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Facile Synthesis of Selenium-Containing Bicyclic β-Lactams through Enyne Metathesis

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Novel selenium-containing bicyclic β-lactams were obtained through stereoselective insertion of (but-3-enyl)seleno and propargylseleno moieties at the C(4) positions of azetidinones with subsequent ring-closing enyne metathesis.

Introduction

The β-lactams are an important class of heterocyclic compounds, thanks to their antibiotic activity[1] and their utility as versatile building blocks[2] in organic synthesis. Much effort has been expended in recent years in the preparation of new structural types containing the common feature of an azetidin-2-one ring, which should overcome bacterial defense mechanisms. In this context, however, there are very few reports relating to the synthesis of selenium-containing bicyclic β-lactams available in the literature, due to the difficulties involved in their preparation.[3] We have recently reported a 2-(trimethylsilyl)ethyl (TSE) protection approach[4] for the incorporation of selenium into penam and cephem nuclei and an iodocyclization approach[5] for the synthesis of selenium-containing β-lactams with a selenium atom at the 3- or 4-position, with the aim of providing some novel compounds of potential biological significance.

The generation of β-lactams by ring-closing metathesis (RCM)[6] and ring-closing enyne metathesis (RCEYM)[7] has received considerable attention (Figure 1). As part of our ongoing work, we have recently reported the first ring-closing metathesis (RCM)[8] of allylseleno compounds for the synthesis of seven-, eight-, or nine-membered selenium-containing bicyclic β-lactams.[9] Here we have extended the reaction to related enyne systems and wish to report the first RCEYMs of (but-3-enyl)seleno or propargylseleno compounds in the presence of the second-generation Grubbs catalyst for the formation of seven- and eight-membered selenium-containing bicyclic β-lactams.

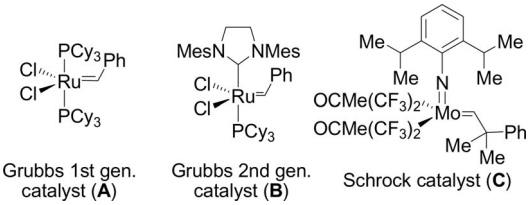
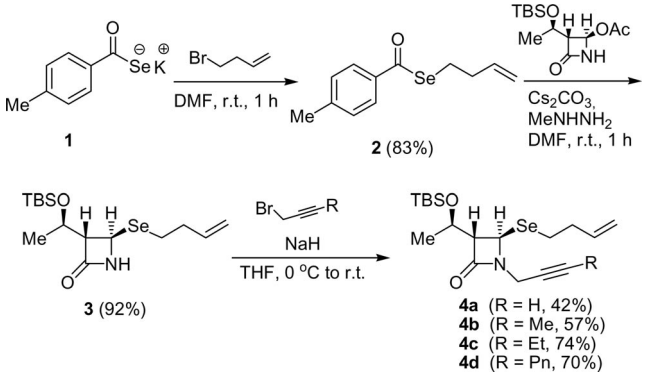


Figure 1. Ruthenium and molybdenum carbene complexes used in ring-closing enyne metathesis (RCEYM) reactions.

Results and Discussion

As part of our program relating to the synthesis of selenium-containing bicyclic β-lactams, we have recently reported the novel selenating reagent **2** (Scheme 1),[9] prepared by treatment of potassium *p*-methylselenobenzoate (**1**)[10] with 4-bromobut-1-ene, for the synthesis of these compounds by RCM. This selenating reagent **2** has a carbonyl carbon as the reactive site and is susceptible to nucleophilic attack by amines, thereby producing (but-3-enyl)-selenolate anion. This anion was further treated with an optically pure 4-acetoxyazetidinone, allowing the incorporation of the (but-3-enyl)seleno moiety onto the β-lactam skeleton with complete retention of configuration in compound **3** (92% yield, Scheme 1).[9]



Scheme 1. Synthesis of the selenating reagent **2** and the key intermediates **4**.

