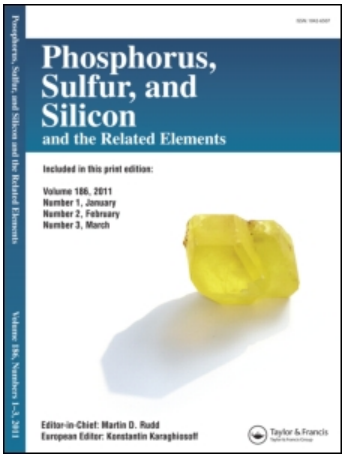

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*A series of some novel substituted selenolopyrano[2,3-b]quinolines **2a-c**, **3a-c**, and **4a-c** were prepared from substituted 2-seleno-3-formyl-quinoliness **1a-c**, which on reaction with ethyl acetoacetate, diethyl malonate, and ethyl cyanoacetate under microwave irradiation in the presence of piperidine gives the title compounds. The structure of newly synthesized compounds have been evaluated based on analytical, IR, ^1H NMR, and mass spectral data.*

Keywords Formylquinolines; Microwave irradiation; Selenolopyrano[2,3-*b*]quinolines

INTRODUCTION

Interest in selenium-containing therapeutics has grown over the last 30 years[1] simple organoselenium compounds have been prepared, such as selenazolopyrimidone that showed antitumor activity against mouse leukemia.[2]Aminoethylphenylselenide showed excellent antihypertensive activity, and selenazine derivatives exhibited both antibacterial and antitumor activity.[3] Despite this, the major therapeutic benefit that selenium offers currently is in the form of dietary supplements.[4] Selenium is an essential trace element, and dietary deficiency can lead to ailments including gum disease, as well as debilitating conditions, such as Keshan's disease. The biochemistry and pharmacology of selenium is an intense current interest. Selenium is now known to be intimately involved in the activity of enzymes—such as glutathione peroxidase and thioredoxin reductase—that catalyze chemistry essential to the protection of biomolecules against oxidative stress and free radical damage.[5]

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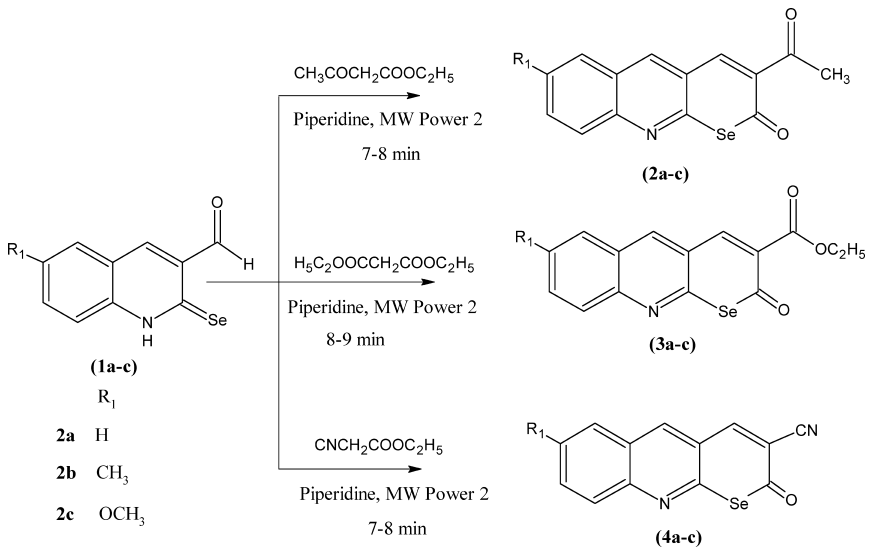
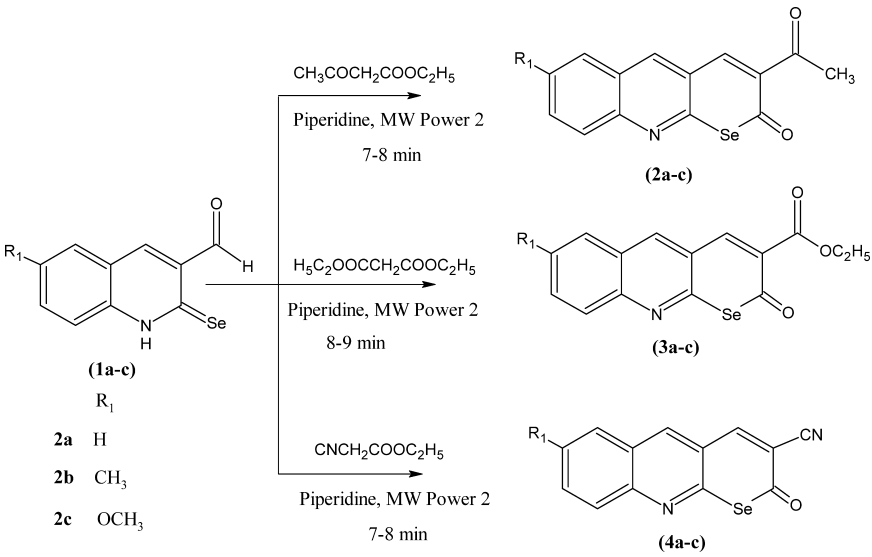


