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Base-Promoted Carbonylative Cyclization of Propargylic Amines with Selenium under CO gas-free Conditions

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Abstract. We report here a new carbonylative procedure for the cyclization of propargylic amines with elemental selenium (Se). By using *t*BuOK as the promoter, various 1,3-selenazolidin-2-ones were produced without the usage of toxic CO gas. Benzene-1,3,5-triyl triformate (TFBen) was employed as a safe and convenient CO surrogate here, and a broad class of substrates (29 examples) were effectively transformed into the desired products in 50-97% yields under mild conditions.

Keywords: Carbonylation; Cyclization; Domino reactions; Catalyst free; Heterocycle synthesis

Introduction

The element selenium, an essential micronutrient, plays a vital role in human health.[1] Although selenium is toxic at high levels[1-2] and could lead to cancer in certain forms,[3] selenium-containing compounds are important intermediates in synthetic chemistry,[4] and are present widely in biological and medicinal chemistry.[4b,5] Among them, 1,3-selenazolidines are interesting and have been reported as inhibitors of NO-synthase,[6] antimutagenes[7] and cancer preventing agents.[5m,8] As is shown in Figure 1, benzoselenazolinones was found to have anti-inflammatory properties,[4b] and 2-oxoselenazolidine-4-carboxylic acid (2-OxoSCA) has chemopreventive activity.[5k,5m]

Due to their obvious importance, synthetic methodologies have been developed for the preparation of 1,3-selenazolidin-2-ones.[9] In 1984, Woodgate and co-workers reported the use of *trans*-1-iodo-2-isocyanatocyclohexane as the starting material with NaHSe or Li_2Se to give the corresponding (*3aS,7aR*)-hexahydrobenzo[*d*][1,3]selenazol-2(3*H*)-one in 55% and 42% yields, respectively.[10] In 2001, Robert's group prepared 2-oxoselenazolidine-4(*R*)-carboxylic acid from chloroamino acid, Se and sodium borohydride in about 36% overall yield through three steps procedure.[8a] In 2002, Fujiwara et al. reported a method for the synthesis of 5-alkylideneselenazolin-2-ones from 3-aminoalkynes (9 examples, up to 95% yield) under carbon monoxide gas atmospheric (1 bar) in the presence of 1,8-diazabicyclo(5.4.0)undec-7-ene (DBU).[9a] In 2013, Lu's group developed a facile access to 1,3-selenazolidin-2-ones (4 examples, up to 79% yield) by the reaction of 2-chloroethyl amine salts with Se and carbon monoxide (1 bar) in the presence of NaOH.[11] However, the disadvantages of these protocols are obvious as well: 1) the use of toxic and flammable CO gas; 2) the limitation of substrates scope.

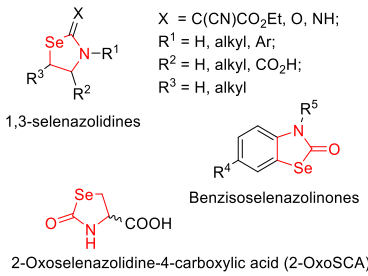


Figure 1. Selected examples of bio-active 1,3-selenazolidines and 1,3-selenazolidin-2-ones motifs.

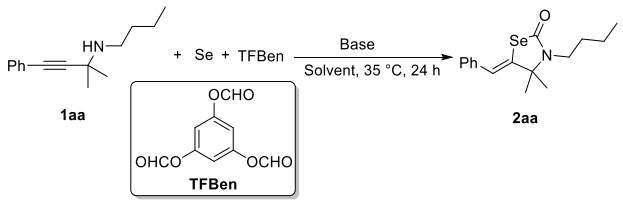
In order to avoid the manipulation of toxic CO gas in carbonylative reactions, a number of alternative CO sources such as transition metal carbonyl complexes,[12] formaldehyde,[13] methanol,[14] formic acid,[15] formates,[16] formamides,[17] *N*-formylsaccharin,[18] oxalyl chloride,[19] carbamoylsilane,[20] chloroform[21] and others[22] have been developed recently. Among them, TFBen, developed by our group,[23] is a solid, stable, safe and convenient CO source. Herein, we wish to report our new results on *t*BuOK-promoted carbonylative cyclization of propargylic amines with Se using TFBen (benzene-1,3,5-triyl triformate) as the CO

surrogate. The desired 1,3-selenazolidin-2-ones (29 examples) were isolated in high yields (50-97%).

Results and Discussion

Initially, we carried out the carbonylative cyclization of propargylic amine **1aa** with TFBen and Se at 35 °C and the results are summarized in Table 1. When **1aa** was treated with TFBen (0.4 mmol, 2 equiv.) and selenium powder (0.3 mmol, 1.5 equiv.) using *t*BuOK (0.4 mmol, 2.0 equiv.) as the promoter in 1.0 mL DMSO, the reaction afforded the corresponding cyclic product **2aa** in 99% yield (Table 1, entry 1). Then, a series of bases were screened (Table 1, entries 2-6). It shows that the reaction using DBU gave a slightly reduced yield (97%) of **2aa** (Table 1, entry 2). In the cases using NaOH, DABCO, DAMP or K_2CO_3 as the base, the reaction gave remarkably low yields of the product (Table 1, entries 3-6). When the solvent of the reaction was changed from DMSO to DMF (Table 1, entry 8), over 99% yield was obtained, while 1,4-dioxane, MeCN, toluene and THF led to the formation of only a trace or no product (Table 1, entries 7 and 9-11). Excellent yields can still be obtained when the amounts of TFBen and *t*BuOK were decreased (Table 1, entries 12 and 13), and 88% of isolated yield was obtained (Table 1, entry 13). Unfortunately, further reducing the amount of tBuOK to 0.04 mmol (0.2 equiv.), no product was observed (Table 1, entry 14). It was found that the yield was dramatically decreased to 78% when 0.1 mmol (0.5 equiv.) TFBen was employed (Table 1, entry 15).

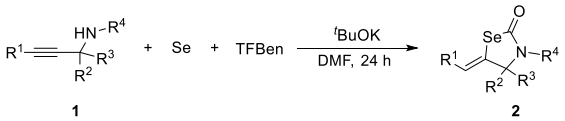
Table 1. Screening of optimal reaction conditions.[a]

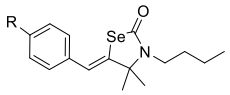


Entry	TFBen	Base (0.4 mmol)	Solvent	Yield[b]
1	0.4 mmol	^{t}BuOK	DMSO	99%
2	0.4 mmol	DBU	DMSO	97%
3	0.4 mmol	NaOH	DMSO	2%
4	0.4 mmol	DABCO	DMSO	11%
5	0.4 mmol	DMAP	DMSO	Trace
6	0.4 mmol	K_2CO_3	DMSO	Trace
7	0.4 mmol	^{t}BuOK	dioxane	0
8	0.4 mmol	^{t}BuOK	DMF	>99%
9	0.4 mmol	^{t}BuOK	MeCN	Trace
10	0.4 mmol	^{t}BuOK	Toluene	0
11	0.4 mmol	^{t}BuOK	THF	0
12	0.2 mmol	^{t}BuOK[c]	DMF	>99%
13	0.2 mmol	^{t}BuOK[d]	DMF	98% (88%)[e]
14	0.2 mmol	^{t}BuOK[f]	DMF	0
15	0.1 mmol	^{t}BuOK[d]	DMF	78%

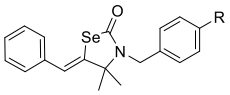
[a] Reaction conditions: **1aa** (0.2 mmol), Se (0.3 mmol, 1.5 equiv.), solvent (1.0 mL). [b] The yield of **2aa** was determined by GC analysis using *n*-dodecane as internal standard. [c] 0.2 mmol. [d] 0.1 mmol. [e] Isolated yield. [f] 0.04 mmol.

