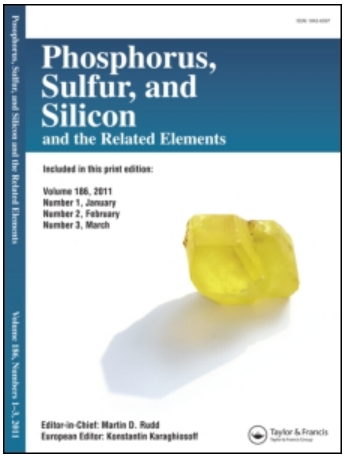

Phosphorus, Sulfur, and Silicon and the Related Elements
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713618290

AN EFFICIENT SYNTHESIS OF DIHYDRO SELENOLO (2,3-*b*)QUINOLINE-2-CARBOXYLIC ETHYL ESTERS AND 2-SELENOXO-1, 2-DIHYDRO-3-CARBETHOXY ETHYL QUINOLINES—THEIR ANTI-BACTERIAL STUDIES

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Online publication date: 16 August 2010

Phosphorus, Sulfur, and Silicon, 179:2355–2364, 2004

ISSN: 1042-6507 print / 1563-5325 online
DOI: 10.1080/10426500490485101

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AN EFFICIENT SYNTHESIS OF DIHYDRO SELENOLO (2,3-b)QUINOLINE-2-CARBOXYLIC ETHYL ESTERS AND 2-SELENOXO-1, 2-DIHYDRO-3-CARBETHOXY ETHYL QUINOLINES—THEIR ANTI-BACTERIAL STUDIES

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(Received March 16, 2004; accepted April 28, 2004)

The compounds dihydro selenolo(2,3-b)quinoline-2-carboxylic ethyl esters and 2-selenoxo-1, 2-dihydro-3-carbethoxy ethyl quinolines were synthesized in varying yields upon reacting 3-(2-chloro-3-quinolyl)-acrylic methyl esters with the nucleophilic reagent sodium hydrogen selenide in ethanol medium under nitrogen atmosphere.

Keywords: Condensation; cyclization using NaHSe in absolute ethanol; dehydroxychlorination; esterification; hydrolysis; Vilsmeier-Haack Reaction

INTRODUCTION

Organoselenium compounds have been extensively investigated due to their close association with various types of activities such as antifungal and antibacterial activities,[1–3] cardiovascular and antihistaminic actions,[4] antiadrenalin, antiradiation,[5] antileukemic activity,[6] antitumour activity,[7] analgesic, antirhuematic and antipyretic activities,[8] parathyroid scanning for the deduction of lymphomas,[9]etc.

Selenium also exhibits protective effects,[10] transcriptional activities,[11] cell cycle control,[12] growth influence,[13] preterm delivery,[13] prevention of endemic arsenism,[14] etc.

We wish to thank Bharathiar University for the award of URF to one of us (V. N). We also thank the services rendered by the Dept of Chemistry Bharathiar University at Coimbatore, IICT at Hyderabad and Sophisticated Instumentation facility at IISc Bangalore for recording elemental analysis, Proton NMR, IR and Mass spectra. We greatly acknowledge Dr. P. Lakshmanaperumalsamy and Mr. Subashkumar-Division of microbiology, Dept of Environmental Sciences, Bharathiar University, Coimbatore, for the microbial studies rendered by them.

