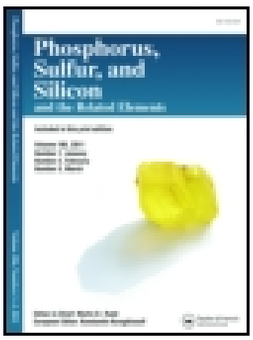

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To cite this article: Amir Rashidi , Mohammad H. Mosslemin & Alireza Hassanabadi (2020): Synthesis of 2-aryl-7-methyl-4-selenoxo-4H-pyrano[3,4-e][1,3]oxazin-5-one via a three-component condensation, Phosphorus, Sulfur, and Silicon and the Related Elements, DOI: 10.1080/10426507.2020.1858082

To link to this article: https://doi.org/10.1080/10426507.2020.1858082

PHOSPHORUS, SULFUR, AND SILICON AND THE RELATED ELEMENTS
https://doi.org/10.1080/10426507.2020.1858082

Synthesis of 2-aryl-7-methyl-4-selenoxo-4H-pyrano[3,4-e][1,3]oxazin-5-one via a three-component condensation

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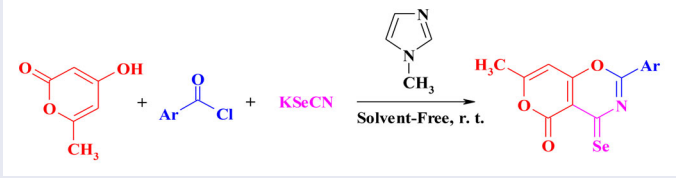
ABSTRACT

The reaction between 4-hydroxy-6-methyl-2H-pyran-2-one and aroyl chlorides with potassium selenocyanate in the presence of catalytic amounts of *N*-methylimidazole under solvent-free conditions provided a simple and efficient one-pot route for the synthesis of 2-aryl-7-methyl-4-selenoxo-4H-pyrano[3,4-e][1,3]oxazin-5-one. The reaction is characterized by mild conditions, short reaction time easy work-up, high yields of biological active products, and does not involve any hazardous solvent and tolerance to various functional groups.

ARTICLE HISTORY
Received 28 September 2020
Accepted 26 November 2020

KEYWORDS
4-Hydroxy-6-methyl-2H-pyran-2-one; aroyl chlorides; potassium selenocyanate; *N*-methylimidazole; pyrano[3,4-e][1,3]oxazines

GRAPHICAL ABSTRACT



Introduction

Multi-component reactions have been receiving great attention from chemists because they can be widely employed for the rapid assembly of arrays with high molecular diversity.[1–4] Derivatives of 1,3-oxazine play a significant role the theoretical organic chemistry[5] and biologically active compounds.[6] They show interesting interactions and can be used as chemotherapy agents.[7] Dihydro-1,3-oxazine skeletons can be formed in proteins as they are converted into polypeptides The thio derivatives of pyrano-1,3-benzoxazine have also shown anti-inflammatory and antipyretic properties.[8,9] Synthesis of 1,3-oxazine derivatives has already been reported[10–19] and this synthesis used a procedure first described by Khalilzadeh et al.[20] in which they synthesized a series of arylated bicyclic and tricyclic thioheterocycles *via* the *N*-methylimidazole-catalyzed reaction of phenol and 1- and 2-naphthols with a series of aroyl chlorides and ammonium thiocyanate. Although many useful and reliable methods for preparation of 1,3-oxazines have been reported in the literature,[10–19] many of these procedures have significant drawbacks such as low yields of the products, long reaction times, and harsh reaction conditions. Thus, new routes for the synthesis of these molecules have attracted considerable attention in search for a rapid entry to these heterocycles. The current interest in selenium-containing organic compounds stems from their remarkable synthetic and biological functions.[21–25] The first synthesis of acyl isoselenocyanates, which were generated by the reaction of acyl chlorides and potassium selenocyanate, has been described by Douglas.[26] Recently, a few synthesis of heterocycles from acyl isoselenocyanates were reported.[27–29] Herein, we describe a synthesis of 2-aryl-7-methyl-4-selenoxo-4H-pyrano[3,4-e][1,3]oxazin-5-one using a reported method.[27]

Results and discussion

Using reaction conditions similar to those previously described,[27] one-pot, three-component reaction between 4-hydroxy-6-methyl-2H-pyran-2-one **1** and aroyl chlorides **2** with potassium selenocyanate **3** in the presence of catalytic amounts of *N*-methylimidazole under solvent-free conditions afford the 2-aryl-7-methyl-4-selenoxo-4H-pyrano[3,4-e][1,3]oxazin-5-one **4** in nearly quantitative yields (Scheme 1).