Table 2. 1,3-Selenazolidin-2-ones synthesis.[a]



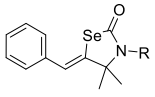


2aa R = H 88% yield
2ab R = Me 90% yield
2ac R = OMe 85% yield
2ad R = ^{n}Pr 94% yield
2ae R = ^{t}Bu 96% yield

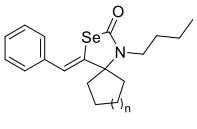


2af R = F 88% yield
2ag R = Cl 81% yield
2ah R = Br 76% yield

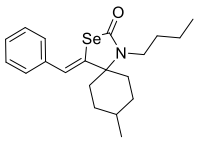
2ba R = H 60% yield[bc]
2bb R = OMe 67% yield[bc]
2bc R = Cl 82% yield[bc]



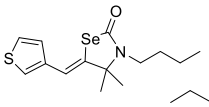
2c R = ^{n}Oct 95% yield
2d R = Cy trace



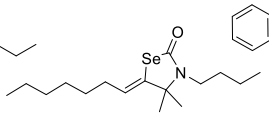
2e n = 1 62% yield[b]
2f n = 2 84% yield
2g n = 3 59% yield



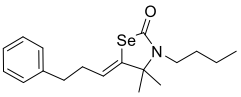
2h 92% yield



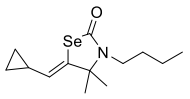
2i 95 % yield



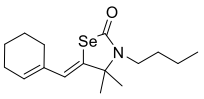
2j 68% yield[b]



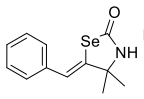
2k 56% yield[b]



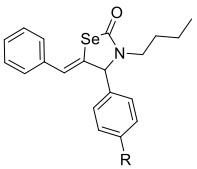
2l 78% yield[b]



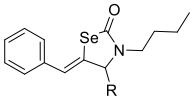
2m 78% yield[b]



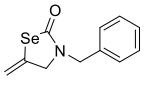
2n 86% yield



2o R = H 50% yield[b]
2p R = OMe 58% yield[b]
2q R = F 64% yield[b]

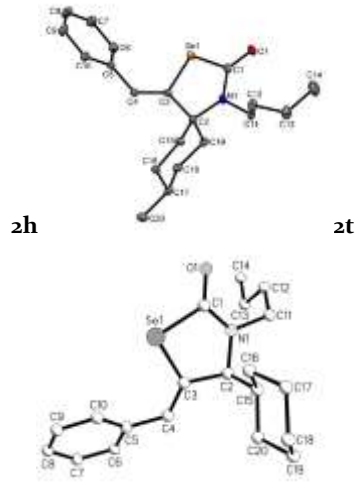


2r R = Et 86% yield
2s R = CH_2CH_2Ph 96% yield
2t R = Cy 97% yield



2u 85% yield

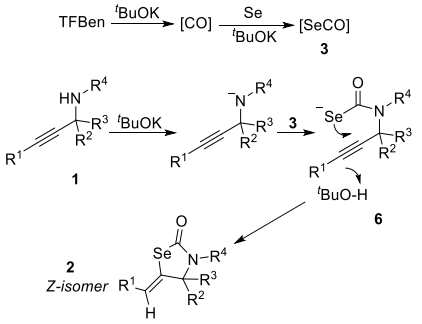
[a] Reaction conditions: substrate (0.2 mmol), Se (0.3 mmol, 1.5 equiv), TFBen (0.2 mmol, 1.0 equiv.), ^{t}BuOK (0.1 mmol, 0.5 equiv.), DMF (1.0 mL), 35 °C, 24 h. [b] 60 °C. [c] TFBen (0.4 mmol, 2.0 equiv.), ^{t}BuOK (0.2 mmol, 1.0 equiv.).



2h **2t**

Figure 2. X-ray structure of **2h** (CCDC 1817355) and **2t** (CCDC 1817678). Displacement ellipsoids correspond to 30% probability. Hydrogen atoms are omitted for clarity.[24]

With the optimized reaction conditions (Table 1, entry 13) in hand, we examined the scope with various propargylic amines as follows (Table 2). It shows that good to excellent yields (76-96%) were achieved for both electron-rich (**2ab-2ae**) and electron-deficient (**2af-2ag**) aromatic propargylic amines. The reactions of substrates containing *n*-octyl group on the nitrogen atom afforded the corresponding product (**2c**) in 95% yield. However, when the propargylic amine with an *N*-cyclohexyl unit was carried out, a trace product **2d** was observed, which might due to the steric bulk on the nitrogen atom. High yields of the desired products (**2ba-2bc**) can be obtained by increasing the reaction temperature (60 °C) and also the loading of *t*BuOK (0.2 mmol, 1.0 equiv.) and TFBen (0.4 mmol, 2.0 equiv.). Compounds having a carbocycle on the triple bond were converted to the desired products (**2e-2h**) in good to excellent yields (59-92%). Notably, thiophene substituted propargylic amine was also transformed to the product **2i** in 95% yield. For substrates containing alkyl groups on the triple bond were conducted at 60 °C, 56-78% yields of products (**2j-2m**) were obtained. Interestingly, primary propargylic amine can also be successfully transformed into the desired product **2n** in 86% yield. Moreover, substrates with aryl and alkyl substituents (R^2 = aryl, alkyl and R^3 = H) were tested as well, 50-97% yields of the corresponding products **2o-2t** can be successfully achieved. In addition, it was found that a compound without substituents (R^2, R^3 = H) can also undergo carbonylative cyclization to provide the product **2u** in a good yield. In order to confirm the structure, X-ray analysis of products **2h** and **2t** have been performed as well (**Figure 2**).



Scheme 1. Possible reaction pathway.

Based on the literature[9a,11] and our results, a possible reaction pathway for this carbonylative cyclization of propargylic amines with Se and TFBen is shown in **Scheme 1**. By using *t*BuOK as the promotor, Se reacts with CO which generated in-situ from TFBen, to give the selenium carbonyl compound **3**. Deprotonation of propargylic amine **1** by *t*BuOK followed by the nucleophilic addition to the selenium carbonyl **3** generates the selenocarbamate **6** as the key intermediate. Then, the intramolecular nucleophilic attack of selenium anion to the triple bond forms the desired product **2** as a *Z*-isomer. It is likely that the steric hindrance of *t*BuOH plays an important role in the observed stereochemistry of product **2**.

Conclusion

In summary, a new and straightforward carbonylation procedure toward 1,3-selenazolidin-2-ones has been established. By using *t*BuOK as the promotor, propargylic amines and elemental selenium were carbonylatively cyclized with TFBen as the CO source. Various desired 1,3-selenazolidin-2-ones with broad functional groups are prepared in 50-97% yields under mild conditions. Further synthetic application of this method and more detailed study of the reaction mechanism are under progress.

Experimental Section

Unless otherwise noted, all reactions were carried out under a nitrogen atmosphere. All reagents were obtained from commercial sources and used as received without further purification. Column chromatography was performed on silica gel (200-300 mesh) using petroleum ether (bp 60-90 °C), ethyl acetate and dichloromethane as eluent. ^{1}H and ^{13}C NMR spectra were taken on 400 MHz instruments, and spectral data were reported in ppm relative to tetramethylsilane (TMS) as internal standard and $CDCl_3$ as solvent.

Preparation of TFBen

Formic acid (8.4 mL, 222.8 mmol, 5.0 equiv.) was added to acetic anhydride (16.8 mL, 178.2 mmol, 4.0 equiv.) at room temperature. The mixture was stirred at 60 °C for 1 h and cooled to room temperature. The resulting solution was poured into a flask containing 1,3,5-trihydroxybenzene (5.62 g, 44.6 mmol, 1.0 equiv.) and AcONa (1.83 g, 22.3 mmol, 0.5 equiv.). The mixture was

stirred for 4 h in a water bath and then diluted with toluene (100 mL), washed with H_2O (50 mL) two times. Keep the organic phase in fridge (2-8 °C) for overnight. Then filtered and dried in vacuo to afford the desired product benzene-1,3,5-triyl triformate (TFBen) as a white solid.

Benzene-1,3,5-triyl triformate, TFBen

5.1 g, 55% yield, white solid, mp 53.2–55.6 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.24 (s, 3H), 6.97 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 158.06, 150.30, 112.62.

Procedure for the Synthesis of Propargylic Amines for 1aa-ah, 1ba-bc, 1c-1m, 1o-1t[25]

In a typical experiment, CuI (1.5 mmol, 30 mol%) was added to a 15 mL tube equipped with a magnetic stirrer which was then placed under vacuum and refilled with nitrogen three times. Amine (5.0 mmol, 1.0 equiv.), aldehyde/ketone (5.0 mmol, 1.0 equiv.) and alkyne (5.0 mmol, 1.0 equiv.) were added, then the tube was sealed and the mixture was stirred at 75 °C for 16 h. After the reaction was completed, the crude reaction mixture was concentrated and purified by silica gel column chromatography (PE/EtOAc=5/1) to provide the desired propargylic amine 1.

