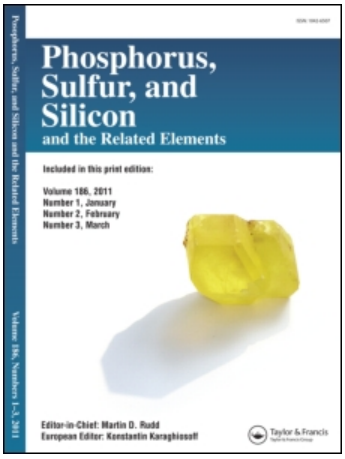

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Synthesis of New Seleno-Substituted Quinolines

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Synthesis of New Seleno-Substituted Quinolines

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The design and synthesis of organoselenium compounds with biological activity currently constitute engaging fundamental problems in applied chemistry in both pharmaceutical and academic laboratories. In this communication we would like to report the synthesis of new organo selenium compounds under mild reaction conditions. The newly synthesized compounds were characterized by elemental analysis, IR, ^1H NMR, and mass spectral studies.

Keywords 3-formyl-2-chloroquinolines; 3-formyl-2-selenoquinolines; substituted imines; substituted azetidins

INTRODUCTION

The quinoline ring has been the subject of continued interest as several derivatives exhibit a wide range of biological activities including antitumor,[1] hypoglycemic,[2] antihistamine,[3] and anticarcinogenic[4] properties, etc. They have been used to synthesize various fused heterocyclic ring systems, which also show a wide range of pharmacological activities.[5–8] In addition, it is well known that a number of heterocyclic

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compounds containing nitrogen and sulfur exhibit a wide variety of biological activities.[9–12]

Sulfur and selenium are considered to be isosteric as defined by Langmuir[13] and Erlenmeyer.[14] A comparison of the physical properties of group VI B elements in the periodic table indicates that sulfur and selenium resemble one another more than sulfur and oxygen. Although a good case can be made for the substitution of selenium for oxygen or sulfur in chemotherapeutically active compounds, the toxic nature of most selenium compounds can be a serious obstacle. In general, the compound in which the selenium atom is a part of an actual or potential functional group capable of reacting with sulfhydryl groups, e.g., —SeH, —SeCN, —Se—Se, and —Se—SO_3H, tends to be considerably more toxic than their sulfur isosteres. On the other hand, when a selenium atom is not readily accessible as in a stable ring system such as selenophene, the toxicity of sulfur and selenium analogs do not differ widely. Moreover, no toxic incidents have been reported involving an organoselenium compound.[15] In some instances, cyclic selenium compounds have been reported to be less toxic than comparable selenium compounds and comparable sulfur compounds.[16] Although sulfur and selenium are considered to be isosteric, reports about selenium-containing heterocyclics are relatively scares.[17–19] However, the medicinal applications of isosterism have been reviewed by Klayman and Gunther.[20]

Our earlier report on the synthesis of various 2-mercapto-3-formylquinolines showed that such species exhibit prominent antimicrobial activity when compared to the standard drug.[21] This intensified our interest to investigate various 2-seleno-3-formylquinolines.

The key intermediate 3-formyl-2-chloroquinolines **1a–d** were prepared from a reported method.[22] Quinolines **1a–d** subsequently converted into 2-seleno-3-formylquinolines **2a–d**, by the action of sodium hydrogen selenide in a quantitative yield. Since the formyl group present in quinoline is exceptionally susceptible to reduction with sodium borohydride in ethanol. To keep the formyl unreactive, sodium hydrogen selenide (NaHSe) was prepared separately. This was achived by the action of sodium borohydride on selenium powder suspended in water, and then it was added to 3-formyl-2-chloroquinolines **1a–d** taken in ethanol.

