W for 9 min. The completion of the reaction was checked by TLC eluting the phase ethyl acetate: carbon tetrachloride (70:30) and after the completion of reaction mixture was poured in ice-cold water. The solid separated was filtered, dried, and recrystallized from methanol to gave (**3a**). The same procedure was used for the

synthesis of compounds (**3a**–**c**). The physicochemical data for the synthesized compounds are as shown below.

4.2.2 2H-Pyrano[2,3-b]-1,8-naphthyridin-2-one (3a). Solid. Yield; 88% (MW), 56% (CM), Time; 9 min (MW), 10 hrs (CM), mp: 170 °C. FT−IR (KBr): 2960 (C−H), 1660 (C=O), ^{1}H NMR (DMSO-d_6, 300 MHz): δ (ppm) = 8.03 (d, 1H, J = 8.5 Hz), 7.73 (d, 1H, J = 8.4 Hz), 7.60 (d, 1H, J = 8.4 Hz), 7.49 (d, 1H, J = 6.9 Hz), 7.38 (d, 1H, J = 6.9 Hz), 7.00 (d, 1H, J = 6.9 Hz). Mass, m/z; 200 ($M^{\cdot+2}$). Calcd. (%) for $C_{11}H_6N_2O_2$: C; 66.67, H; 3.05, N;14.14. Found: C; 66.60, H; 3.00, N; 14.10.

4.2.3 7-Methyl-2H-pyrano[2,3-b]-1,8-naphthyridin-2-one (3b). Solid. Yield; 85% (MW), 55% (CM), Time; 9 min (MW), 11 hrs (CM), mp: 173 °C. FT−IR (KBr): 2962 (C−H), 1682 (C=O), ^{1}H NMR (DMSO-d_6, 300 MHz): δ (ppm) = 8.00 (d, 1H, J = 8.5 Hz), 7.64 (d, 1H, J = 8.4 Hz), 7.47 (d, 1H, J = 6.9 Hz), 7.40 (d, 1H, J = 6.9 Hz), 7.02 (d, 1H, J = 6.9 Hz), 2.60 (3H, s, $-CH_3$). Mass, m/z; 214 ($M^{\cdot+2}$). Calcd. (%) for $C_{12}H_8N_2O_2$: C; 67.92, H; 3.80, N;13.20. Found: C; 67.90, H; 3.79, N; 13.32.

4.2.4 7-Methoxy-2H-pyrano[2,3-b]-1,8-naphthyridin-2-one (3c). Solid. Yield; 87% (MW), 57% (CM), Time; 9 min (MW), 10 hrs (CM), mp: 172 °C. FT−IR (KBr): 2961 (C−H), 1680 (C=O), ^{1}H NMR (DMSO-d_6, 300 MHz): δ (ppm) = 8.02 (d, 1H, J = 8.5 Hz), 7.62 (d, 1H, J = 8.4 Hz), 7.52 (d, 1H, J = 8.4 Hz), 7.40 (d, 1H, J = 6.9 Hz), 7.01 (d, 1H, J = 6.9 Hz), 3.66 (3H, s, $-OCH_3$), Mass, m/z; 230 ($M^{\cdot+2}$). Calcd. (%) for $C_{12}H_8N_2O_3$: C; 63.16, H; 3.53, N;12.28. Found: C; 63.10, H; 3.50, N; 12.20.

4.3 *The synthesis of 2-mercapto-1,8-naphthyridine-3-carbaldehyde (4a)*

Solution of 2-chloro-3-formyl[1,8]naphthyridine (1 mmol) in dry DMF (5 ml), sodium sulphide (1.5 mmol, fused flakes) was taken in a beaker and irradiated in MW at 160 W for 4 min. The completion of the reaction monitored by TLC eluting the phase ethyl acetate: carbon tetrachloride (70:30), the reaction mixture was poured in to crushed ice (15 gm) and made acidic with acetic acid. The product was filtered and washed with water, dried and was pure enough for further use. The same procedure was used for the synthesis of other compounds (**4b**,**c**).

4.3.1 General procedure for the synthesis of substituted 2H-thiopyrano[2,3-b]-1,8-naphthyridin-2-one (5a). To a mixture of 2-mercapto-1,8-naphthyridine-3-carbaldehyde (1 mmol), and acetic anhydride (5 mmol) and catalytic amount of anhydrous sodium acetate was taken in a beaker and irradiated in MW at 160 W for 9 min. The completion of the reaction was checked by TLC eluting the phase ethyl acetate: carbon tetrachloride (70:30) and poured in ice-cold water. The solid separated was filtered, dried, and recrystallized from methanol to gave 2*H*-thiopyrano[2,3-*b*]-1,8-naphthyridin-2-one. The same procedure was used for the synthesis of other compounds (**5b**,**c**).