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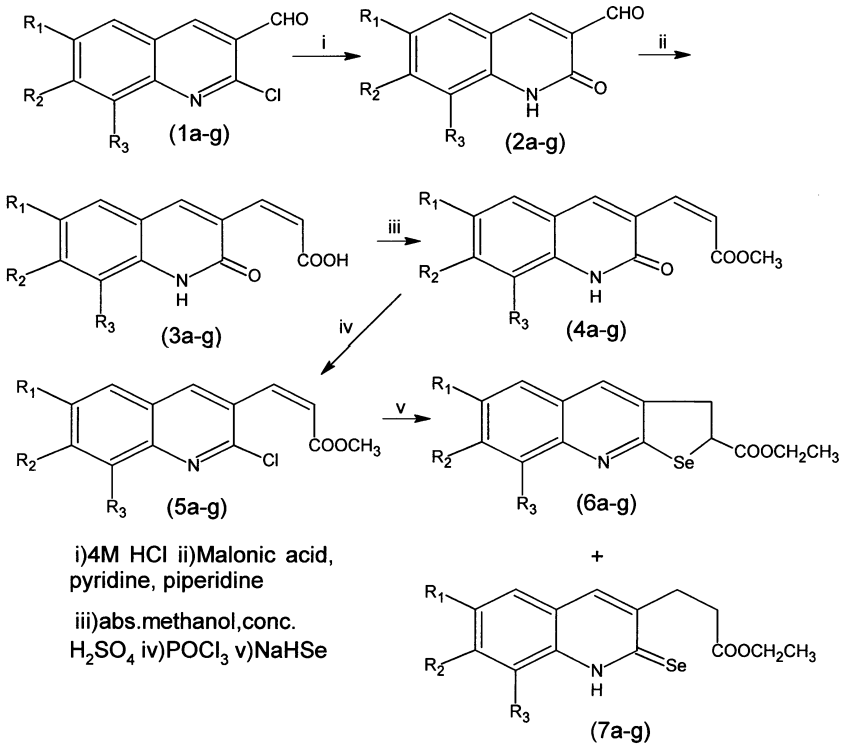
Even though there are many inorganic selenium reagents and organoselenium reagents, the role of NaHSe is excellent when it is used for the introduction of selenium into organic molecules. The ethanolic solution of sodium hydrogen selenide. The reducing agent is advantageous in that it provides a superior solvent for nucleophilic displacement reactions in water-insoluble or hydrolysis-sensitive organic compounds. Early workers from our laboratory have synthesised selenolo(2,3-b)quinolines[15–17] using 2-quinolone-3-ethanols and 2-chloro-3-vinyl quinolines as the starting compounds. In view of their expected activities, we pursued our investigation on selenium heterocycles, and we report herein a convenient synthesis of dihydroselenolo(2,3-b)quinoline-2-carboxylic ethyl esters and 2-selenoxo-1, 2-dihydro-3-carbethoxy ethyl quinolines from 3-(2-chloro-3-quinolyl)acrylic esters[18] (**5**) (Scheme 1).

EXPERIMENTAL

Melting points were determined using Raaga melting point apparatus and were uncorrected. The IR spectra were recorded on FTIR 8201(PC)S spectrometer as KBr pellets, and the absorption frequencies are expressed in reciprocal centimeters (cm^{-1}). Proton NMR spectra were recorded on a Gemini-200 MHz or on a Varian AMX 400 spectrometer in $CDCl_3$. The chemical shifts were expressed in δ (ppm) downfield from tetramethylsilane (TMS) as an internal standard. Elemental analysis was performed by Elementar Analyser Vario EL III, and the values are within the permissible limits (± 0.5). The mass spectra were recorded by EIMS technique on an Autospec mass spectrometer. The crude products were checked by thin layer chromatography (TLC) and purified by column chromatography using silica gel (60–120 mesh).

In our present work selenium heterocycles were synthesized from the potential precursor 2-chloro-3-formyl quinolines, which were prepared by following O. Meth-cohn procedure.[19] 2-chloro-3-formyl quinolines (**1**) were converted to the oxo compounds (**2**) by refluxing with 4MHCl. These were then condensed with malonic acid under the conditions of Knoevenagel reaction to furnish 3-(2-oxo-1,2-dihydro-3-quinolyl)acrylic acids[20] (**3**).

Esterification of (**3**) in absolute methanol and concentrated sulphuric acid at reflux temperature for 5–6 h furnished 3-(2-oxo-3-quinolyl)acrylic methyl ester[18] (**4**) (Scheme 1). Upon dehydroxychlorination with freshly distilled phosphorus oxychloride, the compound (**4**) resulted in 3-(2-chloro-3-quinolyl)acrylic methyl ester (**5**).



a) $R_1 = R_2 = R_3 = H$.

b) $R_1 = R_3 = H$, $R_2 = CH_3$

c). $R_1 = R_2 = H$, $R_3 = CH_3$,

d) $R_1 = OCH_3$, $R_2 = R_3 = H$.

e) $R_1 = R_2 = H$, $R_3 = OCH_3$.

f) $R_1 = H$, $R_2 = R_3 =$ -CH=CH-CH=CH-.

g) $R_1 = R_3 = CH_3$, $R_2 = H$.