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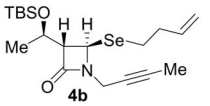
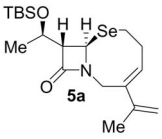
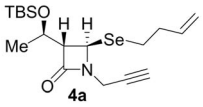
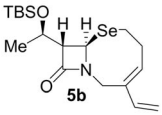
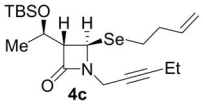
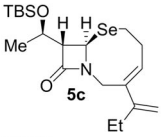
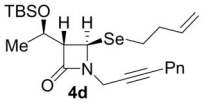
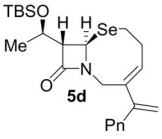
For the synthesis of selenium-containing bicyclic β-lactams through RCEYM reactions the intermediates with (but-3-enyl)seleno or propargylseleno moieties at the C(4) positions in the azetidinones were necessary. Treatment of **3** with sodium hydride in THF at 0 °C and subsequent addition of substituted propargyl bromides afforded the corresponding previously unknown key intermediates **4a**–**4d** for the RCEYM reaction in 42–74% yields (Scheme 1).

The key intermediates **4**, containing both alkyne and alkene groups, were next used for ring-closing enyne metathesis (RCEYM) to provide selenium-containing bicyclic β-lactams (Table 1). When the RCEYM reaction of the key intermediate **4b** was carried out in the presence of the Schrock catalyst **C** (10 mol-%) in toluene the results were disappointing and only the starting material **4b** was recovered (Table 1, Entry 1). Use of the first-generation Grubbs catalyst **A** (10 mol-%) in toluene in the reaction, moreover, afforded the corresponding product **5a** only in trace amounts (Table 1, Entry 2). The use of the second-generation Grubbs catalyst **B** (10 mol-%) in the RCEYM reaction, however, was found to be effective, and an eight-membered cyclized product, the 1,3-selenazocine **5a**, was obtained in 48% yield (Table 1, Entry 4). To the best of our knowledge, this represents the first example of the RCEYM of a (but-3-enyl)-seleno compound. Corresponding reactions of the other key intermediates **4a**–**4d** were carried out under similar conditions, and the results are shown in Table 1. When the RCEYM of the unsubstituted alkyne **4a** was carried out in the presence of catalyst **B** (10 mol-%) the 1,3-selenazocine compound **5b** was obtained only in trace amounts (Table 1, Entry 5), whereas the reactions of the other substituted alkyne compounds **4c** or **4d** in the presence of catalyst **B** (10 mol-%) afforded the corresponding 1,3-selenazocine compounds **5c** or **5d**, respectively, in good yields (Table 1, Entries 6 and 7).

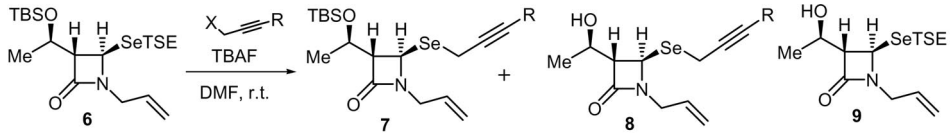
Table 1. Ring-closing enyne metatheses of compounds **4**.[a]

Entry	Starting	Catalyst (10 mol-%)	Time	5 Product	5 Yield[b]
1	 **4b**	**C**	2 days	 **5a**	0[c]
2		**A**	4 days		trace[c]
3		**B**	4 days		trace[d]
4		**B**	1 day		48
5	 **4a**	**B**	2 days	 **5b**	trace
6	 **4c**	**B**	1 day	 **5c**	47
7	 **4d**	**B**	1 day	 **5d**	50

[a] All reactions were carried out on ca. 0.05 mmol scales in toluene at reflux. [b] Isolated yields. [c] Starting material was recovered quantitatively. [d] The reaction was carried out in DCM.