4.3.2 2H-Thiopyrano[2,3-b]-1,8-naphthyridin-2-one (5a). Solid. Yield; 83% (MW), 58% (CM), Time; 9 min (MW), 11 hrs (CM), mp: 176 °C. FT−IR (KBr): 2960 (C−H), 1670 (C=O), ^{1}H NMR (DMSO-d_6, 300 MHz): δ (ppm) = 8.03 (d, 1H, J = 8.5 Hz), 7.75 (d, 1H, J = 8.4 Hz), 7.65 (d, 1H, J = 8.4 Hz), 7.44 (d, 1H, J = 6.9 Hz), 7.20 (d, 1H, J = 6.9 Hz),

7.00 (d, 1H, $J = 6.9$ Hz). Mass, m/z; 216 ($M^{\cdot+2}$). Calcd. (%) for $C_{11}H_6N_2O_1S_1$: C; 61.67, H; 2.82, N;13.08, S;14.97. Found: C; 61.65, H; 2.85, N; 13.12, S; 14.90.

4.3.3 7-Methyl-2H-thiopyrano[2,3-b]-1,8-naphthyridin-2-one (5b). Solid. Yield; 85% (MW), 56% (CM), Time; 9 min (MW), 12 hrs (CM), mp: 178 °C. FT−IR (KBr): 2960 (C−H), 1685 (C=O), ^{1}H NMR (DMSO-d_6, 300 MHz): δ (ppm) = 8.01 (d, 1H, $J = 8.5$ Hz), 7.58 (d, 1H, $J = 8.4$ Hz), 7.47 (d, 1H, $J = 6.9$ Hz), 7.39 (d, 1H, $J = 6.9$ Hz), 7.02 (d, 1H, $J = 6.9$ Hz), 2.65 (3H, s, $-CH_3$), Mass, m/z; 230 ($M^{\cdot+2}$). Calcd. (%) for $C_{12}H_8N_2O_1S_1$: C; 63.14, H; 3.53, N; 12.27, S; 14.05. Found: C; 63.10, H; 3.52, N; 12.25, S; 14.10.

4.3.4 7-Methoxy-2H-thiopyrano[2,3-b]-1,8-naphthyridin-2-one (5c). Solid. Yield; 87% (MW), 59% (CM), Time; 9 min (MW), 12 hrs (CM), mp: 180 °C. FT−IR (KBr): 2960 (C−H), 1700 (C=O), ^{1}H NMR (DMSO-d_6, 300 MHz): δ (ppm) = 8.03 (d, 1H, $J = 8.5$ Hz), 7.63 (d, 1H, $J = 8.4$ Hz), 7.55 (d, 1H, $J = 8.4$ Hz), 7.35 (d, 1H, $J = 6.9$ Hz), 7.01 (d, 1H, $J = 6.9$ Hz), 3.40 (3H, s, $-OCH_3$), Mass, m/z; 246 ($M^{\cdot+2}$). Calcd. (%) for $C_{12}H_8N_2O_2S_1$: C; 59.00, H; 3.30, N; 11.47, S; 13.13. Found: C; 59.03, H; 3.31, N; 11.40, S; 13.11.

4.4 *Preparation of sodium selenide*

Selenium powder (1 g, 0.013 mol) was taken in 500 ml beaker containing water (25 ml). Beaker was kept in the ice water bath to control the heat; sodium borohyride (1 g, 0.026 mol) was added in small portion, with stirring. Considerable foaming (liberation of hydrogen), occurred immediately. After addition of sodium borohydride water was added (25 ml) along the side of the beaker and stirred for 15 min, colourless or deep reddish NaHSe resulted and was readily used with out further purification.

4.4.1 2-Seleno-1,8-naphthyridine-3-carbaldehyde (6a–c). To a solution of sodium hydrogen selenide (0.013 mol) and sodium borohydride (0.026 mol) in water (5 ml) was added to 2-chloro-3-formyl[1,8]naphthyridine (0.01 mol) in ethanol (8 ml). The reaction mixture was taken in a beaker and irradiated in MW at 160 W for 5 min, cooled, poured into ice (10 g) and acidified with dil. (4N) HCl. The resultant solid obtained was washed with 30 ml H_2O and recrystallized from excess alcohol.