Ebselen potent antioxidants that also acts as a glutathione peroxidase mimic;[6] recently, Nithyadevi et al. have reported synthesis and cytotoxic activities of some new Selenolo(2,3-b) quinoline-2-carboxylic ethyl ethers.[7] Gopal et al. have reported the interaction binding of 4-Butylaminopyrimido-[4′,5′:4,5] selenolo (2,3-b)quinoline to DNA.[8] Russian workers have reported that selenium isosters of methapyrilene type compound are two to five times as active antihistamines as their sulfur analogs, and the duration of their action was twelve times longer.[9]

Several polycyclic analogues of natural or synthetic antitumor agents are well known and have attracted considerable interest because of their significant anticancer activity.[10] There is a evidence that anticancer activity is due to the intercalation of these compounds between the base pairs of DNA and interference with normal functioning of the enzyme topoisomerase II, which is involved in the breaking and releasing of DNA strands.[11] The intercalative binding of these drugs is due to the presence of planar linearly fused tri and tetracyclic system.[12]

In view of the above findings and in continuation of our work on microwave assisted synthesis of biologically important quinoline containing heterocyclic compounds[13–16] herein, we wish to report a simple, convenient microwave assisted synthesis of title compounds by the reaction of active methylene compounds with 2-seleno-3-formyl-quinolines **1a-c**.



SCHEME 1

RESULTS AND DISCUSSION

Recently, we have reported the synthesis of 2-seleno-3-formyl-quinolines[17] (**1a-c)** were reacts with, ethyl acetoacetate, diethyl malonate and ethyl cyanoacetate in the presence of a catalytic amount of piperidine in anhydrous DMF, affords selenolopyrano [2,3-*b*] quinolin-2-one derivatives **2a-c**, **3a-c**, and **4a-c** in good yields (Scheme 1). The structure of all the newly synthesized compounds were elucidated on the basis of elemental analysis, IR, ^{1}H NMR, and mass spectral data. The IR spectrum of compound (**2a-c)** showed an absence of tautomeric SeH, NH, groups and CHO stretching frequency in the region 3150–3300, and 1650 cm^{-1}, which appeared in the 2-seleno-3-formyl-quinolines **(1a-c).** Further, the appearance of new band in the region 1620–1625 cm^{-1} attributed to a carbonyl group of selenolopyrano ring and another in the region 1655–1660 cm^{-1} assigned to $COCH_3$ in the newly formed ring was the indication of the ring closure. The ^{1}H NMR spectrum of (**2a-c)** exhibited a singlet in the region δ 2.65–2.70 attributed to a methyl group of the $COCH_3$ in the newly formed selenolopyrano ring system on the quinoline nucleus, C4-H proton showed a singlet in the region δ 9.05–9.10, C5-H showed singlet in the region δ 8.50–8.60, remaining aromatic protons were resonated in the region between δ 7.25–8.20. Compound **2a** showed the molecular ion peak at m/e 302(M+) and 304(M+2). Compound **2b** showed the molecular ion peak

at m/e 316(M+) and 318(M+2) and Compound **2c** showed the molecular ion peak at m/e 332(M+) and 334(M+2) consistent with the molecular formula $C_{14}H_9NO_2Se$, $C_{15}H_{11}NO_2Se$ and $C_{15}H_{11}NO_3Se$, respectively.

In light of the foregoing result, the reaction of 2-seleno-3-formyl-quinoline **(1a-c)** with diethyl malonate in anhydrous DMF containing a piperidine afforded (**3a-c)** in 8 min the structure of **(3a-c)** was confirmed based on elemental and spectral data.

