Rapid Entry into Mono-, Bi-, and Tricyclic β-Lactam Arrays via Alkene Metathesis

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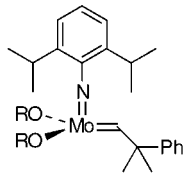
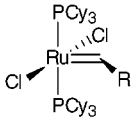
4-Acetoxy-2-azetidinone and (3*R*,4*R*)-4-acetoxy-3-[(1*R*)-1-(*tert*-butyldimethylsilyl)-oxyethyl]-2-azetidinone were converted into 4-alkenyloxy-, 4-(*N*-allyltoluene-4-sulfonamido)-, 4-(allylthio)-, and 4-alkenyl-2-azetidinone systems. In addition, 4-acetoxy-2-azetidinone and (3*R*,4*R*)-4-acetoxy-3-[(1*R*)-1-(*tert*-butyldimethylsilyl)-oxyethyl]-2-azetidinone were converted into β-lactam dienes *via* sequential C-4 substitution using unsaturated alcohols, allyl mercaptan, *N*-allyltoluene-4-sulfonamide, and allyl(chloro)dimethylsilane followed by *N*-allylation. Crossed metathesis of β-lactam alkenes with styrene partners and ring closing metathesis of β-lactam dienes using the Schrock $[(CF_3)_2MeCO]_2Mo(=CHCMe_2Ph)(=NC_6H_3\text{-}2,6\text{-}iso\text{-}Pr_2)$ (**1**) or Grubbs $Cl_2(Cy_3P)_2Ru{=}CHPh$ (**2**) carbenes gave diverse monocyclic and bicyclic β-lactam systems including derivatives of 1-azabicyclo-[4.2.0]octan-8-one, 1-azabicyclo[5.2.0]nonan-9-one and its 6-thia, 6-aza, and 6-oxa analogues, 7-oxa-1-azabicyclo[6.2.0]octan-10-one, 8-oxa-1-azabicyclo[7.2.0]octan-11-one, and 9-oxa-1-azabicyclo[8.2.0]octan-12-one. Ring-closing enyne metathesis and tandem ring-closing enyne and diene metathetic reactions were used to produce bicyclic β-lactam conjugated dienes as exemplified by the conversion of (3*S*,4*R*)-(−)-3-[(1*R*)-(*tert*-butyldimethylsilyl)oxyethyl]-1-(5-oxa-oct-7-en-2-yn-1-yl)-4-(2-propenyl)-azetidin-2-one (**83**) into (6*R*,7*S*)-(+)-7-[(1*R*)-(*tert*-butyldimethylsilyl)oxyethyl]-3-[(2,5-dihydro)-3-furanyl]-1-azabicyclo[4.2.0]oct-3-en-8-one (**98**).

The elaboration of carbon skeletons via the construction of carbon−carbon bonds continues to be one of the most important endeavors in synthetic organic chemistry.[1] Recently, ring-closing metathesis[2] has proved versatile in the synthesis of carbocyclic,[3] heterocyclic,[4] and macrocyclic[5] arrays. Also, enyne metathesis is finding increasing application,[6] in addition to the derivatization of alkenes by cross-metathesis.[7] This is the direct result of the introduction of the isolable, well-characterized, and highly active olefin metathesis catalysts **1**,[8] **2**,[9] and **3**.[10]



1 R = $C(CH_3)(CF_3)_2$



2 R = CH=CPh_2
3 R = Ph

We have recently described the use of olefin cross-metathesis in conjunction with allylboration to elaborate homoallylic alcohols[11] and additionally have described the use of metathesis to provide a wide range of mono-, bi-, and tricyclic β-lactam systems.[12−14] Herein we provide

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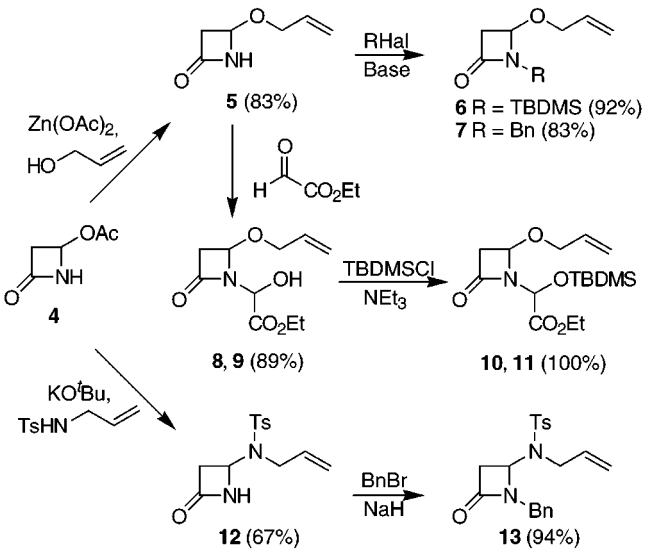
Scheme 1



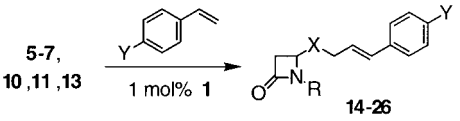
full details of our studies on β-lactam ring-closing and cross-metathesis reactions.

Results and Discussion

Cross-Metathesis. To begin our studies, we chose the simple alkene-containing lactam **5**,[15] readily available from acetate **4**, as the starting point. Suspecting that the unprotected amide may interfere with the molybdenum carbene catalyst **1**, we elected to protect this functionality. Thus, separate *N*-silylation and *N*-benzylation of **5** afforded β-lactams **6** and **7**, respectively (Scheme 1). Condensation of lactam **5** with ethyl glyoxalate followed by silylation of the resulting carbinolamines **8** and **9** yielded the highly functionalized, chromatographically separable epimers **10** and **11**. The analogous 4-aza system **12** was accessed from acetate **4** via base-mediated substitution with *N*-toluene-4-sulfonyl allylamine. Amide *N*-benzylation of lactam **12** passed without incident to provide adduct **13**.