With access to selenium-containing bicyclic β-lactams through RCEYM reactions now available, we next turned our attention to the stereoselective insertion of propargylseleno moieties at the C(4) position in the β-lactam skeleton. Our group has demonstrated the utility of the TSE protection approach for the synthesis of variety of selenium-containing bicyclic β-lactams.[4,9] Compound **6** (Table 2) was therefore selected as an important starting material for the insertion of the propargylseleno moiety at the C(4) position in the azetidin-2-one system. Selective removal of the TSE group of **6** and subsequent in situ alkylation of the selenolate anion was attempted for the synthesis of the key intermediates **7** having propargylseleno moieties at their C(4) positions (Table 2). After screening of several sets of reaction conditions we found that the treatment of **6** with TBAF (2.5 equiv.) resulted in the selective removal of the TSE group and, further, that in situ alkylation with various propargyl bromides readily afforded the key intermediates **7a**–**7d** along with the TBS-deprotected intermediates **8a**–**8d** and **9**, respectively (Table 2, Entries 1, 3, 4, and 6). To ascer-

Table 2. Synthesis of the key intermediates **7**.[a]



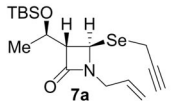
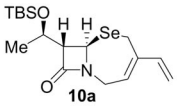
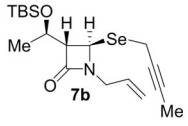
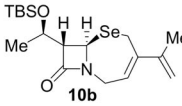
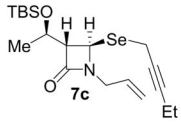
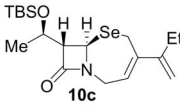
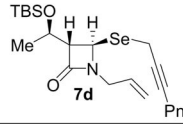
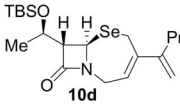
Entry	X	R	Time (min)	Yield (%) [b] 7	8	9
1	Br	H	35 min	43 (**7a**)	41 (**8a**)	7
2	Cl	H	10 min	57 (**7a**)	27 (**8a**)	4
3	Br	Me	25 min	68 (**7b**)	16 (**8b**)	6
4	Br	Et	75 min	46 (**7c**)	25 (**8c**)	19
5	Cl	Et	15 min	61 (**7c**)	14 (**8c**)	5
6	Br	Pn	40 min	54 (**7d**)	15 (**8d**)	10
7	Cl	Pn	10 min	70 (**7d**)	11 (**8d**)	4

[a] The reactions were carried out in DMF with **6** (1 equiv.), the propargyl halide (10 equiv.), and TBAF (2.5 equiv.) at room temp. under argon (bubbling). [b] Isolated yields.

tain the effect of the halide group in the deprotection reaction, the reactions were carried out with substituted propargyl chlorides and the corresponding key intermediate **7** were isolated in higher yields (Entries 2, 5, and 7). Use of propargyl chlorides in the reaction was found to be superior to that of propargyl bromide. Use of longer reaction times resulted in the TBS-deprotected intermediate **9** in high yields.

Now that it had been found that the (but-3-enyl)seleno moiety is compatible with the second-generation Grubbs catalyst **B** (Table 1), we turned out attention to the RCEYM of propargylseleno moieties (Table 3). Attempts to bring about the cyclization of **7a** in the presence of the second-generation Grubbs catalyst **B** failed (Table 3, Entry 1), but treatment of the key intermediate **7b**–**7d** with catalyst **B** (10 mol-%) afforded the corresponding 1,3-selenazepine compounds **10b**–**10d**, respectively, in good yields (Table 3, Entries 2–4).

Table 3. Ring-closing enyne metathesis of **7**.[a]

Entry	Starting	Catalyst (10 mol-%)	Time	10 Product	10 Yield [b]
1	 **7a**	**B**	4 days	 **10a**	0
2	 **7b**	**B**	2 days	 **10b**	44
3	 **7c**	**B**	1 day	 **10c**	46
4	 **7d**	**B**	1 day	 **10d**	59

[a] All reactions were carried out on ca. 0.05 mmol scales in toluene at reflux. [b] Isolated yields.

The synthesis of higher-membered selenium heterocycles has been found to be very difficult. To the best of our knowledge, there are only four reports on the preparation of seven-membered selenium-containing heterocycles (that is, 1,3-selenazepines),[4,9,11] whereas there is only one report for the synthesis of eight-membered selenium heterocycles such as 1,3-selenazocine, by our group.[8] This RCEYM approach allows the synthesis of previously inaccessible selenium-containing heterocycles.