***N*-Butyl-2-methyl-4-phenylbut-3-yn-2-amine** (1aa)

Black oil, 70%. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.39 (dd, *J* = 6.5, 3.1 Hz, 2H), 7.32 – 7.22 (m, 3H), 2.79 (t, *J* = 7.2 Hz, 2H), 1.69 (s, 1H), 1.52 (dd, *J* = 14.9, 7.1 Hz, 2H), 1.45 (s, 6H), 1.39 (dt, J = 14.2, 7.1 Hz, 2H), 0.94 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 131.51, 128.11, 127.67, 123.41, 94.50, 81.91, 50.25, 43.98, 32.53, 29.52, 20.51, 13.93.

***N*-Butyl-2-methyl-4-(*p*-tolyl)but-3-yn-2-amine** (1ab)

Black oil, 63%. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.28 (d, *J* = 8.0 Hz, 2H), 7.07 (d, *J* = 7.9 Hz, 2H), 2.78 (t, *J* = 7.2 Hz, 2H), 2.31 (s, 3H), 1.60 (s, 1H), 1.54 – 1.47 (m, 2H), 1.44 (d, *J* = 14.3 Hz, 6H), 1.38 (d, *J* = 6.9 Hz, 2H), 0.94 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 137.56, 131.35, 128.80, 120.35, 93.71, 81.93, 50.22, 43.92, 32.53, 29.54, 21.24, 20.46, 13.87.

***N*-Butyl-4-(4-methoxyphenyl)-2-methylbut-3-yn-2-amine** (1ac)

Black oil, 65%. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.33 (d, *J* = 8.8 Hz, 2H), 6.81 (d, *J* = 8.8 Hz, 2H), 3.78 (s, 3H), 2.78 (t, *J* = 7.2 Hz, 2H), 1.68 (s, 1H), 1.53 – 1.49 (m, 2H), 1.44 (s, 6H), 1.39 (dd, *J* = 15.1, 7.3 Hz, 2H), 0.94 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 159.15, 132.86, 115.59, 113.75, 92.96, 81.68, 55.15, 50.29, 43.97, 32.55, 29.61, 20.51, 13.91. HRMS (ESI): [M+H$^+$] calcd. for $C_{16}H_{24}NO^+$, 246.1852; found, 246.1855.

***N*-Butyl-2-methyl-4-(4-propylphenyl)but-3-yn-2-amine** (1ad)

Black oil, 50%. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.31 (d, *J* = 8.1 Hz, 2H), 7.09 (d, *J* = 8.0 Hz, 2H), 2.79 (t, *J* = 7.2 Hz, 2H), 2.56 (t, *J* = 7.6 Hz, 2H), 1.66 – 1.59 (m, 4H), 1.55 – 1.47 (m, 4H), 1.4 (s, 6H), 1.40 (dd, *J* = 15.1, 7.1 Hz, 2H), 0.96 – 0.90 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 142.48, 131.41, 128.30, 120.63, 93.81, 82.04, 50.33, 44.03, 37.83, 32.60, 29.62, 24.29, 20.54, 13.94, 13.63. [M+H$^+$] calcd. for $C_{18}H_{28}N^+$, 258.2216; found, 258.2218.

***N*-Butyl-4-(4-(*tert*-butyl)phenyl)-2-methylbut-3-yn-2-amine** (1ae)

Black oil, 66%. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.33 (dd, *J* = 11.1, 4.7 Hz, 4H), 2.79 (t, *J* = 7.2 Hz, 2H), 1.72 (s, 1H), 1.50 (d, J = 7.6 Hz, 2H), 1.44 (s, 6H), 1.40 (d, *J* = 7.8 Hz, 2H), 1.29 (s, 9H), 0.94 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 150.86, 131.23, 125.08, 120.43, 93.77, 81.96, 50.30, 43.98, 34.58, 32.55, 31.10, 29.58, 20.51, 13.92. [M+H$^+$] calcd. for $C_{19}H_{30}N^+$, 272.2373; found, 272.2370.

***N*-Butyl-4-(4-fluorophenyl)-2-methylbut-3-yn-2-amine** (1af)

Black oil, 59%. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.39 – 7.34 (m, 2H), 7.00 – 6.95 (m, 2H), 2.78 (t, *J* = 7.2 Hz, 2H), 1.67 (s, 1H), 1.51 (dd, *J* = 14.9, 7.5 Hz, 2H), 1.44 (s, 6H), 1.40 (dd, *J* = 15.0, 7.2 Hz, 2H), 0.94 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 162.13 (d, *J* = 249.5 Hz), 133.33 (d, *J* = 8.3 Hz), 119.47 (d, *J* = 3.5 Hz), 115.35 (d, *J* = 22.0 Hz), 94.14, 80.89, 50.28, 43.99, 32.52, 29.50, 20.51, 13.93. [M+H$^+$] calcd. for $C_{15}H_{21}FN^+$, 234.1653; found, 234.1658.

***N*-Butyl-4-(4-chlorophenyl)-2-methylbut-3-yn-2-amine** (1ag)

Black oil, 50%. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.31 (d, *J* = 8.5 Hz, 2H), 7.25 (d, *J* = 8.5 Hz, 2H), 2.76 (t, *J* = 7.1 Hz, 2H), 1.57 (s, 1H), 1.53 – 1.47 (m, 2H), 1.43 (s, 6H), 1.42 – 1.34 (m, 2H), 0.94 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 133.59, 132.72, 128.40, 121.92, 95.63, 80.79, 50.20, 43.95, 32.54, 29.45, 20.48, 13.91. [M+H$^+$] calcd. for $C_{15}H_{21}ClN^+$, 250.1357; found, 250.1358.

4-(4-Bromophenyl)-*N*-butyl-2-methylbut-3-yn-2-amine (1ah)

Black oil, 63%. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.41 (d, *J* = 8.3 Hz, 2H), 7.25 (d, *J* = 8.5 Hz, 2H), 2.77 (t, *J* = 7.2 Hz, 2H), 2.34 (s, 1H), 1.55 – 1.49 (m, 2H), 1.45 (s, 6H), 1.39 (dd, *J* = 14.9, 7.5 Hz, 2H), 0.93 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 132.98, 131.34, 122.33, 121.84, 95.54, 81.08, 50.43, 43.93, 32.37, 29.32, 20.46, 13.88.

***N*-Benzyl-2-methyl-4-phenylbut-3-yn-2-amine** (1ba)

Black oil, 76%. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.46 – 7.42 (m, 2H), 7.38 (d, *J* = 7.2 Hz, 2H), 7.34 – 7.28 (m, 5H), 7.23 (d, *J* = 5.6 Hz, 1H), 3.96 (s, 2H), 1.63 (s, 1H), 1.50 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 140.67, 131.60, 128.43, 128.21, 127.81, 126.92, 123.39, 94.45, 82.36, 50.65, 49.13, 29.64.

***N*-(4-Methoxybenzyl)-2-methyl-4-phenylbut-3-yn-2-amine** (1bb)

Black oil, 50%. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.44 (dd, *J* = 6.5, 3.1 Hz, 2H), 7.30 (dd, *J* = 5.1, 1.9 Hz, 5H), 6.86 (d, *J* = 8.6 Hz, 2H), 3.90 (s, 2H), 3.79 (s, 3H), 1.61 (s, 1H), 1.49 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 158.62, 132.81, 131.61, 129.61, 128.23, 127.81, 123.42, 113.86, 94.54, 82.32, 55.25, 50.60, 48.51, 29.63.

***N*-(4-Chlorobenzyl)-2-methyl-4-phenylbut-3-yn-2-amine** (1bc)

Black oil, 43%. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.42 (dd, *J* = 6.5, 3.2 Hz, 2H), 7.31 – 7.25 (m, 7H), 3.91 (s, 2H), 1.57 (s, 1H), 1.48 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 139.21, 132.51, 131.54, 129.67, 128.42, 128.18, 127.83, 123.24, 94.17, 82.43, 50.57, 48.30, 29.60. [M+H$^+$] calcd. for $C_{18}H_{19}ClN^+$, 284.1201; found, 284.1206.