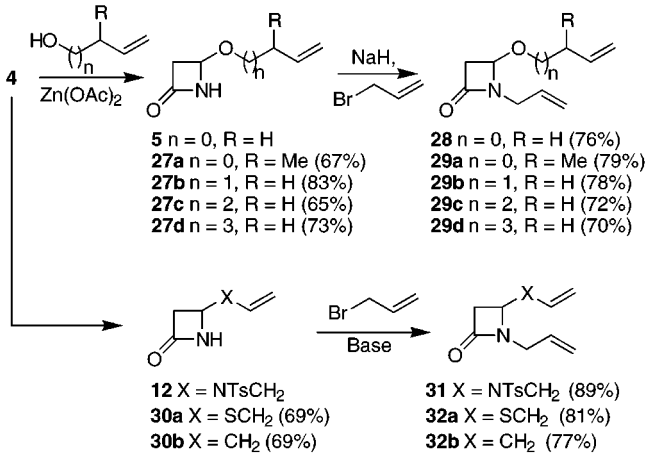
With a range of substrates in hand, we began our cross-metathesis investigations. When alkene **5** was exposed to 1 mol % of molybdenum catalyst **1** and excess styrene, a protocol described by Crowe et al.,[7a] no reaction was evident (Table 1, entry 1). In this case, addition of alkene **5** to the catalyst solution resulted in a rapid color change from the light yellow of the catalyst to a dark green color strongly indicative of catalyst decompostion. To our delight, treatment of the silylated derivative **6** under identical conditions afforded lactam **15** in 74% yield (entry 2) after a rapid solution color change to deep orange, thus confirming the need for amide protection. With this reaction, as with all cross-metatheses reported herein, only the *trans*-isomer was detected by ^{1}H NMR, in which presumably irreversible elimination of ethylene occurs from the *anti*- rather than the *syn*-substituted metallocycle.[7a] The other protected 4-oxa systems, **7**, **10**, and **11**, fared equally well with styrene (entries 6, 10, and 11), and *para*-substituted (*p*-MeO, *p*-Cl, *p*-Me) styrenes were found to perform as satisfactory cross-metathetic partners (entries 3−5 and 7−9). The doubly *N*-protected (Bn and Ts) 4-aza system **13** proved to be less amenable to successful cross-metathesis, and this may be due to the steric bulk of the tosyl grouping (entries 12 and 13). With these preliminary investigations complete and having shown the tolerance of the β-lactam backbone to molybdenum carbene **1**, we considered the possibility of performing ring-closing metathesis (RCM) on such a framework.

Table 1. Cross-Metathesis with Molybdenum Carbene 1



entry	substrate	X	R	Y	product	% yield
1	**5**	O	H	H	**14**	0
2	**6**	O	TBDMS	H	**15**	74
3	**6**	O	TBDMS	Cl	**16**	53
4	**6**	O	TBDMS	Me	**17**	66
5	**6**	O	TBDMS	OMe	**18**	52
6	**7**	O	Bn	H	**19**	81
7	**7**	O	Bn	Cl	**20**	42
8	**7**	O	Bn	Me	**21**	51
9	**7**	O	Bn	OMe	**22**	53
10	**10**	O	OTBDMS, CO_2Et	H	**23**	67
11	**11**	O	OTBDMS, CO_2Et	H	**24**	79
12	**13**	NTs	Bn	H	**25**	35
13	**13**	NTs	Bn	Cl	**26**	43

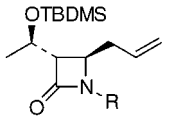
Scheme 2



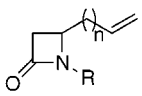
Ring-Closing Metathesis. Ring-closing metathesis of 1,4-bis(ene)-substituted azetidinones, exemplified by **28**, should lead to bicyclic β-lactams. It is clear that realization of such a process would allow synthetically rapid access to a wealth of novel, potentially biologically active, bicyclic β-lactam arrays.

The syntheses of precursor dienes were carried out via a number of distinct routes. 4-Alkoxy-2-azetidinones **28** and **29a**−**d** were prepared by the zinc acetate-catalyzed displacement of acetate **4** with the requisite unsaturated alcohol. All were obtained in consistently high yield following *N*-allylation using sodium hydride and allyl bromide (Scheme 2). 4-Allylamino, 4-allylthio, 4-allyl, and the homochiral-substituted lactams **31**, **32a**−**b**, and **34** were synthesized as previously described.[12−14] [2 + 2]-cycloaddition of chlorosulfonyl isocyanate with 1,3-butadiene[16] or 1,5-hexadiene[17] followed by *N*-alkylation (allyl bromide, crotyl bromide, or 4-bromo-1-butene) of the so-produced β-lactams furnished dienes **38a**−**c** and

(15) Basak, A.; Khamrai, U. *Synth. Commun.* **1994**, *24*, 131.



33 R = H
34 R = $CH_2CH{=}CH_2$



36 n = 0, R = H
37 n = 2, R = H
38a n = 0, R = $CH_2CH{=}CH_2$
38b n = 0, R = $CH_2CH{=}CHCH_3$
38c n = 0, R = $CH_2CH_2CH{=}CH_2$
39 n = 2, R = $CH_2CH{=}CH_2$

39. Because carboxy groups contiguous to the lactam nitrogen are a prerequisite for biological activity, we also prepared **41** (1:1 mixture of diastereoisomers), **44**, and **45** (Scheme 3). Condensation of **33** with ethyl glyoxalate furnished a separable mixture of diastereoisomeric carbinolamines. A single-crystal X-ray analysis allowed assignment of **42** as the (2*S*)-diastereoisomer and this information proved useful at a later point (vide infra). Allylation of either carbinolamine **42** or **43** resulted in partial epimerization at C1′ in both cases, affording inseparable mixtures of **44** and **45** (approximately 4:1 from **42** and 1:3 from **43**) which were used directly for the ring-closing metathesis experiments.