SCHEME 1

Preparation of dihydroselenolo(2,3-b)-quinoline-2-carboxylic ethyl esters and 2-selenoxo-1,2-dihydro-3-carbethoxy ethyl quinolines (6) and (7)

To a freshly prepared solution of sodium hydrogen selenide[21] under nitrogen atmosphere (selenium powder 1.12 g, 0.01417 mole and sodium

borohydride 0.0134 mole, 0.505 g) in ethanol, chloro ester (**5**) (1 g, 0.00808 mole) was added and heated under reflux temperature for 5–6 h on a steam bath. Thereafter, the solution was evaporated and the residue was dissolved in chloroform followed by washing with water. The organic extract was dried and evaporated. Thin layer chromatography (TLC) analysis of the residue showed three spots. Column chromatography was used to purify the crude product. The isolated products were recrystallized from petroleum ether. The first product was left as an unidentified one, since it was a pasty mass.

The second and third products were identified as dihydroselenolo(2,3-b)quinoline-2-caboxylic ethyl esters (**6**) and 2-selenoxo-1,2-dihydro-3-carbethoxy ethyl quinolines (**7**), respectively.

Antibacterial activity of dihydroselenolo(2, 3-b)quinoline-2-carboxylic ethyl ester (**6a**), 8-methyl-dihydroselenolo(2,3-b)quinoline-2-carboxylic ethyl ester (**6c**), 6-methoxy dihydroselenolo(2,3-b)quinoline-2-carboxylic ethyl ester (**6d**), and 8-methyl-2-selenoxo-1,2-dihydro-3-carbethoxy ethyl quinoline (**7c**) were screened for the in vitro growth inhibitory activity against the pathogens *Escherichia coli, Salmonella typhi, Staphylococcus aureus*, and *Pseudomonas flourosence.*

Methodology for Antibacterial Studies

Antibacterial activity of the synthesized compounds were determined by Disc diffusion technique.[22] The bacterial inoculum 10^5 of cells were enriched in to 5 ml of the nutrient broth (NaCl-5.0 g, Peptone-5 g, beef extract-3 g, yeast extract-3 g in 1000 ml of distilled water, pH = 7.3 ± 0.2). Enriched bacterial cells were swabbed onto Muller Hinton Agar medium (Himedia India). The test compounds were dissolved in $CHCl_3$ to a final concentration of 2, 1, and 0.5% and soaked in filter paper discs (5 mm diameters and 1 mm thickness). These discs were placed on the seeded plates and incubated at 37°C for 24 h. The zones of inhibition around the discs were measured after 24 h. Norfloxacin(Nx) (10 mcg) and Amoxycillin(Ac) (30 mcg) were used as the standard to compare the antibacterial activity of the compounds.

Physical and Spectroscopic Data of 6 and 7

Dihydroselenolo(2,3-b)-Quinoline-2-carboxylic Ethyl Ester (6a)

Colorless crystals, yield, 20%, m.p. 99–100°C. Calc.: C, 54.91; H, 4.27; N, 4.57. Found: C, 55.03: H, 4.30: N, 4.59. IR(KBr, cm^{-1}): 1722 (—C═O of ester), 1172 (—C—O of ester). ^{1}H NMR($CDCl_3$)δppm: 1.34 (t, J = 7.2 Hz, CH_3 of ester), 4.25 (q, J = 7.2 Hz, —CH_2 of ester), 3.61–

3.65 (m, —CH_2—CH—), 7.24–7.92 (m, 5H, C_4, C_5, C_6, C_7, C_8-H), M F: $C_{14}H_{13}O_2NSe$, Mass (m/z)M^+, 306.

7-Methyl-dihydroselenolo(2,3-b)quinoline-2-carboxylic Ethyl Ester (6b)

Colorless crystals, yield, 21%, m.p. 152–153°C. Calc.: C, 56.25; H, 4.72; N, 4.37. Found:, C, 56.20: H, 4.62; N, 4.35. IR(KBr, cm^{-1}): 1711 (C=O of ester), 1252 (—C—O of ester). ^{1}H NMR($CDCl_3$)δppm: 1.44 (t, $J = 7.2$ Hz, CH_3 of ester), 4.45 (q, $J = 7.2$ Hz, CH_2 of ester), 2.62 (s, 3H, —CH_3), 3.68–3.73 (m, —CH_2—CH—), 8.26 (s, 1H, C_8-H), 7.86 (d, $J = 9.4$ Hz, C_5-H), 7.43 (d, $J = 8.2$ Hz, C_6-H), 8.58 (s, C_4-H). M.F: $C_{15}H_{15}O_2NSe$. Mass (m/z)M^+, 320.