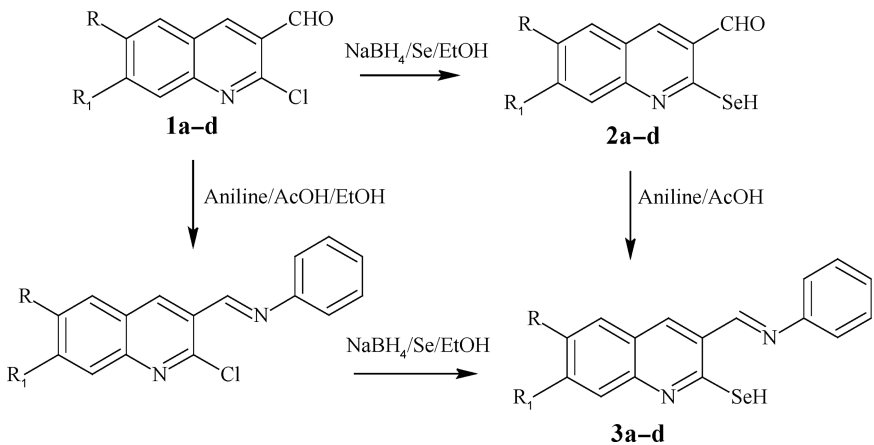
RESULTS AND DISCUSSION

The structures of the resulting new compounds 3-formyl-2-selenoquinolines **2a–d** were characterized by spectral studies. The ^{1}H NMR spectrum of compound **2a** exhibited a broad singlet at 5.88 δ attributed to the —SeH proton (D_2O exchangeable); a multiplet

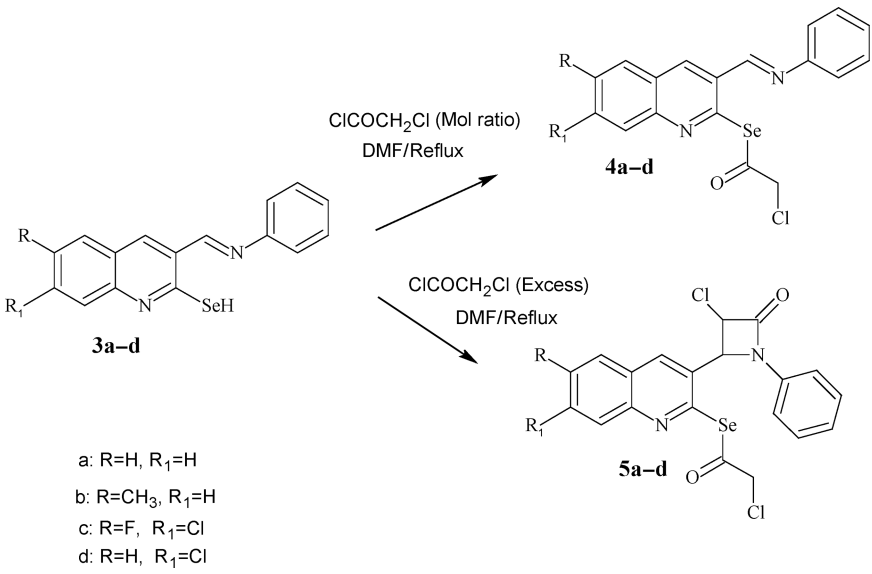
appeared in the region at 7.40–8.15 δ due to five aromatic protons. Another singlet due to the aldehydic proton appeared in the region at 10.3 δ, and the IR spectrum of **2a** showed an absorption band at 1645 cm^{-1}due to the aldehydic carbonyl group. Further, the structure of **2a** was assigned by its mass spectral characteristics. Molecular ion peak at m/z 236 confirmed the structure. Similarly, the spectral details for all other compounds are given in the Experimental section.

In order to study the synthetic utility of 3-formyl-2-selenoquinolines **2a–d**, we have synthesised novel Schiff bases, 3-[(arylimino)methyl]quinoline-2-selenols **3a–d,** by the reaction of 3-formyl-2-selenoquinolines **2a–d** with aniline in glacial acetic acid. An alternative method also was utilized (Scheme 1) to obtain the same compounds. The structure of **3a** was characterized by elemental analysis and spectral data. In the ^1H NMR spectra of **3a**, the signal at 10.3 δ corresponding to the —CHO was found to be absent, and instead a new singlet appeared at 10.24 δ attributed to the imine —CH=N—. A multiplet appeared at 7.40–8.92 δ integrated to ten aromatic protons. The IR spectrum of **3a** showed an absorption band at 1592 cm^{-1} due to —CH=N—, and no peak corresponding to the aldehydic group confirmed the structure. The molecular ion peak detected was at m/z 311.