***N*-(2-Methyl-4-phenylbut-3-yn-2-yl)octan-1-amine (1c)**

Yellow oil, 71%. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.43 – 7.36 (m, 2H), 7.31 – 7.25 (m, 3H), 2.78 (t, *J* = 7.2 Hz, 2H), 1.82 (s, 1H), 1.56 – 1.48 (m, 2H), 1.45 (s, 6H), 1.32 (ddd, *J* = 18.5, 10.1, 4.7 Hz, 10H), 0.88 (dd, *J* = 9.1, 4.6 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 131.54, 128.12, 127.70, 123.42, 94.48, 81.98, 50.31, 44.34, 31.79, 30.41, 29.52, 29.45, 29.21, 27.41, 22.60, 14.04. [M+H$^+$] calcd. for $C_{19}H_{30}N^+$, 272.2373; found, 272.2378.

***N*-(2-Methyl-4-phenylbut-3-yn-2-yl)cyclohexanamine** (1d)

Black oil, 58%. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.38 (dd, *J* = 6.5, 3.2 Hz, 2H), 7.28 (dd, *J* = 4.9, 1.7 Hz, 3H), 2.90 – 2.80 (m, 1H), 2.24 (s, 1H), 2.00 (d, *J* = 11.8 Hz, 2H), 1.74 (d, *J* = 12.8 Hz, 2H), 1.60 (d, *J* = 12.9 Hz, 1H), 1.49 (s, 6H), 1.38 – 1.09 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 131.42, 128.17, 127.73, 123.42, 94.84, 81.63, 53.76, 50.71, 35.90, 30.42, 25.60. [M+H$^+$] calcd. for $C_{17}H_{24}N^+$, 242.1903; found, 242.1906.

***N*-Butyl-1-(phenylethynyl)cyclopentan-1-amine** (1e)

Black oil, 78%. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.41 – 7.39 (m, 2H), 7.30 – 7.26 (m, 3H), 2.78 (t, *J* = 7.1 Hz, 2H), 2.02 (dd, *J* = 13.4, 7.1 Hz, 2H), 1.80 (s, 6H), 1.50 (dt, *J* = 14.1, 7.0 Hz, 2H), 1.39 (dq, *J* = 14.0, 7.0 Hz, 3H), 0.93 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 131.48, 128.10, 127.55, 123.66, 94.66, 82.44, 61.04, 44.95, 40.61, 32.67, 23.72, 20.54, 13.96.

***N*-Butyl-1-(phenylethynyl)cyclohexan-1-amine** (1f)

Yellow oil, 80%. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.45 – 7.38 (m, 2H), 7.32 – 7.21 (m, 3H), 2.80 (t, *J* = 7.1 Hz, 2H), 1.94 (d, *J* = 12.0 Hz, 2H), 1.66 (dd, *J* = 19.4, 9.8 Hz, 5H), 1.56 – 1.33 (m, 7H), 1.30 – 1.16 (m, 1H), 0.94 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 131.44, 128.02, 127.50, 123.61, 93.48, 84.30, 54.87, 42.76, 38.08, 32.64, 25.81, 22.93, 20.46, 13.88.

***N*-Butyl-1-(phenylethynyl)cycloheptan-1-amine** (1g)

Yellow oil, 60%. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.39 (dd, *J* = 16.9, 5.1 Hz, 2H), 7.28 (d, *J* = 5.3 Hz, 3H), 2.78 (t, *J* = 6.5 Hz, 2H), 2.59 – 2.33 (m, 1H), 2.03 – 1.92 (m, 2H), 1.69 (s, 8H), 1.60 – 1.46 (m, 4H), 1.39 (dq, *J* = 14.1, 7.1 Hz, 2H), 0.93 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 131.48, 128.09, 127.57, 123.60, 94.31, 83.60, 58.17, 43.77, 43.46, 40.56, 32.54, 30.33, 28.00, 24.24, 22.61, 20.52, 13.93.

***N*-Butyl-4-methyl-1-(phenylethynyl)cyclohexan-1-amine** (1h)

Yellow oil, 71%. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.46 – 7.39 (m, 2H), 7.34 – 7.25 (m, 3H), 2.82 (t, *J* = 7.2 Hz, 2H), 1.97 (d, *J* = 11.0 Hz, 2H), 1.67 (m, 3H), 1.55 – 1.46 (m, 3H), 1.45 – 1.36 (m, 6H), 0.99 – 0.90 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 131.48, 128.09, 127.58, 123.63, 93.20, 84.69, 55.04, 43.02, 38.14, 32.65, 33.37, 31.84, 22.13, 20.49, 13.92. [M+H$^+$] calcd. for $C_{19}H_{28}N^+$, 270.2216; found, 270.2218.

***N*-Butyl-2-methyl-4-(thiophen-3-yl)but-3-yn-2-amine** (1i)

Yellow oil, 56%. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.35 (dd, *J* = 2.9, 1.0 Hz, 1H), 7.22 (dd, *J* = 4.9, 3.0 Hz, 1H), 7.06 (dd, *J* = 5.0, 1.0 Hz, 1H), 2.76 (t, *J* = 7.0 Hz, 2H), 1.62 (s, 1H), 1.54 – 1.46 (m, 2H), 1.43 (s, 6H), 1.40 – 1.34 (m, 2H), 0.94 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 129.88, 127.69, 124.91, 122.34, 94.03, 76.85, 50.21, 43.90, 32.51, 29.48, 20.43, 13.87. [M+H$^+$] calcd. for $C_{13}H_{20}NS^+$, 222.1311; found, 222.1315.

***N*-Butyl-2-methyldec-3-yn-2-amine** (1j)

Yellow oil, 63%. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 2.67 (t, *J* = 7.0 Hz, 2H), 2.14 (t, *J* = 6.9 Hz, 2H), 1.75 (s, 1H), 1.48 – 1.41 (m, 5H), 1.36 (dd, *J* = 13.9, 6.8 Hz, 5H), 1.30 (s, 6H), 1.27 – 1.26 (m, 2H), 0.92 – 0.84 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 84.99, 81.90, 49.89, 43.89, 32.50, 31.28, 29.80, 28.93, 28.41, 22.51, 20.55, 18.54, 13.96, 13.93. [M+H$^+$] calcd. for $C_{15}H_{30}N^+$, 224.2373; found, 224.2378.

***N*-Butyl-2-methyl-6-phenylhex-3-yn-2-amine** (1k)

Yellow oil, 69%. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.31 – 7.25 (m, 2H), 7.23 – 7.17 (m, 3H), 2.79 (t, *J* = 7.4 Hz, 2H), 2.62 (s, 2H), 2.47 (t, *J* = 7.4 Hz, 2H), 2.02 (s, 1H), 1.49 – 1.40 (m, 2H), 1.39 – 1.33 (m, 2H), 1.31 (s, 6H), 0.92 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 140.73, 128.45, 128.16, 126.10, 85.71, 81.19, 49.92, 43.80, 35.32, 32.36, 29.62, 20.76, 20.49, 13.94. [M+H$^+$] calcd. for $C_{17}H_{26}N^+$, 244.2060; found, 244.2062.

***N*-Butyl-4-cyclopropyl-2-methylbut-3-yn-2-amine** (1l)

Yellow oil, 51%. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 2.59 (s, 2H), 1.88 (s, 1H), 1.39 (d, *J* = 5.7 Hz, 2H), 1.32 – 1.27 (m, 2H), 1.23 (s, 6H), 1.16 – 1.09 (m, 1H), 0.84 (t, *J* = 7.1 Hz, 3H), 0.68 – 0.60 (m, 2H), 0.55 – 0.48 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 85.01, 80.00, 49.76, 43.67, 32.33, 29.64, 20.38, 13.77, 8.11, -0.78. [M+H$^+$] calcd. for $C_{12}H_{22}N^+$, 180.1747; found, 180.1748.

***N*-Butyl-4-(cyclohex-1-en-1-yl)-2-methylbut-3-yn-2-amine** (1m)

Yellow oil, 75%. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.03 (s, 1H), 2.72 (s, 2H), 2.14 – 1.99 (m, 5H), 1.65 – 1.55 (m, 4H), 1.48 (dd, *J* = 14.3, 7.2 Hz, 2H), 1.39 (d, *J* = 13.3 Hz, 8H) 0.93 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 133.60, 120.48, 91.41, 83.72, 50.16, 43.80, 32.37, 29.53, 29.47, 25.43, 22.23, 21.42, 20.43, 13.83. [M+H$^+$] calcd. for $C_{15}H_{26}N^+$, 220.2060; found, 220.2065.

2-Methyl-4-phenylbut-3-yn-2-amine (1n)

Yellow oil, 52%. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.38 (dd, *J* = 6.5, 3.0 Hz, 2H), 7.29 – 7.19 (m, 3H), 2.02 (s, 2H), 1.46 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 131.07, 127.78, 127.39, 122.97, 96.58, 79.67, 45.19, 31.35.