8-Methyl-dihydroselenolo(2,3-b)quinoline-2-carboxylic Ethyl Ester (6c)

Colorless crystals, yield, 22%, m.p. 94–95°C. Calc.: C, 56.25; H, 4.72; N, 4.37. Found: C, 56.20; H, 4.52; N, 4.35. IR(KBr, cm^{-1}); 1721 (—C=O of ester), 1206 (C—O of ester). H^1 NMR($CDCl_3$)δppm: 1.25 (t, $J = 4.4$ Hz, —CH_3 of ester), 4.20 (q, $J = 7.6$ Hz, —CH_2 of ester), 2.71 (s, 3H, —CH_3), 3.63–3.67 (m, —CH_2—CH—), 7.75 (s, 1H, C_4-H), 7.52 (d, $J = 7.6$ Hz, C_5-H), 7.44 (d, $J = 6.6$ Hz, C_7-H), 7.34 (t, $J = 7.8$ Hz, C_6-H), M.F: $C_{15}H_{15}O_2NSe$, Mass(m/z) M^+: 320.

6-Methoxy Dihydroselenolo(2,3-b)quinoline-2-carboxylic Ethyl Ester (6d)

Colorless crystals, yield, 19%, m.p. 110–111°C. Calc.: C, 53.58; H, 4.49; N, 4.16. Found: C, 53.40; H, 4.42; N, 4.10. IR(KBr, cm^{-1}): 1700 (—C=O of ester), 1170 (—C—O of ester), H^1NMR($CDCl_3$)δppm: 1.43 (t, $J = 7.2$ Hz, —CH_3 of ester), 4.42 (q, $J = 7.2$ Hz, —CH_2 of ester), 3.96 (s, 3H, —OCH_3), 3.81–3.86 (m, —CH_2—CH—), 8.51 (s, 1H, C_4-H), 8.25 (s, 1H, C_5-H), 7.46 (d, $J = 9.4$ Hz, C_8-H), 8.02 (d, 1H, $J = 9.4$ Hz, C_7-H), M. F: $C_{15}H_{15}O_3NSe$, Mass(m/z)M^+, 336.

8-Methoxy-dihydroselenolo(2,3-b)quinoline-2-carboxylic Ethyl Ester (6e)

Colorless crystals, yield, 17%, m.p. 59–60°C. Calc.: C, 53.58; H, 4.49; N, 4.16. Found: C, 53.50; H, 4.48; N, 4.16. IR(KBr, cm^{-1}): 1730 (—C=O of ester), 1175 (—C—O of ester), H^1NMR($CDCl_3$)δppm: 1.46 (t, $J = 7.2$ Hz, —CH_3 of ester), 4.44 (q, $J = 7.2$ Hz,CH_2 of ester), 3.98 (s, 3H, —OCH_3), 3.89–3.95 (m, —CH_2—CH—), 8.51 (s, 1H, C_4-H), 7.32–8.07 (m, 3H, C_5, C_6, C_7-H), M. F: $C_{15}H_{15}O_3NSe$, Mass(m/z)M^+, 336.

Dihydroselenolo(2,3-b)benzo(h)quinoline-2-carboxylic Ethyl Ester (6f)

Colorless crystals, yield, 17%, m.p. 195–196°C. Calc.: C, 60.68; H, 4.24; N, 3.93; Found: C, 60.65; H, 4.20; N, 3.90. IR(KBr, cm^{-1}): 1720 (C=O of ester), 1172 (—C—O of ester). H^1NMR($CDCl_3$)δppm: 1.46 (t, $J = 7.2$ Hz, —CH_3 of ester), 4.44 (q, $J = 7.2$ Hz, CH_2 of ester), 3.92–3.97 (m, —CH_2—CH—), 7.45–9.43 (m, 7H, C_4, C_5, C_6, C_7, C_8, C_9, C_{10}-H), M. F: $C_{18}H_{15}O_2NSe$, Mass(m/z)M^+, 356.