To study the scope of the reaction, a series of aroyl chlorides were applied. The results are shown in Table 1. In all cases, aroyl chloride substituted with either electron-donating or electron-withdrawing groups underwent the reaction smoothly and gave products in excellent yields.

To find out the optimum quantity of *N*-methylimidazole, the reaction of potassium selenocyanate and 4-nitro benzoylchlorides with 4-hydroxy-6-methyl-2H-pyran-2-one was carried out under solvent-free conditions using different quantities of *N*-

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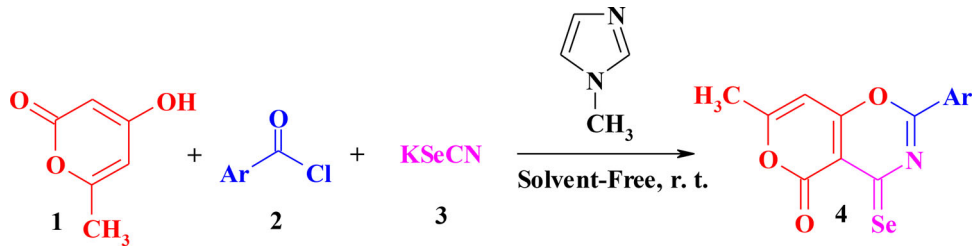
Supplemental data for this article is available online at https://doi.org/10.1080/10426507.2020.1858082.



Scheme 1. Three-component condensation of 4-hydroxy-6-methyl-2H-pyran-2-one, aroyl chlorides and potassium selenocyanate.

Table 1. Yields of a series of 2-aryl-7-methyl-4-selenoxo-4H-pyrano[3,4-e][1,3]oxazin-5-one (4) prepared from 4-hydroxy-6-methyl-2H-pyran-2-one (1), aroyl chlorides (2) and potassium selenocyanate (3) (Scheme 1).

Entry	Ar	Product	Yield (%)[a]	m.p. (°C)
1	4-$NO_2C_6H_4$	**4a**	91	125–127
2	4-ClC_6H_4	**4b**	83	154–156
3	4-BrC_6H_4	**4c**	81	138–140
4	3-$NO_2C_6H_4$	**4d**	92	113–115
5	C_6H_5	**4e**	76	96–98
6	4-$CH_3C_6H_4$	**4f**	73	101–103

[a]Yields refer to the pure isolated products.

Table 2. Optimization amount of *N*-methylimidazole on the reaction of condensation of potassium selenocyanate and aroyl chlorides with 4-hydroxy-6-methyl-2H-pyran-2-one.

Entry	Catalyst (mol%)	Time (min)	Yield (%)[a]
1	3	90	45
2	5	70	60
3	7	60	70
4	10	40	91
5	12	40	91

[a]Isolated yield.

methylimidazole (Table 2). *N*-methylimidazole of 10% gave an excellent yield in 40 min (Table 2, entry 4).

The structures of compounds **4a–f** were deduced from their high-field ^{1}H NMR, ^{13}C NMR, IR spectral data, elemental analysis, and mass spectra. The mass spectra of compounds **4a–f** are fairly similar and display molecular ion peaks. For example, the mass spectrum of compound **4a** showing a molecular ion peak at *m/z* 363 confirmed that compound **4a** is a condensation product of 4-hydroxy-6-methyl-2H-pyran-2-one, 4-nitrobenzoyl chloride, and potassium selenocyanate. The ^{1}H NMR spectrum of **4a** exhibited two sharp singlets readily recognized as arising from methyl ($\delta = 2.01$ ppm), and methine ($\delta = 5.82$ ppm) protons, along with multiplets ($\delta = 7.97$–8.08 ppm) for aromatic protons. The ^{13}C NMR spectrum of compound **4a** shows 12 distinct signals consistent with the proposed structure. The C = Se, C = O, and C = N groups resonance in ^{13}C NMR spectra of **4a** appears at 193.8, 163.1, and 161.7 ppm respectively. The selenone ^{13}C resonances of compounds **4** were observed at 192.5–164.6 ppm.[27,30]