The ring-closing metathesis runs were performed using either the molybdenum catalyst **1** or the ruthenium catalyst **3**, depending on the requirements of the reaction. Cyclization of **28**, a seven-membered ring closure, to the homooxacephem **46** was achieved using 5 mol % of **1** in an excellent isolated yield of 84% (Table 2, entry 1), clearly demonstrating the synthetic utility of this method in these systems. Methyl-substituted oxacycle **47** was produced in only 60% yield under identical conditions (entry 2), presumably a result of increased steric interference at the reaction site.[18] Closure of bicyclic β-lactams with increasing ring sizes was possible, albeit progressively less successful. The eight-membered ring system **48** was produced in 52% yield using 5 mol % of **1** and in an improved 76% yield using 10 mol % of **1** (entries 3 and 4). The [7.2.0] system **49** was isolated in only 12% yield under the same conditions (entry 5) but could be optimized to 23% using 20 mol % of **1** at lower concentration (entry 6). The 10-membered ring containing lactam **50** was produced in 3% yield using 5 mol % of **1** (entry 7) and in 10% yield under high dilution and at higher catalyst loading (entry 8). Aza-diene **31** was cyclized in 36% yield using 5 mol % of **3** (entry 9), but the yield was vastly improved by employing **1**, which gave **51** in 92% yield (entry 10). When **32a** was treated with 5 mol % of **3**, a 22% yield of cyclized material **52** was isolated, and to our knowledge, this represents the only reported use of **3** for the cyclization of a sulfide (entry 11). The superior tolerance of **1** to sulfides is well documented,[2b] and the use of 5 mol % of molybdenum carbene **1** improved the yield for the ring-closing metathesis of **32a** to **52** to 79% (entry 12). 4-Allyl-substituted lactam **32b** smoothly afforded carbacephem **53** in 81% yield (entry 13), and homocarbacephem **54** was isolated in equally good yield after treatment of **39** with 5 mol % of **3** (entry 14).

Ring-closing metathesis of the *anti*-1,3,4-substituted azetidinone **34** with ruthenium carbene **3** (5 mol %) proceeded smoothly to **55**, demonstrating that *anti*-3-substitution does not interfere with reaction between dienes at the 1 and 4 positions. However, attempted ring-closing metathesis using either carbene **1** or **3** on the 4-vinyl-substituted substrates **38a**−**c** met with no success. Intrigued by the failure of these compounds to undergo cyclization we reverted to cross-metathesis to probe their reactivity. Thus, vinyl lactam **36** and allyl and homoallyl lactams **30b** and **37** were routinely *N*-methylated and/or *N*-benzylated to provide subtrates **58**−**62** in good yields. Exposure of alkenes **58**−**61** to the standard cross-metathetic conditions, using styrene, afforded none of the desired adducts. Interestingly, the

Scheme 3

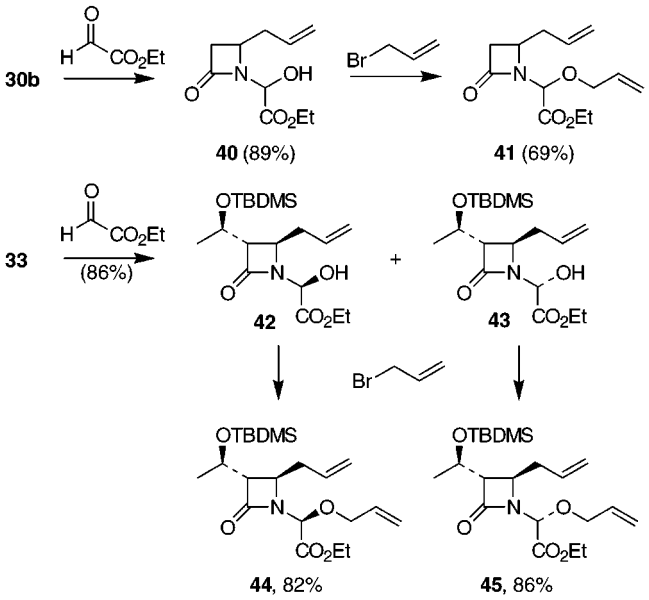
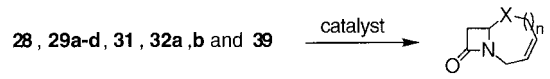
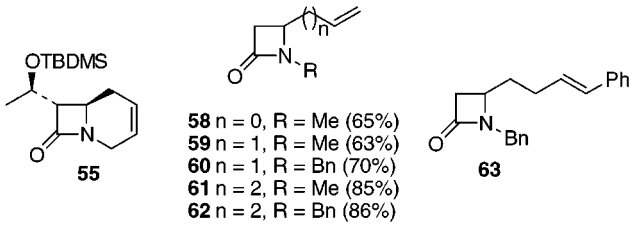


Table 2. Ring-Closing Metathesis on the β-Lactam Framework



entry	substrate	catalyst (%)	X	n	product	% yield
1	**28**	**1** (5)	O	1	**46**	84
2	**29a**	**1** (5)	−OCHMe−	0	**47**	60
3	**29b**	**1** (5)	O	2	**48**	52
4	**29b**	**1** (10)	O	2	**48**	76
5	**29c**	**1** (5)	O	3	**49**	12
6	**29c**	**1** (20)	O	3	**49**	23
7	**29d**	**1** (5)	O	4	**50**	3
8	**29d**	**1** (20)	O	4	**50**	10
9	**31**	**3** (5)	NTs	1	**51**	36
10	**31**	**1** (5)	NTs	1	**51**	92
11	**32a**	**3** (5)	S	1	**52**	22
12	**32a**	**1** (5)	S	1	**52**	79
13	**32b**	**3** (5)	CH_2	0	**53**	81
14	**39**	**3** (5)	CH_2	1	**54**	83