The IR spectrum of **3a-c** exhibited absorption band in the region 1615–1620 cm^{-1} due to CO of the selenolopyrano ring and another band in the region 1708–1715 cm^{-1} corresponding to $COOC_2H_5$ of the newly formed selenolopyrano ring on the quinoline nucleus. No band was observed in the region 3150–3300 cm^{-1} corresponds to the tautomeric SeH and NH groups. The ^1H NMR spectrum of a compound **3a-c** showed a triplet in the region δ 1.40–1.50 which corresponds to CH_3 of OCH_2CH_3 and a quartet in the region δ 4.40–4.50 attributed to CH_2 of OCH_2CH_3, C4-H proton showed a signal in the region δ 9.00–9.10, C5-H in the region δ 8.35–8.40, signals for remaining aromatic protons appeared between δ 7.25–8.55. Compound **3a** showed the molecular ion peak at m/e 332(M+) and 334(M+2). Compound **3b** showed the molecular ion peak at m/e 346(M+) and 348(M+2) and Compound **3c** showed the molecular ion peak at m/e 362(M+) and 364(M+2) consistent with the molecular formula $C_{14}H_9NO_2Se$, $C_{16}H_{13}NO_3Se$ and $C_{16}H_{13}NO_4Se$, respectively.

On the other hand, treatment of 2-seleno-3-formyl-quinolines **(1a-c)** with ethylcyanoacetate afforded **(4a-c)** in the same experimental conditions. The IR spectrum of **4a-c** showed absorption band in the region 2220–2225 cm^{-1} corresponding to a cyano group stretching frequency and in the region 1625–1630 cm^{-1} corresponding to CO group of the selenolopyrano ring. In ^1H NMR compound **4a-c** showed a signal in the region δ 9.00–9.10 due to C4-H proton, C5-H proton showed a signal in the region δ 8.50–8.60 remaining aromatic protons were resonated in the region δ 7.25–8.50. Compound **4a** showed the molecular ion peak at m/e 302(M+) and 304(M+2). Compound **4b** showed the molecular ion peak at m/e 316(M+) and 318(M+2) and Compound **4c** showed the molecular ion peak at m/e 332(M+) and 334(M+2) consistent with the molecular formula $C_{13}H_6N_2OSe$, $C_{14}H_8N_2OSe$, and $C_{14}H_8N_2O_2Se$, respectively.

EXPERIMENTAL

Melting points were determined in open capillaries and are uncorrected. The FT-IR spectra were recorded on NICOLETAVATAR 360-FTIR instrument by using KBr pellets. The ^1H NMR were recorded on a BRUCKER AMX-400 spectrometer operating at 400 MHz. Mass

spectra were recorded on AGILENT LC-MSD-TRAP-XCT mass spectrometer Elemental analyses were done on Vario EL. CHNOS elemental analyzer.

The General Microwave Procedure for the Synthesis of 3-Acetyl-2*H*-selenolopyrano [2,3-*b*]quinolin-2-one (2a)

Mixture of 2-seleno-3-formyl-quinoline **1a** (0.944 g, 0.0040 mol), ethyl acetoacetate (0.585 g, 0.0045 mol) and piperidene (5 drops) were mixed thoroughly with 5 mL of anhydrous DMF. The mixture was then subjected to microwave irradiation for about 8 min at an interval of 30 s at 160 W, to prevent excess evaporation of the solvent after the completion of the reaction (monitored by TLC). The reaction mixture was poured into ice-cold water and stirred well; the solid obtained was filtered, washed with water, dried, and recrystallized from acetonitrile or from aqueous DMF to give 2.718 g (90%) of **2a**. Similarly, other compounds were prepared in the same way with 84–90% yield.

The General Conventional Method for the Synthesis of 3-Acetyl-2*H*-selenolopyrano [2,3-*b*]quinolin-2-one (2a)

A mixture of 2-seleno-3-formyl-quinoline **1a** (0.944 g, 0.0040 mol), ethyl acetoacetate (0.585 g, 0.0045 mol) and piperidene (5 drops) and 10mL of anhydrous DMF were taken in 100-ml round-bottom flask, refluxed on water bath for about 6 h after the completion of the reaction (TLC). The reaction mixture was poured into ice-cold water and stirred; the solid obtained was filtered and dried. The crude product was recrystallized from aqueous DMF

3-Acetyl-2H-selenolopyrano [2,3-b]quinolin-2-one (2a)

Irradiation time: 7 min, Yield: 90% (MW), 71% (Conventional). M.p.: 225–227°C. IR (KBr) cm^{-1} 1620 cm^{-1} (CO), 1655 ($COCH_3$). ^1H NMR (400 MHz, DMSO-d_6) δ 2.70 (s, 3H, $COCH_3$), 9.10 (s, 1H, C4), 8.50 (s, 1H, C5), 7.50–8.20 (m, 4H, Ar—H). MS m/z: 302 (M^+), and 304 (M+2). Anal. calcd. for $C_{14}H_9NO_2Se$: C, 55.64; H, 3.00; N, 4.64. Found: C, 55.60; H, 3.33; N, 4.68.