The ^{77}Se NMR of compound **4a** consisted of a singlet at $\delta = 745$ ppm. The IR spectrum of compound **4a** also supported the suggested structure exhibiting strong absorption bands at 1711 cm^{-1} for the C = O and 1280 cm^{-1} for the C = Se groups. The mechanism of the reaction is probably similar to that published by Yavari et al.[27] A tentative mechanism for this transformation is proposed in Scheme 2.

In conclusion, here we reported a facile and efficient one-pot synthesis of 2-aryl-7-methyl-4-selenoxo-4H-pyrano[3,4-e][1,3]oxazin-5-one by one-pot condensation reaction of between 4-hydroxy-6-methyl-2H-pyran-2-one and aroyl chlorides with potassium selenocyanate in the presence of catalytic amounts of *N*-methylimidazole under solvent-free conditions. The present procedure has the advantages such as mild reaction conditions, short reaction time, easy work-up, high yields of biological active products.

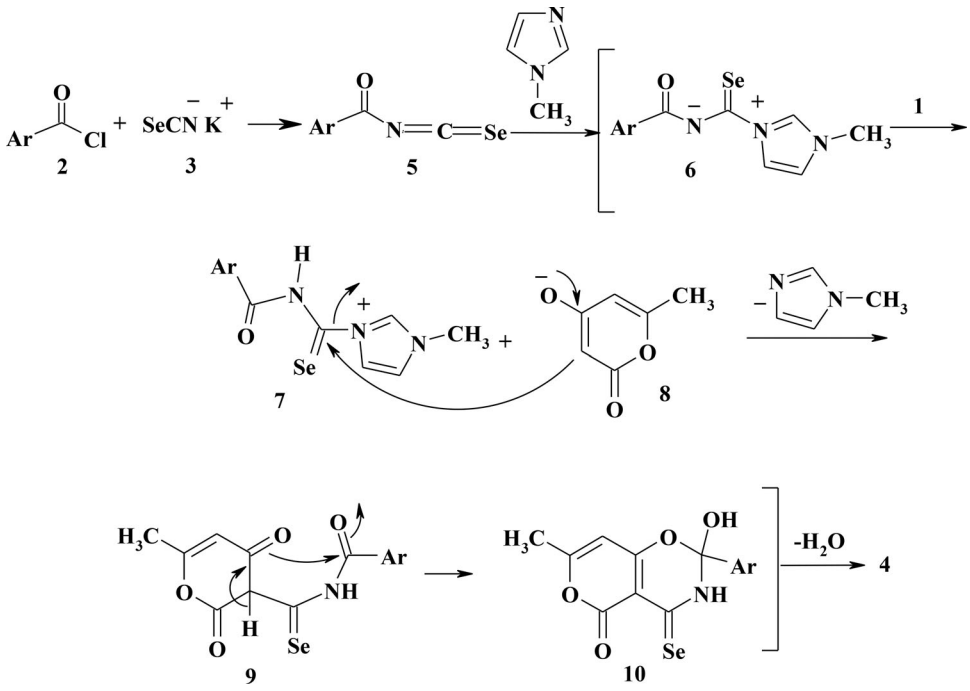
Experimental

Melting points were determined with an Electrothermal 9100 apparatus. Elemental analysis were performed using a Heraeus CHN-O-Rapid analyzer. Mass spectra were recorded on a FINNIGAN-MAT 8430 mass spectrometer operating at an ionization potential of 70 eV. IR spectra of selected compounds were recorded on a Shimadzu IR-470 spectrometer in KBr disks. NMR spectra were obtained on a Bruker DRX-400 Avance spectrometer (^{1}H NMR at 400 MHz, ^{13}C NMR at 100 MHz, and ^{77}Se NMR at 76.2 MHz) in DMSO-d_6 using TMS as internal standard. The chemicals used in this work were purchased from Fluka (Buchs, Switzerland) and were used without further purification.