6, 8-Methyl-dihydroselenolo(2,3-b)quinoline-2-carboxylic Ethyl Ester (6g)

Colorless crystals,yield, 23%, m.p. 85–86°C. Calc.: C, 57.49; H, 5.13; N, 4.19. Found: C, 57.49; H, 5.12; N, 4.16. IR(KBr, cm^{-1}): 1725 (C=O of ester), 1180 (C—O of ester), H^1NMR($CDCl_3$)δppm: 1.42 (t, $J = 7.2$ Hz, —CH_3 of ester), 4.34 (q, $J = 7.2$ Hz, —CH_2 of ester), 2.41, 2.53 (s_{each}, 3H, —CH_3), 3.79–3.85 (m, —CH_2—CH—) 8.11 (s, 1H, C_4-H), 7.52 (s, 1H, C_7-H), M. F: $C_{16}H_{17}O_2NSe$, Mass(m/z)M^+, 334

2-Selenoxo-1,2-dihydro-3-carbethoxy Ethyl Quinoline (7a)

Colorless crystals, yield, 65%, m.p. 135–136°C. Calc.: C, 54.55; H, 4.90; N, 4.54. Found: C, 54.50; H, 4.89; N, 4.50. IR(KBr, cm^{-1}): 1732(C=O of ester), 3414 (—NH), 1259 (C=Se), H^1NMR($CDCl_3$) δ ppm: 1.23 (t, $J = 6.2$ Hz, —CH_3 of ester), 4.13 (q, $J = 7.4$ Hz, OCH_2 of ester), 2.78 (t, $J = 7.2$ Hz,—CH_2—$C\underline{H}_2$—COO^-), 3.01 (t, $J = 7.2$ Hz, CH_2—CH_2—COO^-), 7.69 (s, 1H, C_4—H), 7.23–7.54 (m, 4H, C_5, C_6, C_7, C_8-H), M. F: $C_{14}H_{15}O_2NSe$, Mass(m/z)M^+, 308.

7-Methyl- 2-selenoxo-1,2-dihydro-3-carbethoxy Ethyl Quinoline (7b)

Colorless crystals, yield, 55%, m.p. 101–102°C. Calc.: C, 55.90; H, 5.32; N, 4.35. Found: C, 55.89; H, 5.32; N, 4.30. IR(KBr, cm^{-1}): 1732 (C=O of ester), 3443 (NH), 1230 (C=Se), H^1 NMR($CDCl_3$) δppm: 1.21 (t, $J = 7.2$ Hz, —CH_3 of ester), 4.11 (q, $J = 7.4$ Hz, OCH_2 of ester), 2.56 (s, 3H, —CH_3), 2.73 (t, $J = 8.4$ Hz,—$C\underline{H}_2$—CH_2—COO^-), 3.03 (t, $J = 7.2$ Hz, CH_2— $C\underline{H}_2$—COO^-), 7.81 (s, 1H, C_4-H), 7.72 (s, C_8-H), 7.61 (d, $J = 8.2$ Hz, C_5-H), 7.29 (d, $J = 7.4$ Hz, C_6-H), M. F: $C_{15}H_{17}O_2NSe$, Mass(m/z)M^+, 322.

8-Methyl-2-selenoxo-1,2-dihydro-3-carbethoxy Ethyl Quinoline (7c)

Colorless crystals, yield, 55%, m.p. 140–142°C. Calc.: C, 55.90; H, 5.32; N, 4.35, Found: C, 55.89; H, 5.32; N, 4.30. IR(KBr, cm^{-1}): 1722 (—C=O of ester), 3417 (—NH), 1252 (C=Se), H^1 NMR($CDCl_3$) δppm:

1.26 (t, $J = 7.8$ Hz, $-CH_3$ of ester), 4.17 (q, $J = 7.8$ Hz, $-OCH_2$ of ester), 2.51 (s, 3H,$-CH_3$), 2.89 (t, $J = 7.8$ Hz,$-C\underline{H}_2-CH_2-COO^-$), 3.34 (t, $J = 6.6$ Hz, $CH_2-C\underline{H}_2-COO^-$), 7.76 (s,1H, C_4-H), 7.24–7.54 (m,3H, C_5,C_6,C_7-H), M. F: $-C_{15}H_{17}O_2NSe$, Mass (m/z) M^+, 322.