Conclusions

We have demonstrated the stereoselective insertion of (but-3-enyl)seleno and propargylseleno moieties at the C(4) positions in azetidinones. Subsequent ring-closing enyne metathesis (RCEYM) afforded the novel selenium-containing bicyclic β-lactams. Further expansion of these strategies is in progress.

Experimental Section

Typical Synthesis Procedure: Catalyst **B** (10 mol-%) was added to a compound **4** or a compound **7** (0.05 mmol) in toluene (2 mL). The mixture was allowed to stir for 1–4 d under reflux prior to destruction of the catalyst by exposure to air. The mixture was concentrated and chromatographed to afford a compound **5** or a compound **10**, respectively.

(8*R*,9*S*)-9-[(*R*)-1-(*tert*-Butyldimethylsilyloxy)ethyl]-3-(prop-1-en-2-yl)-1-aza-7-selenabicyclo[6.2.0]dec-3-en-10-one (5a): Yield 10 mg (48%). ^{1}H NMR ($CDCl_3$): δ = 0.02 (s, 3 H), 0.05 (s, 3 H), 0.84 (s, 9 H), 1.20 (d, J = 6.3 Hz, 3 H), 1.88 (s, 3 H), 2.72–2.82 (m, 3 H), 2.94–3.05 (m, 2 H), 3.89 (d, J = 13.7 Hz, 1 H), 4.17–4.23 (m, 1 H), 4.35 (d, J = 13.7 Hz, 1 H), 4.88 (d, J = 2.7 Hz, 1 H), 4.99 (s, 1 H), 5.26 (s, 1 H), 5.97 (t, J = 8.3 Hz, 1 H) ppm. ^{13}C NMR ($CDCl_3$): δ = –4.77, –4.73, 18.0, 20.7, 22.3, 24.2, 25.8, 32.2, 36.7, 50.7, 63.0, 64.7, 113.9, 130.1, 134.8, 140.4, 164.4 ppm. IR (neat): $\tilde{\nu}$ = 2954, 1754, 1633, 1608, 1471, 1386, 1254, 1056, 836 cm^{-1}. MS (EI): m/z = 358 [M – tBu]$^+$. HRMS: calcd. for $C_{15}H_{24}NO_2SeSi$ 358.0742; found 358.0732.

(8*R*,9*S*)-3-(But-1-en-2-yl)-9-[(*R*)-1-(*tert*-butyldimethylsilyloxy)-ethyl]-1-aza-7-selenabicyclo[6.2.0]dec-3-en-10-one (5c): Yield 10.1 mg (47%). ^{1}H NMR ($CDCl_3$): δ = 0.02 (s, 3 H), 0.05 (s, 3 H), 0.85 (s, 9 H), 1.07 (t, J = 7.6 Hz, 3 H), 1.20 (d, J = 6.2 Hz, 3 H), 2.25 (q, J = 7.6 Hz, 2 H), 2.72–2.82 (m, 3 H), 2.95–3.05 (m, 2 H), 3.90 (d, J = 14.4 Hz, 1 H), 4.15–4.21 (m, 1 H), 4.28 (d, J = 14.4 Hz, 1 H), 4.88 (d, J = 2.1 Hz, 1 H), 4.97 (s, 1 H), 5.23 (s, 1 H), 5.99 (t, J = 8.2 Hz, 1 H) ppm. ^{13}C NMR ($CDCl_3$): δ = –4.80, –4.64, 13.1, 18.0, 22.3, 24.2, 25.8, 26.2, 32.0, 37.3, 50.9, 63.1, 65.0, 111.8, 129.5, 134.3, 146.9, 164.4 ppm. ^{77}Se NMR ($CDCl_3$): δ = 303.3 ppm. IR (neat): $\tilde{\nu}$ = 2957, 1756, 1633, 1470, 1375, 1255, 1067, 836 cm^{-1}. MS (EI): m/z = 372 [M – tBu]$^+$. HRMS: calcd. for $C_{16}H_{26}NO_2SeSi$ 372.0898; found 372.0870.