The previously discussed synthesis impelled us to expand the chemistry of 3-[(arylimino)methyl]quinoline-2-selenols **3a–d**. Furthermore, compounds **3a–d** on refluxing with a stoichiometric and nonstoichiometric amount of chloroacetyl chloride in DMF afforded the corresponding 3-{[(arylimino)methyl]quinolin-2-yl} chloroethaneselenates **4a–d**



SCHEME 1 Synthesis of 2-selenoquinoline-3-formaldehydes **(2a–d)** and 3-[(phenylimino)methyl]quinoline-2-selenols **(3a–d)**.



SCHEME 2 {3-[(Phenylimino)methyl]quinolin-2-yl}chloroethaneselenoates **(4a–d)** and [3-(3-chloro-4-oxo-1-phenylazetidin-2-yl)quinolin-2-yl] chloroethan eselenates **(5a–d)**.

and [3-(3-chloro-4-oxo-1-arylazetidin-2-yl)quinolin-2-yl] chloroethaneselenates **5a–d,** respectively. The spectral details for all these compounds were satisfactory for the assigned structure and are given in the Experimental section.

EXPERIMENTAL

IR spectra were recorded on a Perkin Elmer 157 infrared spectrophotometer. ^{1}H NMR spectra (300 MHz) were recorded on a Bruker Supercon FT NMR instrument using TMS as the internal standard and mass spectra on a Jeol JMS-D 300 mass spectrometer operating at 70 eV. Melting points were determined in open capillaries and are uncorrected. The purity of the compounds was checked by TLC, and further purification was achived by column chromatography.

Preparation of Selenoquinolines: 2-Selenoquinoline-3-formaldehyde (2a)

To selenium powder (1 g, 0.013 mol) in water (25 mL) at ice-bath temperature was added sodium borohydride (1 g, 0.026 mol) in small portions,

with continuous stirring. Considerable foaming (liberation of hydrogen) occurred immediately. After the addition of the sodium borohydride was complete, water (25 mL) was added. The reaction mixture was stirred for 15 min. The virtual colorless solution (or slightly deep reddish color) of sodium hydrogen selenide (NaHSe) resulted and was ready for use without further treatment.

To the solution of sodium hydrogen selenide (freshly prepared from selenium [1 g, 0.013 mol] and sodium borohydride [1 g, 0.026 mol] in water [50 mL]) was added to 3-formyl-2-chloroquinoline **1a** (1.89 g, 0.01 mol) in ethanol (80 mL). The reaction mixture was refluxed for 1 h, cooled, and poured into ice water. After it acidified with diluted hydrochloric acid, the resultant solid 3-formyl-2-selenoquinoline **2a** (80%) that was obtained was filtered, washed with water, dried, and recrystallized from ethyl acetate. Compounds **2b–d** were synthesized similarly (70–78%).

2-Selenoquinoline-3-formaldehyde (2a)

Yield (1.88 g, 80%); m.p. 168–169°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 5.88 (1H, s, SeH), 7.40–8.15 (5H, m, Ar—H), 10.33 (1H, s, CHO); IR (KBr) ν (cm^{-1}): 1645; [M+]: 236. Calcd. (%) for $C_{10}H_7NOSe$: C, 50.87; H, 2.99; N, 5.93; Se, 33.44. Found: C, 50.75; H, 2.86; N, 5.81; Se, 33.27.