6-Methoxy-2-selenoxo-1,2-dihydro-3-carbethoxy Ethyl Quinoline (7d)

Colorless crystals, yield, 57%, m.p. 146–148°C. Calc.: C, 53.26; H, 5.07; N, 4.14. Found: C, 53.20; H, 5.00; N, 4.10. IR(KBr, cm^{-1}): 1707 ($-C=O$ of ester), 3416($-NH$), 1228 (C=Se), H^1 NMR($CDCl_3$) δppm: 1.21 (t, $J = 7.2$ Hz, $-CH_3$ of ester), 4.11 (q, $J = 7.4$ Hz, $-OCH_2$ of ester), 3.91 (s, 3H,$-CH_3$), 2.72 (t, $J = 7.2$ Hz,$-C\underline{H}_2-CH_2-COO^-$), 3.02 (t, $J =$ 7.2 Hz, $CH_2-C\underline{H}_2-COO^-$), 7.01–7.95 (m, 4H, C_4, C_5, C_6, C_7-H), M. F: $C_{15}H_{17}O_3NSe$, Mass(m/z)M^+, 338.

8-Methoxy-2-selenoxo-1,2-dihydro-3-carbethoxy Ethyl Quinoline (7e)

Colorless crystals, yield, 50%, m.p. 105–107°C. Calc.: C, 53.26; H, 5.07; N, 4.14. Found: C, 53.20; H, 5.00; N, 4.10. IR(KBr, cm^{-1}): 1730 (C=O of ester), 3415 ($-NH$), 1292 (C=Se), H^1NMR($CDCl_3$) δppm: 1.23 (t, $J = 7.2$ Hz, $-CH_3$ of ester), 4.17 (q, $J = 7.8$ Hz, OCH_2 of ester), 4.05 (s, 3H,$-OCH_3$), 2.77 (t, $J = 8.2$ Hz,$-C\underline{H}_2-CH_2-COO^-$), 3.11(t, $J = 7.4$ Hz, $CH_2-C\underline{H}_2-COO^-$), 7.71 (s, 1H, C_4-H), 6.98–7.35 (m, 3H, C_5, C_6, C_7-H), M. F: $C_{15}H_{17}O_3NSe$, Mass(m/z)M^+: 338.

2-Selenoxo-1,2-dihydro-3-carbethoxy Ethyl-benzo(h)quinoline (7f)

Colorless crystals, yield, 60%, m.p. 235–236°C. Calc.: C, 60.34; H, 4.78; N, 3.90. Found: C, 60.30; H, 4.78; N, 3.89. IR(KBr, cm^{-1}): 1730 (C=O of ester), 3410 ($-NH$), 1230 (C=Se), H^1NMR($CDCl_3$) δppm: 1.36 (t, $J = 7.8$ Hz, $-CH_3$ of ester), 4.23 (q, $J = 7.8$ Hz, $-OCH_2$ of ester), 2.92 (t, $J = 7.8$ Hz,$-C\underline{H}_2-CH_2-COO^-$), 3.44 (t, $J = 6.6$ Hz, $CH_2-C\underline{H}_2-COO^-$), 7.45–9.13(m, 7H, C_4, C_5, C_6, C_7, C_8, C_9, C_{10}-H), M. F: $C_{18}H_{17}O_2NSe$, Mass(m/z)M^+, 358.

6, 8-Dimethyl-2-selenoxo-1, 2-dihydro-3-carbethoxy Ethyl Quinoline (7g)

Colorless crystals, yield, 60%, m.p. 245–246°C. Calc.: C, 57.14; H, 5.69; N, 4.17. Found: C, 57.11; H, 5.67; N, 4.16. IR(KBr, cm^{-1}): 1735 (C=O of ester), 3420 ($-NH$), 1245 (C=Se), H^1 NMR($CDCl_3$) δppm: 1.16 (t, $J = 7.6$ Hz, $-CH_3$ of ester), 4.05 (q, $J = 7.6$ Hz, $-OCH_2$ of ester), 2.35, 2.41 (s_{each}, 3H, $-CH_3$), 2.89 (t, $J = 7.6$ Hz, $-C\underline{H}_2-CH_2-COO^-$), 3.34 (t, $J = 6.4$ Hz, $CH_2-C\underline{H}_2-COO^-$), 7.86 (s, 1H, C_4-H), 8.01