***N*-(1,3-Diphenylprop-2-yn-1-yl)butan-1-amine** (1o)

Yellow oil, 60%. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.58 (d, *J* = 7.4 Hz, 2H), 7.45 (dd, *J* = 6.6, 2.9 Hz, 2H), 7.35 (t, *J* = 7.4 Hz, 2H), 7.31 – 7.24 (m, 4H), 4.78 (s, 1H), 2.84 (dt, *J* = 11.2, 7.3 Hz, 1H), 2.71 (dt, *J* = 11.2, 6.9 Hz, 1H), 1.55 (s, 1H), 1.54 – 1.46 (m, 2H), 1.42 – 1.31 (m, 2H), 0.91 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 140.52, 131.57, 128.36, 128.12, 127.96, 127.55, 127.46, 123.10, 89.52, 85.15, 54.60, 46.93, 32.00, 20.38, 13.89.

***N*-(1-(4-Methoxyphenyl)-3-phenylprop-2-yn-1-yl)butan-1-amine** (1p)

Yellow oil, 68%. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.55 – 7.41 (m, 4H), 7.32 – 7.25 (m, 3H), 6.89 (d, *J* = 8.7 Hz, 2H), 4.75 (s, 1H), 3.78 (s, 3H), 2.83 (dt, *J* = 11.2, 7.2 Hz, 1H), 2.70 (dt, *J* = 11.2, 7.2 Hz, 1H), 1.80 (s, 1H), 1.57 – 1.45 (m, 2H), 1.42 – 1.31 (m, 2H), 0.91 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 159.07, 132.77, 131.59, 128.62, 128.13, 127.94, 123.20, 113.76, 89.80, 85.02, 55.17, 54.01, 46.85, 32.01, 20.41, 13.89.

***N*-(1-(4-Fluorophenyl)-3-phenylprop-2-yn-1-yl)butan-1-amine** (1q)

Yellow oil, 63%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (dd, *J* = 8.2, 5.7 Hz, 2H), 7.45 (dd, *J* = 6.3, 2.8 Hz, 2H), 7.26 (dd, *J* = 4.4, 2.0 Hz, 3H), 7.01 (t, *J* = 8.6 Hz, 2H), 4.75 (s, 1H), 2.81 (dt, *J* = 11.0, 7.2 Hz, 1H), 2.68 (dt, *J* = 11.1, 7.2 Hz, 1H), 1.56 – 1.42 (m, 3H), 1.41 – 1.30 (m, 2H), 0.90 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 162.08 (d, *J* = 246.7 Hz), 136.26 (d, J = 3.1 Hz), 131.49, 129.04 (d, *J* = 8.1 Hz), 128.04 (d, *J* = 9.9 Hz), 122.88, 115.11, 114.90, 89.20, 85.33, 53.76, 46.72, 31.92, 20.29, 13.80. [M+H$^+$] calcd. for $C_{19}H_{21}FN^+$, 282.1653; found, 282.1656.

***N*-Butyl-1,5-diphenylpent-1-yn-3-amine** (1s)

Yellow oil, 80%. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.44 (dd, *J* = 6.7, 2.8 Hz, 2H), 7.33 – 7.21 (m, 7H), 7.18 (dd, *J* = 12.4, 5.4 Hz, 1H), 3.56 (t, *J* = 6.8 Hz, 1H), 2.97 – 2.76 (m, 3H), 2.63 (dd, *J* = 15.7, 9.0 Hz, 1H), 2.10 – 1.92 (m, 2H), 1.56 – 1.42 (m, 3H), 1.37 (dq, *J* = 13.9, 7.1 Hz, 2H), 0.92 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 141.62, 131.57, 128.45, 128.28, 128.15, 127.82, 125.79, 123.35, 90.92, 84.01, 50.18, 47.13, 37.60, 32.38, 32.15, 20.42, 13.90. [M+H$^+$] calcd. for $C_{21}H_{26}N^+$, 292.2060; found, 292.2065.

***N*-(1-Cyclohexyl-3-phenylprop-2-yn-1-yl)butan-1-amine** (1t)

Yellow oil, 65%. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.41 (dd, *J* = 7.6, 1.8 Hz, 2H), 7.29 – 7.18 (m, 3H), 3.37 (d, *J* = 5.5 Hz, 1H), 2.92 (dt, *J* = 10.7, 7.5 Hz, 1H), 2.62 (dt, *J* = 11.2, 7.6 Hz, 1H), 1.92 – 1.81 (m, 2H), 1.77 (m, 2H), 1.67 (d, *J* = 11.0 Hz, 1H), 1.63 – 1.55 (m, 1H), 1.54 – 1.43 (m, 2H), 1.42 – 1.34 (m, 2H), 1.21 (ddd, *J* = 20.5, 17.0, 9.6 Hz, 6H), 0.93 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 131.31, 127.84, 127.36, 123.44, 90.21, 84.06, 56.11, 47.33, 42.44, 31.96, 30.05, 28.40, 26.29, 26.01, 25.87, 20.25, 13.71.

Procedures for the preparation of 1u[25c, 25d,26],

Propargyl bromide (3 mL, 27 mmol) was added to benzyl amine (18 mL, 162 mmol) over half hours via addition funnel, and allowed to stir overnight. The resulting mixture was diluted in Et_2O, and extracted with saturated aq. $NaHCO_3$ and dried over $MgSO_4$. The reaction mixture was concentrated and purified by silica gel column chromatography (PE/EtOAc=10/1) to afford the corresponding propargylic amine **1u**.

***N*-Benzylprop-2-yn-1-amine** (1u)

Yellow oil, 68%. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.35 – 7.29 (m, 4H), 7.25 (td, *J* = 5.7, 2.6 Hz, 1H), 3.86 (s, 2H), 3.40 (d, *J* = 2.4 Hz, 2H), 2.25 (t, *J* = 2.4 Hz, 1H), 1.74 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 139.26, 128.32, 128.29, 127.05, 81.95, 71.48, 52.14, 37.19.

Typical Procedure for Reaction of Propargylic Amines with Se and TFBen

Se (0.3 mmol, 1.5 equiv.), TFBen (0.2 mmol, 1.0 equiv.) and tBuOK (0.1 mmol, 50 mol%) were added to a 15 mL tube equipped with a magnetic stirrer which was then placed under vacuum and refilled with nitrogen three times. A solution of propargylic amine **1** (0.2 mmol) in DMF (1.0 mL) was added to the reaction tube, then the tube was sealed and the mixture was stirred at 35 °C for 24 h. After the reaction was completed, the reaction mixture was diluted with 50 mL water and extracted with 30 mL EtOAc three times. The combined organic phases were dried with anhydrous Na_2SO_4, concentrated and purified by silica gel column chromatography (PE/EtOAc=10/1) to obtain desired (*Z*)-1,3-selenazolidin-2-one 2.

(*Z*)-5-Benzylidene-3-butyl-4,4-dimethyl-1,3-selenazolidin-2-one (2aa)

56.7 mg, 88% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.36 (t, *J* = 7.6 Hz, 2H), 7.29 – 7.22 (m, 3H), 6.91 (s, 1H), 3.28 – 3.24 (m, 2H), 1.65 – 1.61 (m, 2H), 1.57 (s, 6H), 1.35 (dd, *J* = 15.2, 7.3 Hz, 2H), 0.94 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 165.75, 140.60, 137.03, 128.57, 127.64, 127.26, 122.39, 69.12, 43.28, 31.55, 28.19, 20.35, 13.72. HRMS (ESI): [M+H$^+$] calcd. for $C_{16}H_{22}NOSe^+$, 324.0861; found, 324.0875.

(*Z*)-3-Butyl-4,4-dimethyl-5-(4-methylbenzylidene)-1,3-selenazolidin-2-one (2ab)

60.5 mg, 90% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.18 (m, 4H), 6.88 (s, 1H), 3.28 – 3.24 (m, 2H), 2.34 (s, 3H), 1.67 – 1.61 (m, 2H), 1.57 (s, 6H), 1.36 (dq, *J* = 14.8, 7.4 Hz, 2H), 0.95 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 165.89, 139.36, 137.11, 134.16, 129.25, 127.54, 122.26, 69.04, 43.23, 31.55, 28.15, 21.17, 20.35, 13.73. HRMS (ESI): [M+H$^+$] calcd. for $C_{17}H_{24}NOSe^+$, 338.1018; found, 338.1029.