(8*R*,9*S*)-9-[(*R*)-1-(*tert*-Butyldimethylsilyloxy)ethyl]-3-(hept-1-en-2-yl)-1-aza-7-selenabicyclo[6.2.0]dec-3-en-10-one (5d): Yield 11.8 mg (50%). ^{1}H NMR ($CDCl_3$): δ = 0.02 (s, 3 H), 0.05 (s, 3 H), 0.85 (s, 9 H), 0.89 (t, J = 6.4 Hz, 3 H), 1.20 (d, J = 6.4 Hz, 3 H), 1.23–1.35 (m, 4 H), 1.39–1.48 (m, 2 H), 2.17–2.27 (m, 2 H), 2.70–2.84 (m, 3 H), 2.94–3.05 (m, 2 H), 3.90 (d, J = 14.2 Hz, 1 H), 4.13–4.21 (m, 1 H), 4.25 (d, J = 14.2 Hz, 1 H), 4.87 (d, J = 2.3 Hz, 1 H), 4.95 (s, 1 H), 5.21 (s, 1 H), 5.97 (t, J = 8.7 Hz, 1 H) ppm. ^{13}C NMR ($CDCl_3$): δ = –4.81, –4.64, 14.1, 18.0, 22.3, 22.5, 24.2, 25.8, 28.4, 31.8, 32.0, 33.6, 37.3, 50.9, 63.1, 65.1, 112.7, 129.7, 134.2, 145.7, 164.4 ppm. IR (neat): $\tilde{\nu}$ = 2955, 1757, 1633, 1605, 1469, 1375, 1253, 1067, 835 cm^{-1}. MS (EI): m/z = 414 [M – tBu]$^+$. HRMS: calcd. for $C_{19}H_{32}NO_2SeSi$ 414.1368; found 414.1372.

(7*R*,8*S*)-8-[(*R*)-1-(*tert*-Butyldimethylsilyloxy)ethyl]-4-(prop-1-en-2-yl)-1-aza-6-selenabicyclo[5.2.0]dec-3-en-9-one (10b): Yield 8.9 mg (44%). ^{1}H NMR ($CDCl_3$): δ = 0.06 (s, 3 H), 0.07 (s, 3 H), 0.86 (s, 9 H), 1.20 (d, J = 6.2 Hz, 3 H), 1.93 (s, 3 H), 2.23 (t, J = 7.4 Hz, 2 H), 3.15 (dd, J = 1.8, 3.4 Hz, 1 H), 3.49 (d, J = 13.1 Hz, 1 H), 3.67 (d, J = 13.1 Hz, 1 H), 3.85 (dd, J = 6.2, 16.5 Hz, 1 H), 4.19–4.24 (m, 1 H), 4.34 (dd, J = 6.2, 16.5 Hz, 1 H), 5.07 (s, 1 H), 5.08 (d, J = 1.4 Hz, 1 H), 5.13 (s, 1 H), 5.71 (t, J = 6.9 Hz, 1 H) ppm. ^{13}C NMR ($CDCl_3$): δ = –5.16, –4.22, 17.8, 18.5, 20.8, 22.6, 25.7, 39.5, 52.3, 64.7, 68.1, 113.0, 118.6, 141.6, 146.1, 165.3 ppm. IR ($CHCl_3$): $\tilde{\nu}$ = 2953, 1759, 1646, 1540, 1462, 1386, 1253, 1066,

836 cm^{-1}. MS (EI): *m*/*z* = 344 [M – *t*Bu]$^+$. HRMS: calcd. for $C_{14}H_{22}NO_2SeSi$ 344.0585; found 400.0570.