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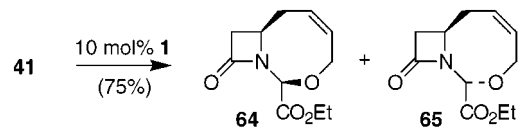
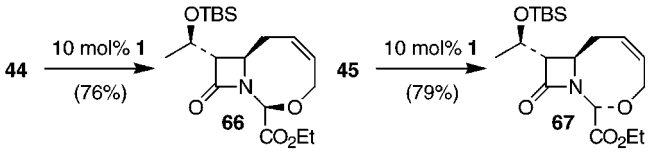
(18) For a thorough investigation of such effects, see: Kirkland, T. A.; Grubbs, R. H. *J. Org. Chem.* **1997**, *62*, 7310.

Scheme 4



Scheme 5



N-benzylated substrate **62** was found to undergo rapid cross-metathesis under identical conditions, and the *trans* double bond adduct **63** was isolated in 72% yield. The sharp contrast in reactivity between **61** and **62** may originate from low N-1 steric congestion in **61**, and thus the amide nitrogen lone pair is able to complex the catalyst metal center with consequent deactivation. Even after allowance for this protecting group phenomenom, the lack of reactivity of **59** and **60** is particularly curious when it is considered that 4-allyl-substituted dienes **32b** and **34** underwent smooth ring-closing metathesis.

Diene **41**, as a 1:1 mix of diastereoisomers, when treated with 10 mol % of carbene **1**, produced the cyclized compounds **64** and **65** as separable diastereoisomers in high yield (Scheme 4). The relative positions in the ^{1}H NMR spectra of the protons α to the ester provided the necessary information to assign their structures by comparison to literature examples.[19] Homochiral dienes **44** and **45** were found to undergo cyclization at different rates. Treatment of epimer **44** with 10 mol % of **1** gave a 76% yield of **66** in 15 h, whereas epimer **45** required a prolonged reaction time of 60 h to give a comparable yield of **67** (Scheme 5). Inspection of their relative structures (elucidated by X-ray crystallography, vide supra) reveals that although isomer **44** can undergo ring-closing metathesis without significant interference from the stereoproximate ester functionality, in diene **45** the ester grouping is directed "into" the incipient ring, thus retarding cyclization (Figure 1).

In addition to the purposely synthesized ring-closing metathesis substrates, one extra diene was produced. During the synthesis of *β*-lactam **5**, a small quantity of primary amide **68** could be isolated. Treatment of this amide with 5 mol % of carbene **3** produced the cyclic acetal **69** in 81% yield (Scheme 6). To the best of our knowledge, this is the only reported example of an RCM reaction using **3** in the presence of a primary amide.

Enyne Metathesis. The ruthenium carbene **3**-catalyzed metathesis of enynes is attracting increasing attention and is proving to be as facile as the ring-closing metathesis of dienes.[6] Enyne metathesis has the added advantage of delivering a conjugated diene as the final adduct, allowing for Diels−Alder elaboration if required.[20] In designing enyne metathesis substrates, we were guided by a number of considerations. It has previously been noted that terminal acetylenes afford only poor yields in enyne metathesis,[6d] and with the exception of

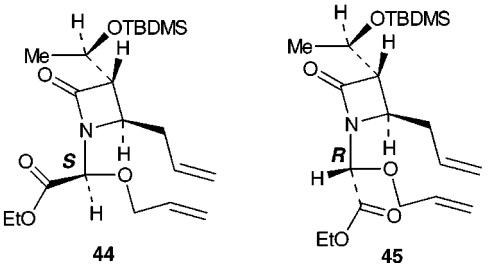
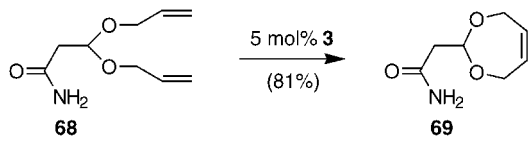
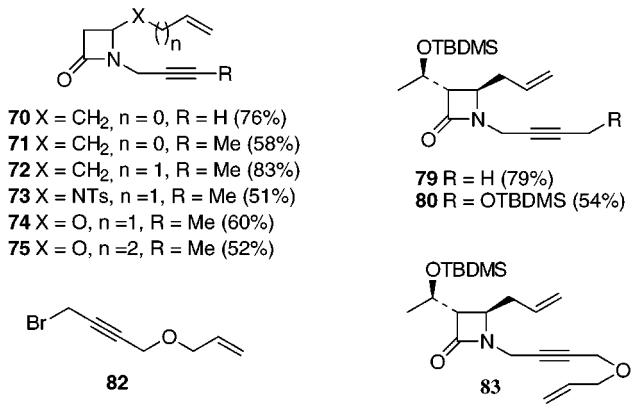


Figure 1.

Scheme 6



70, all of the remaining substrates were prepared as "methyl-capped" acetylenes (vide infra). Second, we noted that within a given framework, switching the relative sites of the tethered ene and acetylene moieties lead to isomeric ring-closed adducts. Accordingly, two sets of substrates were synthesized. *N*-Tethered acetylenic species **70**−**75** and **79**−**80** were prepared from



previously assembled materials (**5**, **10**, **27c**, **30b**, **37,** and **33**, respectively) via reaction with potassium hydroxide and a propargylic bromide.[21] The acetylenes **77a**−**d** and **78a**−**b** were derived from the zinc acetate-catalyzed displacement of acetate **4** with propargylic alcohols followed by *N*-allylation, *N*-silylation, or *N*-benzylation in the usual manner (Scheme 7). Additionally, we produced dienyne **83** from **33** via *N*-alkylation with the novel propargylic bromide **82**.