2-Seleno-6-methylquinoline-3-formaldehyde (2b)

Yield (1.95 g, 78%); m.p. 153–154°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 2.55 (3H, s, CH_3), 5.85 (1H, s, SeH), 7.39–8.24 (4H, m, Ar—H), 10.31 (1H, s, CHO); IR (KBr) ν (cm^{-1}): 1643. [M+]: 250. Calcd. (%) for $C_{11}H_9NOSe$: C, 52.81; H, 3.63; N, 5.60; Se, 31.56. Found: C, 52.76; H, 3.54; N, 5.49; Se, 31.42.

7-Chloro-6-fluoro-2-selenoquinoline-3-formaldehyde (2c)

Yield (1.93 g, 67%); m.p. 175–178°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 5.87 (1H, s, SeH), 7.23–8.32 (3H, m, Ar—H), 10.32 (1H, s, CHO); IR (KBr) ν (cm^{-1}): 1642. [M+]: 288. Calcd. (%) for $C_{10}H_5ClFNOSe$: C, 41.62; H, 1.75; N, 4.85; Se, 27.36. Found: C, 41.58; H, 1.64; N, 4.73; Se, 27.25.

7-Chloro-2-selenoquinoline-3-formaldehyde (2d)

Yield (1.97 g, 73%); m.p. 159–160°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 5.89 (1H, s, SeH), 7.23–8.45 (4H, m, Ar—H), 10.31 (1H, s, CHO);

IR (KBr) ν (cm^{-1}): 1641. [M+]: 270. Calcd. (%) for $C_{10}H_6ClNOSe$: C, 44.39; H, 2.24; N, 5.18; Se, 29.18. Found: C, 44.28; H, 2.18; N, 5.11; Se, 29.12.

Preparation of Imines: 3-[(Phenylimino)methyl]quinoline-2-selenol (3a)

Method A

2-selenoquinoline-3-formaldehyde **2a** (2.36 g, 0.01 mol) and aniline (0.93 g, 0.01 mol) were stirred in glacial acetic acid (30 mL) by using a magnetic stirrer for 2 h. The completion of reaction was monitored by TLC, whereupon the reaction mixture was poured into ice-cold water, stirred well, filtered, dried, and purified by column chromatography to give **3a** (2.42 g, 78%). Compounds **3b–d** were synthesized similarly (68–75%).

Method B

To a mixture of *N*-[(2-chloroquinolin-3-yl)methylene]-*N*-phenylamine (2.67 g 0.01 mol) and selenium powder (1 g, 0.013 mol) in ethanol was added sodium borohydride (1g, 0.026 mol) in water (50 mL). The reaction mixture was refluxed for 1–2 h, cooled, poured into crushed ice, and acidified with diluted hydrochloric acid. The resultant solid 3-[(phenylimino)methyl]quinoline-2-selenol **3a** was filtered, washed with water, dried, and recrystallized from ethyl acetate. The yield obtained by this method was similar to the previous method. The authenticity of the compounds was confirmed by overlapping spectras and by mixed melting points.

3-[(Phenylimino)methyl]quinoline-2-selenol (3a)

Yield (2.42 g, 78%); m.p. 213–215°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 5.86 (1H, s, SeH), 7.40–8.92 (10H, m, Ar—H), 10.22 (1H, s, —CH=N); IR (KBr) ν (cm^{-1}): 1592. [M+]: 311. Calcd. (%) for $C_{16}H_{12}N_2Se$: C, 61.74; H, 3.89; N, 9.00; Se, 25.37. Found: C, 61.62; H, 3.76; N, 9.08; Se, 25.24.

6-Methyl-3-[(phenylimino)methyl]quinoline-2-selenol (3b)

Yield (2.27 g, 70%); m.p. 195–196°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 2.56 (1H, s, CH_3), 5.85 (1H, s, SeH), 7.46–8.96 (9H, m, Ar—H), 10.23 (1H, s, —CH=N); IR (KBr) ν (cm^{-1}): 1598. [M+]: 325. Calcd. (%)

for $C_{17}H_{14}N_2Se$: C, 62.77; H, 4.34; N, 8.61; Se, 24.28. Found: C, 62.64; H, 4.26; N, 8.53; Se, 24.26.