3-Acetyl-7-methyl-2H-selenolopyrano[2,3-b] quinolin-2-one (2b)

Prepared from methyl derivative of quinoline **1b** (1.000 g, 0.0040 mol) and ethylacetoacetate (0.585 g, 0.0045 mol), the compound was recrystallized from aqueous DMF. Irradiation time: 8 min, Yield: 88% (MW), 72% (Conventional). M.p.: 234–236°C. IR (KBr) cm^{-1} 1625 (CO).

1660 ($COCH_3$). ^{1}H NMR (400 MHz, DMSO-d_6) δ 2.65 (s, 3H, $COCH_3$), 9.05 (s, 1H, C4), 8.55 (s, 1H, C5), 7.40–8.50 (m, 3H, Ar—H). MS m/z: 316 (M^+), and 318 (M+2). Anal. calcd. for $C_{15}H_{11}NO_2Se$: C, 56.97; H, 3.51; N, 4.43. Found: C, 56.99; H, 3.54; N, 4.47.

3-Acetyl-7-methoxy-2H-selenolopyrano[2,3-b]quinolin-2-one (2c)

Prepared from methoxy derivative of quinoline **1c** (1.064 g, 0.0040 mol) and ethylacetoacetate (0.585g, 0.0045mol); the compound was recrystallized from aqueous DMF. Irradiation time: 7 min, Yield: 89% (MW), 68% (Conventional). M.p.: 241–243°C. IR (KBr) cm^{-1} 1625 (CO), 1660 ($COCH_3$). ^{1}H NMR (400 MHz, DMSO-d_6) δ 2.65 (s, 3H, $COCH_3$), 9.10 (s, 1H, C4), 8.60 (s, 1H, C5), 7.25–8.45 (m, 3H, Ar—H). MS m/z: 332 (M^+), and 334 (M+2). Anal. calcd. for $C_{15}H_{11}NO_3Se$: C, 54.23; H, 3.34; N, 4.22. Found: C, 54.25; H, 3.35; N, 4.25.

Ethyl 2-oxo-2H-selenolopyrano[2,3-b]quinoline-3-carboxylate (3a)

Prepared from quinoline **1a** (0.944 g, 0.0040 mol) and diethylmelonate (0.720 g, 0.0045 mol). The compound recrystallized from aqueous DMF. Irradiation time: 8 min, Yield: 88% (MW), 70% (Conventional). M.p.: 210–215°C. IR (KBr) cm^{-1} 1616 (CO), 1708 ($COOC_2H_5$). ^{1}H NMR (400 MHz, DMSO-d_6) δ 1.45 (t, CH_3 of OCH_2CH_3), 4.40 (q, OCH_2), 9.00 (s, 1H, C4), 8.40 (s, 1H, C5), 7.25–8.50 (m, 4H, Ar—H). MS m/z: 332 (M^+), and 334 (M+2). Anal. calcd. for $C_{15}H_{11}NO_3Se$: C, 54.23; H, 3.34; N, 4.22. Found: C, 54.27; H, 3.37; N, 4.18.

Ethyl 7-methyl-2-oxo-2H-selenolopyrano [2,3-b] quinoline-3-carboxylate (3b)

Prepared from methyl derivative of quinoline **1b** (1.000g, 0.0040mol) and diethylmelonate (0.720 g, 0.0045 mol). The compound recrystallized from aqueous DMF. Irradiation time: 8 min, Yield: 84% (MW), 73% (Conventional). M.p.: 221–223°C. IR (KBr) cm^{-1} 1615, (CO), 1715 ($COOC_2H_5$). ^{1}H NMR (400 MHz, DMSO-d_6) δ 1.40 (t, OCH_2CH_3), 4.45 (q, OCH_2), 9.05 (s, 1H, C4), 8.35 (s, 1H, C5), 7.30–8.55 (m, 3H, Ar—H). MS m/z: 346 (M^+), and 348 (M+2). Anal. calcd. for $C_{16}H_{13}NO_3Se$: C, 55.50; H, 3.78; N, 4.05. Found: C, 55.53; H, 3.73; N, 4.08.