All of the enyne metathesis runs were performed with a ruthenium-based catalyst, in line with the literature.[6] Treatment of the parent *N*-tethered propargylic enyne **70** with up to 10 mol % of **3** afforded none of the desired cyclized material **84** (Table 3, entry 1) as expected.[6d] In contrast, we were pleased to find that an optimized yield of 88% was attainable for the cyclization of enyne **71** through the use of 10 mol % of **3** (entry 2). Cyclization to the [5.2.0] system **86** was achieved in comparable yield (Table 3, entry 3), and similarly excellent yields were obtained for the seven-membered ring closures using the aza- and oxa-cycles **87** and **88** (entries 4 and 5). A dramatic decrease in yield was observed for the cycliza-

(19) (a) Stoodley, R. J.; Whitehouse, N. R. *J. Chem. Soc., Perkin Trans. 1* **1973**, 32. (b) Brown, A. G.; Corbett, D. F.; Howarth, T. T. *Chem. Commun.* **1977**, 359.

(20) Kotha, S.; Sreenivasachary, N.; Brahmachary, E. *Tetrahedron Lett.* **1998**, *39*, 2806.

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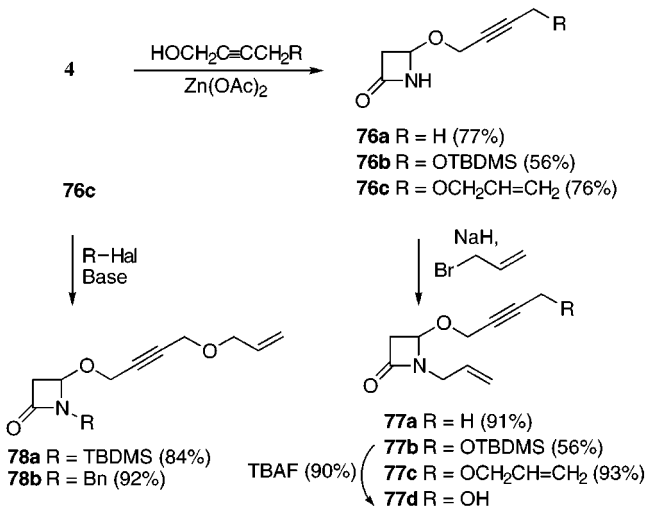
Scheme 7



tion of **75** generating an eight-membered ring (entry 6). 4-Azetidinone-tethered acetylenes perfomed equally well under the same conditions: oxacycles **90** and **91** were produced in high yield (entries 7 and 8), but hydroxy groups evidently require protection (entry 9). The enynes **79** and **80** closed cleanly to carbacycles **93** and **94** (entries 10 and 11), and equally high yields were observed for the "external" cyclization of enynes **78a** and **78b** to lactams **95** and **96** (entries 12 and 13). Pleasingly, dienyne metatheses with **77c** and **83** was also possible, although lower substrate concentrations were required, affording the tricyclics **97** and **98** in 64% (entry 14) and 67% yield (entry 15), respectively.

It is clear from the above examples that alkene metathesis is very tolerant of the common β-lactam functionality, and the ring closure reaction may well prove to be a general strategy for the production of bi- and tricyclic arrays. With a view to the production of novel, potentially active β-lactam antibiotics, introduction of a carboxylic acid residue contiguous to the lactam amide functionality prior to ring closure is currently being explored.[22] These results will be reported in due course.

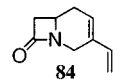
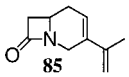
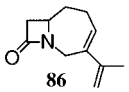
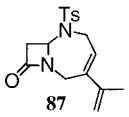
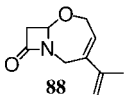
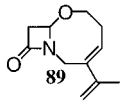
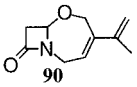
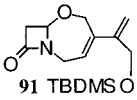
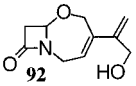
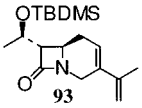
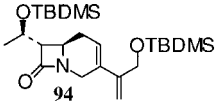
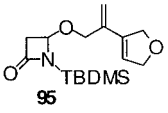
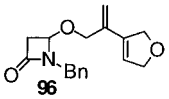
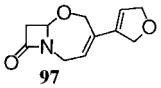
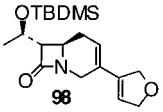
Experimental Section

General. All reactions, except those using molybdenum catalyst **1**, were run in oven-dried glassware under a nitrogen atmosphere. Reactions using molybdenum catalyst **1** were performed in a glovebox under a nitrogen atmosphere. THF and Et_2O were distilled from Na/Ph_2CO. CH_2Cl_2, DMF, and NEt_3 were distilled from CaH_2.

Column chromatography was performed on BDH silica gel 60, 230−400 mesh ASTM. Concentrations were performed in vacuo. Analytical thin-layer chromatography (TLC) was performed on precoated glass-backed plates (Merck Kieselgel 60 F_{254}) and visualized with ultraviolet light (254 nm) or potassium permanganate, as appropriate.

Molybdenum carbene **1**,[8] ruthenium carbene **3**,[10] 4-(2-propenyl-1-oxy)azetidin-2-one (**5**),[15] 4-vinylazetidin-2-one (**36**),[16] 4-(3-butenyl)azetidin-2-one (**37**),[17] *N*-(4-toluenesulfonyl)allylamine,[23] 4-(2-propenoxy)-2-butyn-1-ol,[24] and 1-(*tert*-butyldimethylsilyl)oxy-2-butyn-4-ol[25] were synthesized according to literature procedure, with minor modifications where necessary.