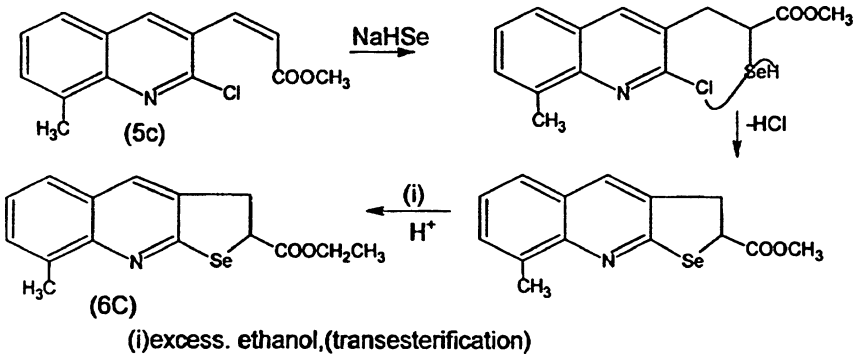


FIGURE 1

(s, 1H, C_7-H), 8.17 (s, 1H, C_5-H). M. F: $C_{16}H_{19}O_2NSe$, Mass(m/z)M^+, 336.

RESULTS AND DISCUSSION

Raja and Shanmugam[23] synthesized the novel diselenolo(3,4-b)quinolines by reacting the vinyl quinolines with NaHSe prepared in situ. The present work was started by reacting 8-methyl-3-(2-chloro-3-quinolyl)acrylic methyl ester (**5c**) with NaHSe. The products obtained were not the expected methyl esters, but the unexpected ethyl esters which was an interesting observation. The conversion from (**5c**) to (**6c**) was found to be due to reduction, cyclisation and transesterification in the presence of excess ethanol and the eliminated acid within the reaction medium (Figure 1).

The conversion from (**5c**) to (**7c**) (Figure 2) was found to be due to reduction and transesterification in the presence of excess ethanol and the eliminated acid within the reaction medium The above reaction sequence was further extended to synthesize other derivatives **6a**, **6b**, **6d**, **6e**, **6f**, **6g**, **7a**, **7b**, **7d**, **7e**, **7f**, and **7g**, respectively.

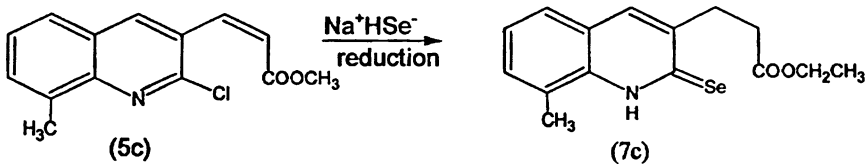


FIGURE 2

TABLE I Antibacterial Activity

	Diameter of inhibition zone in mm											
	Escherichia coli			*Salmonella typhi*			*Staphylococcus aureus*			*Pseudomonas flourosence*		
Compound	2%	1%	0.5%	2%	1%	0.5%	2%	1%	0.5%	2%	1%	0.5%
6a	10	16	11	—	14	10	9	14	10	14	10	—
6c	13	11	11	11	10	—	11	10	—	—	—	—
6d	14	12	10	15	11	10	14	11	—	14	14	12
7c	—	—	—	—	—	—	—	—	—	—	—	—

Dihydroselenolo(2,3-b)-quinoline-2-carboxylic ethyl ester (**6a**) showed intermediate activity against the test organism *Escherichia coli* at 1% $CHCl_3$. 8-methyl-dihydroselenolo (2,3-b) quinoline-2-carboxylic ethyl ester (**6c**) and 8-methyl-2-selenoxo-1,2-dihydro-3-carbethoxy ethyl quinoline (**7c**) were resistant to all the pathogenic bacteria. 6-methoxy dihydroselenolo (2,3-b)quinoline-2-carboxylic ethyl ester (**6d**) showed intermediate activity against the test organism *Salmonella typhi* at 2% $CHCl_3$ (Table I).

The overall results showed that the derivatives did not reach the effectiveness of the conventional bactericides Norfloxacin and Amoxycillin.

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