7-Chloro-6-fluoro-3-[(phenylimino)methyl]quinoline-2-selenol (3c)

Yield (2.40 g, 66%); m.p. 213–214°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 5.85 (1H, s, SeH), 7.48–8.96 (8H, m, Ar—H), 10.20 (1H, s, —CH=N); IR (KBr) ν (cm^{-1}): 1595. [M+]: 363. Calcd. (%) for $C_{16}H_{10}ClFN_2Se$: C, 52.84; H, 2.77; N, 7.70; Se, 21.71. Found: C, 52.72; H, 2.64; N, 7.59; Se, 21.59.

7-Chloro-3-[(phenylimino)methyl]quinoline-2-selenol (3d)

Yield (2.48 g, 72%); m.p. 227–228°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 5.88 (1H, s, SeH), 7.48–8.96 (9H, m, Ar—H), 10.23 (1H, s, —CH=N); IR (KBr) ν (cm^{-1}): 1600. [M+]: 345. Calcd. (%) for $C_{16}H_{11}ClN_2Se$: C, 55.59; H, 3.21; N, 8.10; Se, 22.84. Found: C, 55.42; H, 3.19; N, 8.16; Se, 22.69.

Preparation of {3-[(Phenylimino)methyl]quinolin-2-yl} chloroethaneselenoate (4a)

A mixture of 3-[(phenylimino)methyl]quinoline-2-selenol **3a** (3.110 g, 0.01 mol) and chloroacetyl chloride (1.693 g, 0.015 mol) containing DMF (20 mL) was heated on a water bath for 2–3 h. Completion of the reaction was monitored by TLC; the reaction mixture was poured into crushed ice, and stirred well, and the solid obtained was filtered, dried, and purified by column chromatography to provide **4a** (65%). The same procedure was adopted for the synthesis of **4b–d,** and the yields were found to be 60–72%.

{3-[(Phenylimino)methyl]quinolin-2-yl}chloroethaneselenoate (4a)

Yield (2.52 g, 65%); m.p. 203–205°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 4.45 (2H, s, —CH_2—) 7.52–8.92 (10H, m, Ar—H), 10.24 (1H, s, —CH=N); IR (KBr) ν (cm^{-1}): 1603, 1665; [M+]: 387. Calcd. (%) for $C_{18}H_{13}ClN_2OSe$: C, 55.81; H, 3.35; N, 7.23; Se, 20.40. Found: C, 55.92; H, 3.24; N, 7.15; Se, 20.32.

{6-Methyl-3-[(phenylimino)methyl]quinolin-2-yl} chloroethaneselenoate (4b)

Yield (2.89 g, 72%); m.p. 209–211°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 2.56 (3H, s, CH_3), 4.43 (2H, s, —CH_2—) 7.54–8.96 (9H, m, Ar—H), 10.24 (1H, s, —CH=N); IR (KBr) ν (cm^{-1}): 1600, 1667; [M+], 401. Calcd. (%) for $C_{19}H_{15}ClN_2OSe$: C, 56.75; H, 3.73; N, 6.96; Se, 19.65. Found: C, 56.63; H, 3.62; N, 6.83; Se, 19.51.

{7-Chloro-6-fluoro-3-[(phenylimino)methyl]quinolin-2-yl} Chloroethaneselenoate (4c)

Yield (2.72 g, 63%); m.p. 174–178°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 4.44 (2H, s, —CH_2—) 7.50–8.94 (8H, m, Ar—H), 10.24 (1H, s, —CH=N); IR (KBr) ν (cm^{-1}): 1600, 1664; [M+], 440. Calcd. (%) for $C_{18}H_{11}Cl_2FN_2OSe$: C, 49.09; H, 2.50; N, 6.36; Se, 17.94. Found: C, 49.18; H, 2.42; N, 6.23; Se, 17.82.