Table 3. Enyne Metathesis with Ruthenium Carbene 3

Entry	Substrate	mol% **3**	Product	%Yield
1	**70**	1	 **84**	0
		10		0
2	**71**	1	 **85**	20
		5		52
		10		88
3	**72**	10	 **86**	82
4	**73**	10	 **87**	70
5	**74**	10	 **88**	88
6	**75**	10	 **89**	12
7	**77a**	10	 **90**	74
8	**77b**	10	 **91** TBDMS O	82
9	**77d**	10	 **92** HO	32
10	**79**	10	 **93** OTBDMS	100
11	**80**	15	 **94** OTBDMS OTBDMS	72
12	**78a**	10	 **95** TBDMS	100
13	**78b**	10	 **96** Bn	98
14	**77c**	5	 **97**	46
		10		64
15	**83**	10	 **98** OTBDMS	67

(22) Ahmed, M.; Barrett, A. G. M.; Baker, S. P.; Baugh, S. P. D.; Braddock, D. C.; Procopiou, P. A. To be submitted for publication.
(23) Campbell, M. M.; Connarty, B. P. *Heterocycles* **1982**, *19*, 1853.
(24) Fletcher, R. J.; Lampard, C.; Murphy, J. A.; Lewis, N. *J. Chem. Soc., Perkin Trans. 1* **1995**, 623.
(25) Najdi, S. D.; Olmstead, M. M.; Schore, N. E. *J. Organomet. Chem.* **1992**, 431, 335−358.

Table 4. Crystal Data, Data Collection, and Refinement Parameters[a]

	42	**51**	**69**
formula	$C_{18}H_{33}NO_5Si$	$C_{14}H_{16}N_2O_3S$	$C_7H_{11}NO_3$
formula weight	371.5	292.4	157.2
color, habit	clear blocky needles	clear plates	clear platy needles
crystal size, mm	0.70 × 0.70 × 0.40	0.50 × 0.40 × 0.17	0.93 × 0.20 × 0.07
crystal system	orthorhombic	monoclinic	triclinic
space group	$P2_12_12_1$ (No. 19)	$P2_1/c$ (No. 14)	$P\bar{1}$ (No. 2)
cell dimensions			
a, Å	7.842(2)	16.111(3)	4.957(1)
b, Å	14.064(1)	8.026(1)	9.147(1)
c, Å	20.016(2)	10.852(4)	10.103(2)
α, deg			112.29(1)
β, deg		90.52(2)	94.99(2)
γ, deg			103.15(1)
V, Å^3	2207.7(6)	1403.1(7)	405.1(1)
Z	4	4	2
d_c, g cm^{-3}	1.118	1.384	1.289
F(000)	808	616	168
radiation used	Cu-Kα[b]	Cu-Kα	Cu-Kα[b]
μ, mm^{-1}	1.14	2.14	0.85
θ range, deg	3.8−63.5	2.7−63.8	4.8−63.9
no. of unique reflctns			
measd	2080	2329	1304
obsd, $\|F_o\| >$	1878	1653	1100
$4\sigma(\|F_o\|)$ no. of varbls	247	182	109
R_1[c]	0.051	0.064	0.043
wR_2[d]	0.140	0.167	0.106
weighting factors *a*, *b*[e]	0.083, 0.412	0.113, 0.222	0.046, 0.073
largest diff. peak, hole, e Å^{-3}	0.15, −0.20	0.32, −0.47	0.10, −0.15

[a] Details in common: graphite-monochromated radiation, *ω*-scans, Siemens P4 diffractometer, 293 K, refinement based on F^2. [b] Rotating anode source. [c] $R = \sum||F_o| - |F_c||/\sum|F_o|$. [d] $wR_2 = [\sum w(F_o^2 - F_c^2)^2/\sum w(F_o^2)^2]^{1/2}$. [e] $w^{-1} = \sigma^2(F_o^2) + (aP)^2 + bP$.

X-ray Crystallography. Table 4 provides a summary of the crystal data, data collection, and refinement parameters for **42**, **51**, and **69**. The structures were solved by direct methods and were refined by full matrix least-squares based on F^2. In **42**, disorder was found in the $SiMe_3$ and C═C units. In each case, this was resolved into two partial occupancy orientations, and the non-hydrogen atoms of the major occupancy were refined anisotropically. The remaining non-hydrogen atoms in all three structures were refined anisotropically. The O−H hydrogen atom in **42** and the N−H hydrogen atoms in **69** were found from Δ*F* maps and refined isotropically subject to an X−H distance constraint (0.90 Å). The methyl hydrogen atoms in **51** were located from a Δ*F* map; optimized; assigned isotropic thermal parameters, $U(H) = 1.5U_{eq}(C-Me)$; and allowed to ride on their parent atoms. The remaining C−H hydrogen atoms in all three structures were placed in calculated positions; assigned isotropic thermal parameters, $U(H) = 1.2U_{eq}(C)$; and allowed to ride on their parent atoms. The absolute chirality of **42** was determined by *R*-factor tests ($R_1^+ = 0.051$, $R_1^- = 0.052$) and by use of the Flack parameter ($x^+ = 0.04(8)$, $x^- = 0.96(8)$). Computations were carried out using the SHELXTL PC program system.[26]

The crystallographic data (excluding structure factors) for the structures reported in Table 4 have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication numbers CCDC-103212, CCDC-103213, and CCDC-103214 for **42**, **51**, and **69**, respectively. Copies of the data can be obtained free of charge on application to The Director, CCDC, 12 Union Road, Cambridge CB12 1EZ, U.K. (fax: int.code+(1223)336-033. e-mail: teched@chemcrys.cam.ac.uk.)