(7*R*,8*S*)-4-(But-1-en-2-yl)-8-[(*R*)-1-(*tert*-butyldimethylsilyloxy)-ethyl]-1-aza-6-selenabicyclo[5.2.0]dec-3-en-9-one (10c): Yield 9.5 mg (46%). ^{1}H NMR ($CDCl_3$): δ = 0.06 (s, 3 H), 0.07 (s, 3 H), 0.86 (s, 9 H), 1.07 (t, *J* = 7.6 Hz, 3 H), 1.20 (d, *J* = 6.2 Hz, 3 H), 2.24–2.30 (q, *J* = 7.6 Hz, 2 H), 3.15 (dd, *J* = 1.4, 3.5 Hz, 1 H), 3.44 (d, *J* = 13.1 Hz, 1 H), 3.65 (d, *J* = 13.1 Hz, 1 H), 3.84 (dd, *J* = 6.1, 15.9 Hz, 1 H), 4.19–4.24 (m, 1 H), 4.30 (dd, *J* = 6.1, 15.9 Hz, 1 H), 5.02 (s, 1 H), 5.07 (d, *J* = 1.4, $^2J_{^{77}Se,^1H}$ = 19.2 Hz, 1 H), 5.11 (s, 1 H), 5.68 (t, *J* = 6.2 Hz, 1 H) ppm. ^{13}C NMR ($CDCl_3$): δ = –5.15, –4.22, 13.0, 17.8, 19.3, 22.6, 25.7, 26.5, 39.6, 48.8, 64.7, 68.1, 111.1, 118.1, 145.8, 148.6, 165.4 ppm. ^{77}Se NMR ($CDCl_3$): δ = 347.1 ppm. IR ($CHCl_3$): $\tilde{\nu}$ = 2955, 1760, 1630, 1462, 1385, 1253, 1065, 835 cm^{-1}. MS (EI): *m*/*z* = 358 [M – *t*Bu]$^+$. HRMS: calcd. for $C_{15}H_{24}NO_2SeSi$ 358.0742; found 358.0786.

(7*R*,8*S*)-8-[(*R*)-1-(*tert*-Butyldimethylsilyloxy)ethyl]-4-(hept-1-en-2-yl)-1-aza-6-selenabicyclo[5.2.0]dec-3-en-9-one (10d): Yield 13.5 mg (59%). ^{1}H NMR ($CDCl_3$): δ = 0.06 (s, 3 H), 0.07 (s, 3 H), 0.86 (s, 9 H), 0.89 (t, *J* = 6.9 Hz, 3 H), 1.20 (d, *J* = 6.3 Hz, 3 H), 1.24–1.37 (m, 4 H), 1.40–1.48 (m, 2 H), 2.23 (t, *J* = 7.4 Hz, 2 H), 3.15 (dd, *J* = 1.7, 3.4 Hz, 1 H), 3.42 (d, *J* = 13.2 Hz, 1 H), 3.64 (d, *J* = 13.2 Hz, 1 H), 3.83 (dd, *J* = 5.7, 16.0 Hz, 1 H), 4.18–4.24 (m, 1 H), 4.30 (dd, *J* = 5.7, 16.0 Hz, 1 H), 4.99 (s, 1 H), 5.06 (d, *J* = 1.7, $^2J_{^{77}Se,^1H}$ = 17.8 Hz, 1 H), 5.10 (s, 1 H), 5.67 (t, *J* = 6.3 Hz, 1 H) ppm. ^{13}C NMR ($CDCl_3$): δ = –5.16, –4.22, 14.1, 17.8, 19.3, 22.5, 22.6, 25.6, 28.1, 31.6, 33.8, 39.6, 48.7, 64.7, 68.0, 112.1, 118.2, 145.7, 147.3, 165.4 ppm. IR ($CHCl_3$): $\tilde{\nu}$ = 2954, 1760, 1626, 1599, 1462, 1375, 1254, 1066, 835 cm^{-1}. MS (EI): *m*/*z* = 400 [M – *t*Bu]$^+$. HRMS: calcd. for $C_{18}H_{30}NO_2SeSi$ 400.1211; found 400.1254.

Supporting Information (see also the footnote on the first page of this article): ^{1}H and ^{13}C NMR spectra of compounds are available.

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