4.4.2 General procedure for the synthesis of substituted 2H-selenopyrano[2,3-b]-1,8-naphthyridin-2-one (7a). A mixture of 2-seleno-1,8-naphthyridine-3-carbaldehyde (1 mmol), and acetic anhydride (5 mmol) and a catalytic amount of anhydrous sodium acetate was taken in a beaker and irradiated in MW at 160 W for 9 min. The completion of the reaction was checked by TLC eluting the phase ethyl acetate: carbon tetrachloride (70:30) and poured in ice-cold water. The solid separated was filtered, dried, and recrystallized from methanol to gave 2*H*-selenopyrano[2,3-*b*]-1,8-naphthyridin-2-one. The spectral data for the synthesized compounds are as shown below. The same procedure was used for the synthesis of other compounds (**9b**,**c**).

4.4.3 2H-Selenopyrano[2,3-b]-1,8-naphthyridin-2-one (7a). Solid. Yield; 82% (MW), 56% (CM), Time; 9 min (MW), 12 hrs (CM), mp: 175 °C. FT−IR (KBr): 2960 (C−H), 1660 (C=O), ^{1}H NMR (DMSO-d_6, 300 MHz): δ (ppm) = 8.03 (d, 1H, $J = 8.5$ Hz), 7.85 (d, 1H,

$J = 8.4$Hz), 7.70 (d, 1H, $J = 8.4$ Hz), 7.52 (d, 1H, $J = 8.4$ Hz), 7.40 (d, 1H, $J = 6.9$ Hz), 7.00 (d, 1H, $J = 6.9$ Hz). Mass, m/z; 263 ($M^{\cdot+2}$). Calcd. (%) for $C_{11}H_6N_2O_1Se_1$: C; 50.59, H; 2.32, N; 10.73, Se; 30.24. Found: C; 50.55, H; 2.30, N; 10.71, Se; 30.20.

4.4.4 7-Methyl-2H-selenopyrano[2,3-b]-1,8-naphthyridin-2-one (7b). Solid. Yield; 80% (MW), 55% (CM), Time; 9 min (MW), 11 hrs (CM), mp: 178 °C. FT−IR (KBr): 2960 (C−H), 1660 (C=O), ^{1}H NMR (DMSO-d_6, 300 MHz): δ (ppm) = 8.01 (d, 1H, $J = 8.6$ Hz), 7.72 (d, 1H, $J = 8.4$ Hz), 7.49 (d, 1H, $J = 6.9$ Hz), 7.38 (d, 1H, $J = 6.9$ Hz), 7.02 (d, 1H, $J = 6.9$ Hz), 2.58 (3H, s, $-CH_3$). Mass, m/z; 277($M^{\cdot+2}$). Calcd. (%) for $C_{12}H_8N_2O_1Se_1$: C; 52.38, H; 2.93, N; 10.18, Se; 28.70. Found: C; 52.35, H; 2.91, N; 10.15, Se; 28.72.

4.4.5 7-Methoxy-2H-selenopyrano[2,3-b]-1,8-naphthyridin-2-one (7c). Solid. Yield; 83% (MW), 58% (CM), Time; 9 min (MW), 11 hrs (CM), mp: 176 °C. FT−IR (KBr): 2960 (C−H), 1660 (C=O), ^{1}H NMR (DMSO-d_6, 300 MHz): δ (ppm) = 8.02 (d, 1H, $J = 8.5$Hz), 7.68 (d, 1H, $J = 8.4$ Hz), 7.56 (d, 1H, $J = 8.4$ Hz), 7.41 (d, 1H, $J = 6.9$ Hz), 7.01 (d, 1H, $J = 6.9$ Hz), 3.60 (3H, s, $-OCH_3$), Mass, m/z; 293 ($M^{\cdot+2}$). Calcd. (%) for $C_{12}H_8N_2O_2Se_1$: C; 49.50, H; 2.77, N; 9.62, Se; 27.12. Found: C; 49.51, H; 2.76, N; 9.60 Se; 27.10.

Acknowledgements

One of the authors (R.N.) is thankful to Kuvempu University, for awarding research fellowship and SIF, IISc, Bangalore for ^{1}H NMR and mass spectral data.

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