1-*tert*-Butyldimethylsilyl-4-(2-propenyl-1-oxy)azetidin-2-one (6). To an ice-cooled solution of **5** (50 mg, 0.39 mmol) and Et_3N (60 mg, 0.55 mmol) in CH_2Cl_2 (2 mL) was added *tert*-butyldimethylsilyl chloride (71 mg, 0.47 mmol). The mixture was stirred for 3 h, diluted with Et_2O (10 mL), washed with water (3 × 10 mL), dried ($MgSO_4$), and concentrated, and the resulting oil was chromatographed (3:7 Et_2O/hexanes) to give **6** as a colorless oil (84 mg, 92%): TLC R_f 0.32 (1:1 Et_2O/hexanes); IR (film) 1757 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) *δ* 5.85−5.72 (m, 1H), 5.18 (br d, *J* = 17.0 Hz, 1H), 5.09 (br d, *J* = 11.5 Hz, 1H) 4.86 (m, 1H), 3.89 (m, 2H), 3.00 (dd, *J* = 3.5, 15.5 Hz, 1H), 2.75 (dd, *J* = 1.5, 15.5 Hz, 1H), 0.85 (s, 9H), 0.14 (s, 3H), 0.13 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) *δ* 170.8, 133.8, 117.2, 79.3, 67.5, 46.0, 26.1, 18.2, −5.8, −6.0; MS (CI) *m*/*z* 259 $(M + NH_4)^+$, 242 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{12}H_{24}NO_2Si$ $(M + H)^+$ 242.1576, found $(M + H)^+$ 242.1574.

1-Benzyl-4-(2-propenyl-1-oxy)azetidin-2-one (7). To an ice-cooled solution of **5** (0.10 g, 0.79 mmol) in DMF (5 mL) was added NaH (0.035 g as a 60% dispersion in oil, 0.87 mmol). The mixture was stirred for 1 min, and benzyl bromide (0.10 mL, 0.87 mmol) was added in one portion. After 4 h, the mixture was diluted with Et_2O (20 mL), washed with water (3 × 20 mL), dried ($MgSO_4$), and concentrated. The residue was chromatographed (4:1 Et_2O/hexanes) to produce **7** as a colorless oil (0.14 g, 83%): TLC R_f 0.63 (Et_2O); IR (film) 1759 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) *δ* 7.30−7.20 (m, 5H), 5.78−5.66 (m, 1H), 5.16−5.06 (m, 2H), 4.96−4.84 (m, 1H), 4.60 (d, *J* = 15.5 Hz, 1H), 4.21 (d, *J* = 15.5 Hz, 1H), 3.90−3.82 (m, 2H), 3.01 (dd, *J* = 4.0, 15.0 Hz, 1H), 2.82 (dd, *J* = 1.0, 15.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) *δ* 166.1, 135.8, 133.6, 128.9, 128.3, 127.8, 117.7, 80.8, 68.6, 44.8, 44.5; MS (CI) *m*/*z* 235 $(M + NH_4)^+$, 218 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{13}H_{16}NO_2$ $(M + H)^+$ 218.1181, found $(M + H)^+$ 218.1189. Anal. Calcd for $C_{13}H_{15}NO_2$: C, 71.87; H, 6.96; N, 6.45. Found: C, 71.76; H, 6.71; N, 6.69.

Ethyl 2-Hydroxy-2-[2-oxo-4-(2-propenyl-1-oxy)azetidinyl]acetate (8 and 9). To a solution of ethyl glyoxalate (50% solution in PhMe, 0.82 mL, 4.10 mmol) in PhMe (50 mL) was added **5** (0.50 g, 3.93 mmol), and the solution was heated to reflux for 4 h. Ethyl glyoxalate (50% solution in PhMe, 0.41 mL, 2.05 mmol) was added, and the solution was heated to reflux for a further 15 h. The solution was allowed to cool and concentrated, and the yellow oil was chromatographed (1:1 Et_2O/hexanes) to give first **8** and then **9** as a colorless oils. **8** (0.69 g, 78%): TLC R_f 0.61 (Et_2O); IR (film) 3380, 1741 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) *δ* 5.99−5.86 (m, 1H), 5.37−5.23 (m, 4H), 4.34 (q, *J* = 7.0 Hz, 2H), 4.31−4.11 (m, 3H), 3.13 (dd, *J* = 4.0, 15.5 Hz, 1H), 2.96 (dd, *J* = 1.5, 15.5 Hz, 1H), 1.35 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) *δ* 168.4, 165.4, 133.5, 117.9, 80.7, 71.8, 68.9, 63.6, 44.9, 14.1; MS (CI) *m*/*z* 247 $(M + NH_4)^+$, 230 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{10}H_{16}$-

(26) SHELXTL PC version 5.03; Siemens Analytical X-ray Instruments, Inc., Madison, WI, 1994.

NO_5 $(M + H)^+$ 230.1029, found $(M + H)^+$ 230.1037. **9**: TLC R_f 0.45 (Et_2O); IR (film) 3412, 1747 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 5.93−5.81 (m, 1H), 5.43−5.21 (m, 4H), 4.40−4.26 (m, 2H), 4.11−4.01 (m, 3H), 3.11 (dd, J = 4.0, 15.0 Hz, 1H), 2.95 (d, J = 15.0 Hz, 1H), 1.34 (t, J = 7.0 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.6, 165.6, 133.5, 117.6, 79.2, 70.7, 69.0, 63.1, 44.6, 14.9; MS (CI) m/z 247 $(M + NH_4)^+$, 230 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{10}H_{16}NO_5$ $(M + H)^+$ 230.1029, found $(M + H)^+$ 230.1019.