(*Z*)-3-Butyl-5-(4-methoxybenzylidene)-4,4-dimethyl-1,3-selenazolidin-2-one (2ac)

59.9 mg, 85% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.21 (d, *J* = 8.6 Hz, 2H), 6.90 (d, *J* = 8.6 Hz, 2H), 6.85 (s, 1H), 3.81 (s, 3H), 3.28 – 3.24 (m, 2H), 1.66 – 1.61 (m, 2H), 1.56 (s, 6H), 1.40 – 1.30 (m, 2H), 0.94 (t, J = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 165.85, 158.61, 138.01, 129.57, 128.91, 121.85, 113.93, 69.00, 55.20, 43.20, 31.53, 28.10, 20.32, 13.71. HRMS (ESI): [M+H$^+$] calcd. for $C_{17}H_{24}NO_2Se^+$, 354.0967; found, 354.0978.

(*Z*)-3-Butyl-4,4-dimethyl-5-(4-propylbenzylidene)-1,3-selenazolidin-2-one (2ad)

68.5 mg, 94% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.22 – 7.17 (m, 4H), 6.89 (s, 1H), 3.29 – 3.25 (m, 2H), 2.60 – 2.56 (m, 2H), 1.69 – 1.60 (m, 4H), 1.57 (s, 6H), 1.36 (dq, *J* = 14.7, 7.4 Hz, 2H), 0.94 (td, *J* = 7.3, 4.9 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 165.87, 141.93, 139.24, 134.33, 128.67, 127.54, 122.29, 69.08, 43.23, 37.66, 31.54, 28.17, 24.34, 20.34, 13.73. HRMS (ESI): [M+H$^+$] calcd. for $C_{19}H_{28}NOSe^+$, 366.1331; found, 366.1342.

(*Z*)-3-Butyl-5-(4-(*tert*-butyl)benzylidene)-4,4-dimethyl-1,3-selenazolidin-2-one (2ae)

72.7 mg, 96% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.39 (d, *J* = 8.2 Hz, 2H), 7.23 (t, *J* = 5.8 Hz, 2H), 6.88 (s, 1H), 3.28 – 3.24 (m, 2H), 1.67 – 1.61 (m, 2H), 1.56 (s, 6H), 1.37 (dd, *J* = 15.1, 7.8 Hz, 2H), 1.31 (s, 9H), 0.94 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 165.82, 150.27, 139.27, 134.01, 127.37, 125.47, 122.11, 69.09, 43.22, 34.53, 31.52, 31.14, 28.17, 20.32, 13.71. HRMS (ESI): [M+H$^+$] calcd. for $C_{20}H_{30}NOSe^+$, 380.1487; found, 380.1500.

(*Z*)-3-Butyl-5-(4-fluorobenzylidene)-4,4-dimethyl-1,3-selenazolidin-2-one (2af)

59.9 mg, 88% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.27 – 7.24 (m, 2H), 7.06 (dd, *J* = 11.9, 5.3 Hz, 2H), 6.87 (s, 1H), 3.28 – 3.24 (m, 2H), 1.67 – 1.59 (m, 2H), 1.57 (s, 6H), 0.95 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 165.40, 161.63 (d, *J* = 246.4 Hz), 140.64, 133.22 (d, *J* = 3.2 Hz), 129.30 (d, *J* = 7.9 Hz), 121.30, 115.55 (d, *J* = 21.5 Hz), 69.04, 43.33, 31.54, 28.16, 20.35, 13.73. HRMS (ESI): [M+Na$^+$] calcd. for $C_{16}H_{20}FNNaOSe^+$, 364.0586; found, 364.0605.

(*Z*)-3-Butyl-5-(4-chlorobenzylidene)-4,4-dimethyl-1,3-selenazolidin-2-one (2ag)

58.7 mg, 81% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.33 (d, *J* = 8.4 Hz, 2H), 7.21 (d, *J* = 8.4 Hz, 2H), 6.85 (s, 1H), 3.28 – 3.24 (m, 2H), 1.67 – 1.61 (m, 2H), 1.57 (s, 6H), 1.40 – 1.31 (m, 2H), 0.95 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 165.25, 141.72, 135.55, 132.84, 128.89, 128.74, 121.23, 69.12, 43.36, 31.52, 28.18, 20.35, 13.73. HRMS (ESI): [M+Na$^+$] calcd. for $C_{16}H_{20}ClNNaOSe^+$, 380.0291; found, 380.0310.

(*Z*)-5-(4-Bromobenzylidene)-3-butyl-4,4-dimethyl-1,3-selenazolidin-2-one (2ah)

61.0 mg, 76% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.48 (d, *J* = 8.3 Hz, 2H), 7.15 (d, *J* = 8.3 Hz, 2H), 6.83 (s, 1H), 3.28 – 3.24 (m, 2H), 1.67 – 1.61 (m, 2H), 1.57 (s, 6H), 1.35 (dd, *J* = 15.1, 7.4 Hz, 2H), 0.94 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 165.19, 141.86, 135.98, 131.66, 129.16, 121.26, 120.95, 69.13, 43.35, 31.50, 28.14, 20.33, 13.72. HRMS (ESI): [M+Na$^+$] calcd. for $C_{16}H_{20}BrNNaOSe^+$, 423.9786; found, 423.9770.

(*Z*)-3-Benzyl-5-benzylidene-4,4-dimethyl-1,3-selenazolidin-2-one (2ba)

42.8 mg, 60% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.37 (t, *J* = 7.6 Hz, 2H), 7.32 – 7.24 (m, 8H), 6.90 (s, 1H), 4.62 (s, 2H), 1.52 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 167.03, 140.32, 137.88, 136.92, 128.61, 128.53, 127.67, 127.40, 127.19, 126.94, 122.58, 69.39, 45.90, 28.26. HRMS (ESI): [M+Na$^+$] calcd. for $C_{19}H_{19}NNaOSe^+$, 380.0524; found, 380.0518.

(*Z*)-5-Benzylidene-3-(4-methoxybenzyl)-4,4-dimethyl-1,3-selenazolidin-2-one (2bb)

49.4 mg, 67% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.37 (t, *J* = 7.6 Hz, 2H), 7.29 – 7.21 (m, 5H), 6.89 (s, 1H), 6.84 (d, *J* = 8.6 Hz, 2H), 4.56 (s, 2H), 3.78 (s, 3H), 1.51 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 166.96, 158.77, 140.45, 136.94, 130.03, 128.60, 128.38, 127.67, 127.37, 122.48, 113.90, 69.41, 55.20, 45.40, 28.30. HRMS (ESI): [M+Na$^+$] calcd. for $C_{20}H_{21}NNaO_2Se^+$, 410.0630; found, 410.0639.

(*Z*)-5-Benzylidene-3-(4-chlorobenzyl)-4,4-dimethyl-1,3-selenazolidin-2-one (2bc)

64.1 mg, 82% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.38 (t, *J* = 7.6 Hz, 2H), 7.26 (dt, *J* = 12.9, 5.5 Hz, 7H), 6.91 (s, 1H), 4.57 (s, 2H), 1.51 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 167.23, 139.99, 136.83, 136.45, 133.04, 128.71, 128.65, 128.39, 127.67, 127.50, 122.80, 69.31, 45.28, 28.26. HRMS (ESI): [M+Na$^+$] calcd. for $C_{19}H_{18}ClNNaOSe^+$, 414.0134; found, 414.0150.

(*Z*)-5-Benzylidene-4,4-dimethyl-3-octyl-1,3-selenazolidin-2-one (2c)

71.9 mg, 95% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.36 (t, *J* = 7.6 Hz, 2H), 7.29 – 7.22 (m, 3H), 6.91 (s, 1H), 3.27 – 3.23 (m, 2H), 1.64 – 1.62 (m, 2H), 1.57 (s, 6H), 1.31 – 1.27 (m, 10H), 0.88 (t, *J* = 6.7 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 165.67, 140.57, 136.99, 128.55, 127.62, 127.23, 122.35, 69.08, 43.50, 31.72, 29.44, 29.16, 28.17, 27.11, 22.56, 14.04, 0.95. HRMS (ESI): [M+Na$^+$] calcd. for $C_{20}H_{29}NNaOSe^+$, 402.1307; found, 402.1317.