Caution! All workers must be thoroughly trained on the use of selenium-containing compounds, and they must wear appropriate personal safety gear. All of the reactions involving selenium-containing compounds must be carried out in a well-ventilated hood. The Supporting Information contains sample ^{1}H, ^{13}C, and ^{77}Se NMR spectra of the products **4a–f** (Supporting Information Figures S1–S18).

General procedure

To potassium selenocyanate (1 mmol) in a 10 mL flask at r.t. was added Aroyl chloride (1 mmol) via syringe. The reaction mixture was stirred in a water bath at about 90 °C for 10 min. Then, 4-hydroxy-6-methyl-2H-pyran-2-one (1 mmol) was added at this temperature. The reaction mixture was allowed to cool to room temperature. Finally, *N*-methylimidazole (0.082 g; 1 mmol) was added via syringe. The resulting mixture was stirred at r.t. for 40 min. The progress of the reaction was monitored by TLC. After completion of the reaction, the residue was separated by silica gel (Merck 230–240 mesh) column chromatography using hexane–ethyl acetate mixture (3:1) as eluent.



Scheme 2. Proposed mechanism for **4**.

2-(4-Nitrophenyl)-7-methyl-4-selenoxo-4H-pyrano[3,4-e][1,3]oxazin-5-one (4a)

Brown powder; m.p. 125–127 °C; IR (KBr) (ν_{max} cm^{-1}): 1711, 1668, 1552, 1344, 1280, 1087; ^{1}H NMR: δ 2.01 (3H, s, CH_3), 5.82 (1H, s, =CH), 7.97 (2H, d, $^3J_{HH}$ = 8 Hz, 2CH of $C_6H_4NO_2$), 8.08 (2H, d, $^3J_{HH}$ = 8 Hz, 2CH of $C_6H_4NO_2$); ^{13}C NMR: δ 19.3 (CH_3), 88.0, 100.3, 123.1, 128.7, 130.2, 132.6, 135.3, 148.7, 161.7 (C = N), 163.1 (C = O), and 193.8 (C = Se); ^{77}Se NMR: δ = 745; MS *m/z* (%): 363 (12); Anal. Calcd for $C_{14}H_8N_2O_5Se$: C, 46.30; H, 2.22; N, 7.71%. Found: C, 46.37; H, 2.30; N, 7.82.

2-(4-Chlorophenyl)-7-methyl-4-selenoxo-4H-pyrano[3,4-e][1,3]oxazin-5-one (4 b)

Brown powder; m.p. 154–156 °C; IR (KBr) (ν_{max} cm^{-1}): 1719, 1664, 1618, 1255, 1087; ^{1}H NMR: δ 2.10 (3H, s, CH_3), 5.95 (1H, s, =CH), 7.45 (2H, d, $^3J_{HH}$ = 8 Hz, 2CH of C_6H_4Cl), 7.85 (2H, d, $^3J_{HH}$ = 8 Hz, 2CH of C_6H_4Cl); ^{13}C NMR: δ 19.4 (CH_3), 88.0, 100.3, 122.9, 128.2, 129.3, 130.4, 135.5, 145.9, 162.1 (C = N), 164.3 (C = O), and 193.5 (C = Se); ^{77}Se NMR: δ = 733; MS *m/z* (%): 352 (8); Anal. Calcd. for $C_{14}H_8ClNO_3Se$: C, 47.69; H, 2.29; N, 3.97%. Found: C, 47.75; H, 2.37; N, 4.05.