Ethyl 7-Methoxy-2-oxo-2H-selenolopyrano[2,3-b]quinoline-3-carboxylate (3c)

Prepared from methoxy derivative of quinoline **1c** (1.064 g, 0.0040 mol) and diethylmelonate (0.720 g, 0.0045 mol). The compound

recrystallized from aqueous DMF. Irradiation time: 9 min, Yield: 87% (MW), 65% (Conventional). M.p.: 231–233°C. IR (KBr) cm^{-1} 1620 (CO), 1715 ($COOC_2H_5$). ^1H NMR (400 MHz, DMSO-d_6) δ 1.50 (t, OCH_2CH_3), 4.50 (q, OCH_2), 9.10 (s, 1H, C4), 8.35 (s, 1H, C4), 7.25–8.50 (m, 3H, Ar—H). MS m/z: 362 (M^+), and 364 (M+2). Anal. calcd. for $C_{16}H_{13}NO_4Se$: C, 53.05; H, 3.62; N, 3.87. Found: C, 53.08; H, 3.65; N, 3.84.

2-Oxo-2H-selenolopyrano[2,3-b]quinoline-3-carbonitrile (4a)

Prepared from quinoline **1a** (0.944 g, 0.0040 mol) and ethyl cyanoacetate (0.508 g, 0.0045 mol). The compound recrystallized from aqueous DMF. Irradiation time: 7 min, Yield: 89% (MW), 68% (Conventional). M.p.: 218–220°C. IR (KBr) cm^{-1} 2225 (CN), 1625 (CO). ^1H NMR (400 MHz, DMSO-d_6), δ 9.00 (s, 1H, C4), δ 8.50 (s, 1H, C5), 7.25–8.30 (m, 4H, Ar—H). MS m/z: 285 (M^+), and 287 (M+2). Anal. calcd. for $C_{13}H_6N_2OSe$: C, 54.75; H, 2.12; N, 9.82. Found: C, 54.78; H, 2.15; N, 9.84.

7-Methyl-2-oxo-2H-selenolopyrano[2,3-b]quinoline-3-carbonitrile (4b)

Prepared from methyl derivative of quinoline **1b** (1.000 g, 0.0040 mol) and ethyl cyanoacetate (0.508 g, 0.0045 mol).The compound recrystallized from aqueous DMF. Irradiation time: 8 min, Yield: 90% (MW), 70% (Conventional). M.p.: 229–231°C. IR (KBr) cm^{-1} 2220 (CN), 1630 (CO). ^1H NMR (400 MHz, DMSO-d_6), δ 2.60 (3H, s, CH_3), 9.05 (s, 1H, C4), 8.60 (s, 1H, C5), 7.30–8.40 (m, 3H, Ar—H). MS m/z: 299 (M^+), and 301 (M+2). Anal. calcd. for $C_{14}H_8N_2OSe$: C, 56.20; H, 2.70; N, 9.36. Found: C, 56.24; H, 2.66; N, 9.32.

7-Methoxy-2-oxo-2H-selenolopyrano[2,3-b]quinoline-3-carbonitrile (4c)

Prepared from methoxy derivative of quinoline **1c** (1.064 g, 0.0040 mol) and ethyl cyanoacetate (0.508 g, 0.0045 mol). The compound recrystallized from aqueous DMF. Irradiation time: 8 min, Yield: 89% (MW), 71% (Conventional). M.p.: 236–238°C. IR (KBr) cm^{-1} 2223 (CN), 1625 (CO). ^1H NMR (400 MHz, DMSO-d_6), δ 9.10 (s, 1H, C4), 8.60 (s, 1H, C5), 7.30–8.45 (m, 3H, Ar—H). MS m/z: 315 (M^+), and 317 (M+2). Anal. calcd. for $C_{14}H_8N_2O_2Se$: C, 53.35; H, 2.56; N, 8.89. Found: C, 53.38; H, 2.59; N, 8.93.

CONCLUSION

In conclusion, a simple, efficient, and environmentally benign method has been developed for the synthesis of selenolopyrano[2,3-*b*]quinolines

under microwave irradiation conditions. This microwave irradiation method is superior from the view of yield and reaction time, when compared to the conventional (thermal) method.

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