(*Z*)-4-Benzylidene-1-butyl-3-selena-1-azaspiro[4.4]nonan-2-one (2e)

43.2 mg, 62% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.36 (t, *J* = 7.6 Hz, 2H), 7.28 – 7.23 (m, 3H), 6.81 (s, 1H), 3.24 – 3.20 (m, 2H), 2.23 – 2.17 (m, 2H), 2.03 (dt, *J* = 13.8, 8.3 Hz, 2H), 1.88 (t, *J* = 7.2 Hz, 4H), 1.65 (ddd, *J* = 11.9, 10.4, 6.7 Hz, 2H), 1.35 (dd, *J* = 15.1, 7.5 Hz, 2H), 0.94 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 167.34, 141.69, 136.08, 128.51, 128.14, 127.11, 118.96, 77.89, 42.69, 38.82, 31.55, 25.35, 20.41, 13.70. HRMS (ESI): [M+Na$^+$] calcd. for $C_{18}H_{23}NNaOSe^+$, 372.0837; found, 372.0835.

(*Z*)-4-Benzylidene-1-butyl-3-selena-1-azaspiro[4.5]decan-2-one (2f)

60.9 mg, 84% yield, pale yellow solid, mp. 132.9-135.1 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.36 (t, *J* = 7.4 Hz, 2H), 7.30 – 7.26 (m, 3H), 7.24 (s, 1H), 3.28 – 3.24 (m, 2H), 2.17 (d, *J* = 9.2 Hz, 2H), 1.79 (dd, *J* = 22.8, 12.0 Hz, 6H), 1.58 – 1.51 (m, 2H), 1.38 – 1.26 (m, 4H), 0.94 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 167.44, 140.68, 137.37, 128.41, 127.90, 127.54, 126.68, 70.86, 43.02, 33.30, 32.21, 24.66, 23.01, 20.26, 13.76. HRMS (ESI): [M+Na$^+$] calcd. for $C_{19}H_{25}NNaOSe^+$, 386.0994; found, 386.0988.

(*Z*)-4-Benzylidene-1-butyl-3-selena-1-azaspiro[4.6]undecan-2-one (2g)

44.4 mg, 59% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.38 (t, *J* = 7.5 Hz, 2H), 7.29 (dd, *J* = 13.6, 8.8 Hz, 3H), 7.05 (s, 1H), 3.39 – 3.35 (m, 2H), 2.19 (dd, *J* = 15.5, 8.9 Hz, 2H), 2.08 (dd, *J* = 15.6, 7.4 Hz, 2H), 1.69 – 1.62 (m, 10H), 1.36 (dd, *J* = 15.0, 7.5 Hz, 2H), 0.95 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 166.06, 141.05, 137.21, 128.54, 127.95, 127.47, 124.31, 75.51, 43.57, 38.69, 31.60, 31.51, 24.23, 20.50, 13.75. HRMS (ESI): [M+Na$^+$] calcd. for $C_{20}H_{27}NNaOSe^+$, 400.1150; found, 400.1158.

(*Z*)-4-Benzylidene-1-butyl-8-methyl-3-selena-1-azaspiro[4.5]decan-2-one (2h)

64.3 mg, 92% yield, pale yellow solid, mp 103.1-106.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.37 (t, *J* = 7.5 Hz, 2H), 7.26 (d, *J* = 5.0 Hz, 3H), 7.24 (s, 1H), 3.26 – 3.22 (m, 2H), 2.18 (d, *J* = 13.7 Hz, 2H), 1.87 – 1.77 (m, 4H), 1.58 – 1.50 (m, 2H), 1.44 (t, *J* = 9.8 Hz, 3H), 1.34 (dd, *J* = 15.0, 7.5 Hz, 2H), 0.98 – 0.91 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 167.56, 140.68, 137.33, 128.38, 127.89, 127.55, 126.79, 70.59, 42.90, 33.13, 32.18, 31.67, 31.47, 22.01, 20.22, 13.74. HRMS (ESI): [M+H$^+$] calcd. for $C_{20}H_{28}NOSe^+$, 378.1331; found, 378.1315.

(*Z*)-3-Butyl-4,4-dimethyl-5-(thiophen-3-ylmethylene)-1,3-selenazolidin-2-one (2i)

62.4 mg, 95% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.33 (dd, *J* = 5.0, 2.9 Hz, 1H), 7.18 (d, J = 1.8 Hz, 1H), 7.10 (dd, *J* = 5.0, 0.9 Hz, 1H), 6.93 (s, 1H), 3.27 – 3.23 (m, 2H), 1.63 (ddd, *J* = 11.9, 10.3, 6.6 Hz, 2H), 1.55 (s, 6H), 1.35 (dq, *J* = 14.8, 7.4 Hz, 2H), 0.94 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 165.26, 139.82, 138.37, 127.47, 125.76, 122.27, 116.88, 68.84, 43.34, 31.49, 28.16, 20.33, 13.73. HRMS (ESI): [M+H$^+$] calcd. for $C_{14}H_{20}NOSSe^+$, 330.0425; found, 330.0446.

(*Z*)-3-Butyl-5-heptylidene-4,4-dimethyl-1,3-selenazolidin-2-one (2j)

44.9 mg, 68% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 5.78 (t, *J* = 6.9 Hz, 1H), 3.19 – 3.15 (m, 2H), 1.93 (dd, *J* = 14.3, 7.1 Hz, 2H), 1.62 – 1.54 (m, 2H), 1.42 (s, 6H), 1.40 – 1.36 (m, 2H), 1.34 – 1.26 (m, 8H), 0.89 (dt, *J* = 13.5, 7.1 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 165.75, 139.13, 122.70, 67.57, 43.07, 33.63, 31.61, 31.59, 28.73, 28.62, 27.92, 22.52, 20.34, 14.02, 13.74. HRMS (ESI): [M+H$^+$] calcd. for $C_{16}H_{30}NOSe^+$, 332.1487; found, 332.1504.

(*Z*)-3-Butyl-4,4-dimethyl-5-(3-phenylpropylidene)-1,3-selenazolidin-2-one (2k)

39.2 mg, 56% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.30 – 7.26 (m, 2H), 7.18 (dd, *J* = 15.0, 7.6 Hz, 3H), 5.81 (t, *J* = 6.9 Hz, 1H), 3.19 – 3.15 (m, 2H), 2.73 (t, *J* = 7.6 Hz, 2H), 2.27 (dd, *J* = 14.9, 7.2 Hz, 2H), 1.58 (tt, *J* = 8.0, 6.6 Hz, 2H), 1.40 (s, 6H), 1.32 (dd, *J* = 15.1, 7.5 Hz, 2H), 0.92 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 165.51, 140.99, 140.37, 128.40, 128.33, 126.01, 121.46, 67.60, 43.09, 35.25, 34.71, 31.57, 27.89, 20.32, 13.73. HRMS (ESI): [M+Na$^+$] calcd. for $C_{18}H_{25}NNaOSe^+$, 374.0994; found, 374.1004.

(*Z*)-3-Butyl-5-(cyclopropylmethylene)-4,4-dimethyl-1,3-selenazolidin-2-one (2l)

44.7 mg, 78% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 5.29 (d, *J* = 8.4 Hz, 1H), 3.19 – 3.15 (m, 2H), 1.61 – 1.53 (m, 2H), 1.41 (s, 6H), 1.31 (dd, *J* = 15.1, 7.5 Hz, 2H), 1.11 – 1.03 (m, 1H), 0.91 (t, *J* = 7.3 Hz, 3H), 0.83 – 0.78 (m, 2H), 0.45 – 0.41 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 165.81, 137.11, 125.86, 67.53, 43.08, 31.61, 27.80, 20.34, 15.02, 13.74, 6.92. HRMS (ESI): [M+Na$^+$] calcd. for $C_{13}H_{21}NNaOSe^+$, 310.0681; found, 310.0688.

(*Z*)-3-Butyl-5-(cyclohex-1-en-1-ylmethylene)-4,4-dimethyl-1,3-selenazolidin-2-one (2m)

50.9 mg, 78% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.27 (s, 1H), 5.71 (s, 1H), 3.22 – 3.18 (m, 2H), 2.14 – 2.11 (m, 4H), 1.66 – 1.62 (m, 2H), 1.60 – 1.54 (m, 4H), 1.46 (s, 6H), 1.35 – 1.29 (m, 2H), 0.92 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ = 166.76, 135.53, 135.30, 128.88, 125.31, 68.82, 43.05, 31.59, 28.05, 27.84, 25.63, 22.59, 21.88, 20.36, 13.74. HRMS (ESI): [M+Na$^+$] calcd. for $C_{16}H_{25}NNaOSe^+$, 350.0994; found, 350.1003.