2-(4-Bromophenyl)-7-methyl-4-selenoxo-4H-pyrano[3,4-e][1,3]oxazin-5-one (4c)

Brown powder; m.p. 138–140 °C; IR (KBr) (ν_{max} cm^{-1}): 1718, 1661, 1615, 1250, 1053; ^{1}H NMR: δ 2.31(3H, s, CH_3), 5.68 (1H, s, =CH), 7.05 (2H, d, $^3J_{HH}$ = 8 Hz, 2CH of C_6H_4Br), 7.41 (2H, d, $^3J_{HH}$ = 8 Hz, 2CH of C_6H_4Br); ^{13}C NMR: δ 19.3 (CH_3), 88.1, 99.7, 123.1, 128.3, 129.3, 130.1, 132.6, 141.6, 161.8 (C = N), 165.3 (C = O), and 193.9 (C = Se); ^{77}Se NMR: δ = 730; MS *m/z* (%): 397 (6); Anal. Calcd. for $C_{14}H_8BrNO_3Se$: C, 42.35; H, 2.03; N, 3.53%. Found: C, 42.41; H, 2.10; N, 3.62.

2-(3-Nitrophenyl)-7-methyl-4-selenoxo-4H-pyrano[3,4-e][1,3]oxazin-5-one (4d)

Brown powder; m.p. 113–115 °C; IR (KBr) (ν_{max} cm^{-1}): 1716, 1669, 1564, 1347, 1285, 1090; ^{1}H NMR: δ 2.43 (3H, s, CH_3), 5.62 (1H, s, =CH), 7.14–8.30 (4H, m, 4CH of $C_6H_4NO_2$); ^{13}C NMR: δ 19.3 (CH_3), 87.9, 100.8, 122.8, 129.8, 130.4, 130.8, 132.6, 135.7, 136.5, 149.1, 161.4 (C = N), 163.7 (C = O), and 194.6 (C = Se); ^{77}Se NMR: δ = 743; MS *m/z* (%): 363 (9); Anal. Calcd for $C_{14}H_8N_2O_5Se$: C, 46.30; H, 2.22; N, 7.71%. Found: C, 46.40; H, 2.32; N, 7.80.

7-Methyl-2-Phenyl-4-selenoxo-4H-pyrano[3,4-e][1,3] oxazin-5-one (4e)

Brown powder; m.p. 96–98 °C; IR (KBr) (ν_{max} cm^{-1}): 1716, 1658, 1627, 1257, 1041; ^{1}H NMR: δ 2.24 (3H, s, CH_3), 5.95 (1H, s, =CH), 7.47–7.77 (5H, m, 5CH of C_6H_5); ^{13}C NMR: δ 19.9 (CH_3), 89.8, 101.5, 127.4, 127.8, 128.6, 129.2, 132.3, 133.8, 161.8 (C = N), 165.7 (C = O), and 194.0 (C = Se); ^{77}Se NMR: δ = 705; MS (*m/z*, %): 318 (5); Anal. Calcd. for $C_{14}H_9NO_3Se$: C, 52.85; H, 2.85; N, 4.40%. Found: C, 52.95; H, 2.93; N, 4.46.

7-Methyl-4-selenoxo-2-p-tolyl-4H-pyrano[3,4-e][1,3] oxazin-5-one (4f)

Brown powder; m.p. 101–103 °C; IR (KBr) (ν_{max} cm^{-1}): 17,013, 1670, 1612, 1267, 1080; ^{1}H NMR: δ 2.06 (3H, s, CH_3), 2.45 (3H, s, CH_3), 5.61 (1H, s, =CH), 7.06 (2H, d,

$^{3}J_{HH}$ = 8 Hz, 2CH of $C_6H_4CH_3$), 7.45 (2H, d, $^{3}J_{HH}$ = 8 Hz, 2CH of $C_6H_4CH_3$); ^{13}C NMR: δ 19.2 (CH_3), 21.9 (CH_3), 89.2, 101.5, 126.9, 129.1, 129.5, 134.3, 135.5, 144.4, 161.3 (C = N), 163.8 (C = O), and 192.5 (C = Se); ^{77}Se NMR: δ = 713; MS (*m/z*, %): 332 (6); Anal. Calcd. for $C_{15}H_{11}NO_3Se$: C, 54.23; H, 3.34; N, 4.22%. Found: C, 54.30; H, 3.41; N, 4.31.

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