{7-Chloro-3-[(phenylimino)methyl]quinolin-2-yl} Chloroethaneselenoate (4d)

Yield (2.78 g, 66%); mp. 186–187°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 4.44 (2H, s, —CH_2—) 7.50–8.94 (9H, m, Ar—H), 10.24 (1H, s, —CH=N); IR (KBr) ν (cm^{-1}): 1605, 1665; [M+], 422. Calcd. (%) for $C_{18}H_6ClNOSe$: C, 51.16; H, 2.84; N, 6.63; Se, 18.70. Found: C, 51.25; H, 2.72; N, 6.52; Se, 18.59.

Preparation of azetidines: [3-(3-Chloro-4-oxo-1-phenylazetidin-2-yl)quinolin-2-yl] Chloroethaneselenate (5a)

3-[(Phenylimino)methyl]quinoline-2-selenol **3a** (3.11 g, 0.01 mol) and chloroacetyl chloride in excess (5 mL) were dissolved in DMF (20 mL). The resultant mixture was stirred for 30 min and then warmed on a water bath for 6 h. The reaction mixture was cooled and poured into crushed ice, and the precipitate obtained was filtered, dried, and purified by column chromatography to afford [3-(3-chloro-4-oxo-1-phenylazetidin-2-yl)quinolin-2-yl] chloroethaneselenate **5a** (3.62 g, 68%).

[3-(3-Chloro-4-oxo-1-phenylazetidin-2-yl)quinolin-2-yl] Chloroethaneselenate (5a)

Solid, yield (3.156 g, 68%); m.p. 175–176°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 4.44 (2H, s, —CH_2—), 7.52–8.95 (11H, m, Ar—H), IR (KBr)

ν (cm^{-1}): 1746. [M+]: 464. Calcd. (%) for $C_{20}H_{14}Cl_2N_2O_2Se$: C, 51.70; H, 3.01; N, 6.03; Se, 17.00. Found: C, 51.59; H, 3.08; N, 6.09; Se, 17.05.

[3-(3-Chloro-4-oxo-1-phenylazetidin-2-yl)-6-methylquinolin-2-yl] Chloroethane Selenate (5b)

Yield (3.15 g, 66%); m.p. 193–195°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 2.54 (3H, s, CH_3), 4.43 (2H, s, —CH_2—), 7.54–8.98 (10H, m, Ar—H); IR (KBr) ν (cm^{-1}): 1747. [M+]: 478. Calcd. (%) for $C_{21}H_{16}Cl_2N_2O_2Se$: C, 52.69; H, 3.34; N, 5.84; Se, 16.51. Found: C, 52.58; H, 3.21; N, 5.73; Se, 16.40.

[3-(3-Chloro-4-oxo-1-phenylazetidin-2-yl)-6-chloro-7-fluoroquinolin-2-yl] Chloroethane Selenate (5c)

Yield (3.09 g, 60%); m.p. 179–180°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 4.42 (2H, s, —CH_2—), 7.52–8.94 (9H, m, Ar—H); IR (KBr) ν (cm^{-1}): 1748. [M+], 516. Calcd. (%) for $C_{20}H_{12}Cl_3FN_2O_2Se$: C, 46.45; H, 2.32; N, 5.41; Se, 15.28. Found: C, 46.32; H, 2.21; N, 5.29; Se, 15.19.

[3-(3-Chloro-4-oxo-1-phenylazetidin-2-yl)-6-chloroquinolin-2-yl] Chloroethane Selenate (5d)

Yield (3.14 g, 63%); m.p. 197–199°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 4.42 (2H, s, —CH_2—), 7.52–8.94 (10H, m, Ar—H); IR (KBr) ν (cm^{-1}): 1746. [M+]: 498. Calcd. (%) for $C_{20}H_{13}Cl_3N_2O_2Se$: C, 48.13; H, 2.60; N, 5.61; Se, 15.83. Found: C, 48.26; H, 2.49; N, 5.50; Se, 15.72.

CONCLUSION

In summary, a novel, general, and efficient method for the synthesis of a variety of new seleno quinoline derivatives was developed. By demonstrating the broad application of this chemistry, this methodology may be very useful in organic synthesis.

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