(*Z*)-5-Benzylidene-4,4-dimethyl-1,3-selenazolidin-2-one (2n)

45.8 mg, 86% yield, pale yellow solid, mp 123.4-125.7 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.40 (t, *J* = 7.6 Hz, 2H), 7.38 – 7.28 (m, 3H), 6.87 (s, 1H), 1.64 (s, 6H).^{13}C NMR (100 MHz, $CDCl_3$) δ = 168.70, 141.54, 136.77, 128.59, 127.53, 127.33, 122.76, 64.59, 29.99. HRMS (ESI): [M+Na$^+$] calcd. for $C_{12}H_{13}NNaOSe^+$, 290.0055; found, 290.0069.

(*Z*)-5-Benzylidene-3-butyl-4-phenyl-1,3-selenazolidin-2-one (2o)

37.0 mg, 50% yield, pale yellow solid, mp 93.5-96.9 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.38 (dt, *J* = 10.3, 4.5 Hz, 5H), 7.32 (t, *J* = 7.6 Hz, 2H), 7.25 – 7.15 (m, 3H), 6.73 (s, 1H), 5.43 (s, 1H), 3.77 – 3.69 (m, 1H), 2.70 (ddd, *J* = 13.8, 8.0, 5.7 Hz, 1H), 1.50 – 1.48 (m, 2H), 1.29 – 1.26 (m, 2H), 0.88 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 166.63, 139.69, 136.40, 133.03, 129.16, 128.78, 128.55, 127.61, 127.38, 126.92, 126.65, 71.28, 43.53, 29.23, 19.94, 13.65. HRMS (ESI): [M+Na$^+$] calcd. for $C_{20}H_{21}NNaOSe^+$, 394.0681; found, 394.0671.

(*Z*)-5-Benzylidene-3-butyl-4-(4-methoxyphenyl)-1,3-selenazolidin-2-one (2p)

46.5 mg, 58% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.34 – 7.30 (m, 4H), 7.22 – 7.16 (m, 3H), 6.93 (d, *J* = 8.6 Hz, 2H), 6.69 (s, 1H), 5.40 (s, 1H), 3.82 (s, 3H), 3.71 (dt, *J* = 14.0, 7.8 Hz, 1H), 2.71 (ddd, *J* = 13.8, 8.2, 5.6 Hz, 1H), 1.49 – 1.46 (m, 2H), 1.29 – 1.26 (m, 3H), 0.88 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 166.42, 159.92, 136.48, 133.49, 131.70, 128.55, 128.27, 127.61, 127.33, 126.47, 114.50, 70.84, 55.32, 43.47, 29.21, 19.96, 13.68. HRMS (ESI): [M+Na$^+$] calcd. for $C_{21}H_{23}NNaO_2Se^+$, 424.0786; found, 424.0780.

(*Z*)-5-Benzylidene-3-butyl-4-(4-fluorophenyl)-1,3-selenazolidin-2-one (2q)

49.7 mg, 64% yield, pale yellow solid, mp 83.9-86.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.39 (dd, *J* = 8.5, 5.2 Hz, 2H), 7.33 (t, *J* = 7.6 Hz, 2H), 7.22 (t, *J* = 7.4 Hz, 1H), 7.17 (d, *J* = 7.5 Hz, 2H), 7.10 (t, *J* = 8.6 Hz, 2H), 6.70 (s, 1H), 5.43 (s, 1H), 3.76 – 3.67 (m, 1H), 2.69 (ddd, *J* = 13.9, 8.3, 5.6 Hz, 1H), 1.48 (td, *J* = 15.8, 8.2 Hz, 2H), 1.31 – 1.28 (m, 2H), 0.88 (t, *J* = 7.3 Hz , 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 166.46, 162.87 (d, *J* = 249.0 Hz), 136.26, 135.60 (d, *J* = 2.6 Hz), 132.85, 128.68 (d, *J* = 3.1 Hz), 128.61, 127.61, 127.52, 126.83, 116.20 (d, *J* = 21.8 Hz), 70.52, 43.53, 29.22, 19.93, 13.64. HRMS (ESI): [M+Na$^+$] calcd. for $C_{20}H_{20}FNNaOSe^+$, 412.0586; found, 412.0602.

(*Z*)-5-Benzylidene-3-butyl-4-ethyl-1,3-selenazolidin-2-one (2r)

55.4 mg, 86% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.39 – 7.35 (m, 2H), 7.27 – 7.23 (m, 3H), 6.85 (s, 1H), 4.44 – 4.42 (m, 1H), 3.81 (ddd, *J* = 13.9, 8.8, 7.2 Hz, 1H), 3.00 (ddd, *J* = 13.9, 8.6, 5.3 Hz, 1H), 2.00 – 1.90 (m, 1H), 1.85 – 1.76 (m, 1H), 1.66 – 1.50 (m, 2H), 1.37 – 1.31 (m, 2H), 0.96 (dt, *J* = 11.5, 7.4 Hz, 6H).^{13}C NMR (100 MHz, $CDCl_3$) δ = 166.63, 136.37, 132.65, 128.59, 127.62, 127.32, 124.92, 67.69, 42.96, 29.54, 27.35, 20.00, 13.70, 7.67. HRMS (ESI): [M+H$^+$] calcd. for $C_{16}H_{21}NNaOSe^+$, 346.0681; found, 346.0700.

(*Z*)-5-Benzylidene-3-butyl-4-phenethyl-1,3-selenazolidin-2-one (2s)

76.5 mg, 96% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.38 (t, *J* = 7.6 Hz, 2H), 7.30 – 7.25 (m, 5H), 7.22 – 7.16 (m, 3H), 6.92 (s, 1H), 4.48 (d, *J* = 4.2 Hz, 1H), 3.85 – 3.77 (m, 1H), 2.99 (ddd, *J* = 13.8, 8.3, 5.5 Hz, 1H), 2.80 – 2.67 (m, 2H), 2.27 – 2.18 (m, 1H), 2.11 – 2.03 (m, 1H), 1.61 – 1.51 (m, 2H), 1.32 (dt, *J* = 14.8, 7.3 Hz, 2H), 0.92 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 166.49, 140.76, 136.22, 132.68, 128.62, 128.49, 128.18, 127.67, 127.46, 126.10, 125.29, 66.40, 42.96, 35.83, 29.52, 29.48, 19.93, 13.64. HRMS (ESI): [M+Na$^+$] calcd. for $C_{22}H_{25}NNaOSe^+$, 422.0994; found, 422.1009.

(*Z*)-5-Benzylidene-3-butyl-4-cyclohexyl-1,3-selenazolidin-2-one (2t)

73.0 mg, 97% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.38 (t, *J* = 7.6 Hz, 2H), 7.26 (t, *J* = 8.1 Hz, 3H), 6.80 (s, 1H), 4.23 (d, *J* = 2.2 Hz, 1H), 3.82 (ddd, *J* = 14.1, 8.8, 7.0 Hz, 1H), 3.06 (ddd, *J* = 14.0, 8.8, 5.3 Hz, 1H), 1.80 (d, *J* = 8.6 Hz, 4H), 1.68 – 1.52 (m, 4H), 1.42 – 1.28 (m, 4H), 1.15 (dt, *J* = 26.1, 11.1 Hz, 3H), 0.94 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 167.26, 136.23, 130.98, 128.55, 127.78, 127.41, 126.90, 72.20, 43.70, 42.25, 29.60, 29.29, 26.80, 26.46, 26.36, 26.16, 20.02, 13.72. HRMS (ESI): [M+Na$^+$] calcd. for $C_{20}H_{27}NNaOSe^+$, 400.1150; found, 400.1170.

3-Benzyl-5-methylene-1,3-selenazolidin-2-one (2u)

42.9 mg, 85% yield, yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.38 – 7.26 (m, 5H), 5.55 (s, 1H), 5.24 (s, 1H), 4.55 (s, 2H), 4.13 (s, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ = 167.29, 135.48, 134.77, 128.84, 128.07, 127.95, 110.91, 55.89, 48.57. HRMS (ESI): [M+H$^+$] calcd. for $C_{11}H_{12}NOSe^+$, 254.0079; found, 254.0076.

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[24] CCDC-1817355 and CCDC-1817678 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

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FULL PAPER

Base-Promoted Carbonylative Cyclization of Propargylic Amines with Selenium under CO gas-free conditions

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