Ethyl 2-(*tert*-Butyldimethylsilyl)oxy-2-[2-oxo-4-(2-propenyl-1-oxy)azetidinyl]acetate (10). To an ice-cooled solution of α-hydroxy ester **8** (0.20 g, 0.87 mmol) in CH_2Cl_2 (5 mL) was added 4-(dimethylamino)pyridine (DMAP, 120 mg, 0.96 mmol), followed by *tert*-butyldimethylsilyl chloride (0.66 g, 4.36 mmol) in one portion. The mixture was allowed to warm to room temperature and was stirred for 48 h. The solution was diluted with Et_2O (50 mL), washed with water (3 × 20 mL), dried ($MgSO_4$) and concentrated. The resulting oil was chromatographed (3:7 Et_2O/hexanes) to produce **10** as a colorless oil (0.28 g, 100%): TLC R_f 0.52 (1:1 Et_2O/hexanes); IR (film) 1778, 1758 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 5.90−5.78 (m, 1H), 5.55 (s, 1H), 5.31−5.13 (m, 3H), 4.37−4.29 (m, 1H), 4.20 (q, J = 7.0 Hz, 2H), 4.07−3.99 (m, 1H), 3.13 (dd, J = 4.5, 15.0 Hz, 1H), 2.85 (dd, J = 1.5, 15.0 Hz, 1H), 1.27 (t, J = 7.0, 3H), 0.88 (s, 9H), 0.15 (s, 3H), 0.12 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.7, 165.9, 133.7, 117.3, 81.7, 71.9, 70.2, 61.8, 45.1, 25.5, 18.1, 14.1, −5.0, −5.5; MS (CI, NH_3) m/z 361 $(M + NH_4^+)$, 344 $(M + H^+)$; HRMS (CI) calcd for $C_{16}H_{30}NO_5Si$ $(M + H)^+$ 344.1893, found $(M + H)^+$ 344.1896.

Ethyl 2-(*tert*-Butyldimethylsilyl)oxy-2-[2-oxo-4-(2-propenyl-1-oxy)azetidinyl]acetate (11). Proceeding from **9** in a manner analogous to that employing **8** furnished **11** as a colorless oil (100%): R_f 0.51 (1:1 Et_2O/hexanes); IR (film) 1777, 1752 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 5.81−5.77 (m, 1H), 5.49 (s, 1H), 5.27−5.12 (m, 3H), 4.17 (q, J = 7.0 Hz, 2H), 4.11 (dd, J = 5.0, 13.0 Hz, 1H), 3.99 (dd, J = 6.0, 7.0 Hz, 1H), 3.01 (dd, J = 4.0, 15.0 Hz, 1H), 2.90 (dd, J = 2.0, 15.0 Hz, 1H), 1.24 (t, J = 7.0 Hz, 3H), 0.88 (s, 9H), 0.11 (s, 3H), 0.07 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.0, 165.3, 133.7, 117.3, 78.7, 71.7, 69.4, 61.8, 44.4, 25.5, 18.1, 14.0, −5.1, −5.3; MS (CI, NH_3) m/z 361 $(M + NH_4)^+$, 344 $(M + H)^+$; HRMS (CI) calcd for $C_{16}H_{30}NO_5Si$ $(M + H)^+$ 344.1893, found $(M + H)^+$ 344.1896.

4-[*N*-(2-Propenyl)-4-toluenesulfonamido]azetidin-2-one (12). To a solution of lactam **4** (0.10 g, 0.77 mmol) in MeCN (10 mL) was added *N*-(4-toluenesulfonyl)allylamine (0.18 g, 0.85 mmol), 18-crown-6 (0.02 g, 0.077 mmol), and potassium *tert*-butoxide (0.10 g, 0.85 mmol). The reaction was stirred for 30 min, diluted with Et_2O (100 mL), washed with water (3 × 50 mL), dried ($MgSO_4$), and concentrated. The resulting oil was chromatographed (3:2 Et_2O/hexanes) to produce **12** as a white solid (0.14 g, 67%): mp 109 °C; TLC R_f 0.55 (Et_2O); IR (KBr) 3332, 1772 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 7.69 (d, J = 8.0 Hz, 2H), 7.33 (d, J = 8.0 Hz, 2H), 5.95−5.75 (m, 2H), 5.68 (dd, J = 2.0, 5.0 Hz, 1H), 5.31−5.14 (m, 2H), 3.96−3.84 (m, 1H), 3.77−3.66 (m, 1H), 3.15 (ddd, J = 3.0, 4.0, 15.0 Hz, 1H), 3.00 (dd, J = 3.0, 15.0 Hz, 1H), 2.43 (s, 3H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 165.6, 144.4, 136.4, 135.0, 130.2, 126.9, 117.5, 60.3, 44.2, 43.8, 21.6; MS (CI, NH_3) m/z 298 $(M + NH_4)^+$, 281 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{13}H_{17}N_2O_3S$ $(M + H)^+$ 281.0959, found $(M + H)^+$ 281.0954. Anal. Calcd for $C_{13}H_{16}N_2O_3S$: C, 55.70; H, 5.75; N 9.99. Found: C, 56.00; H, 5.88; N, 9.88.

1-Benzyl-4-[*N*-(2-propenyl)-4-toluenesulfonamido]azetidin-2-one (13). From **12**, the prodedure employed for **7** afforded **13** as a viscous oil (94%): TLC R_f 0.68 (Et_2O); IR (film) 1764 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 7.65−7.18 (m, 9H), 5.79−5.69 (m, 1H), 5.54 (dd, J = 2.0, 5.0 Hz, 1H), 5.18−5.09 (m, 2H), 4.43 (d, J = 15.5 Hz, 1H), 3.87 (d, J = 15.5 Hz, 1H), 3.75−3.68 (m, 2H), 3.03 (dd, J = 4.5, 15.5 Hz, 1H), 2.91 (dd, J = 2.0, 15.5 Hz, 1H), 2.43 (s, 3H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 165.3, 144.0, 137.1, 135.6, 134.4, 129.9, 128.7, 128.4, 127.8, 127.0, 118.0, 63.6, 44.8, 44.4, 42.4, 21.5; MS (CI, NH_3) m/z 388 $(M + NH_4)^+$, 371 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{20}H_{23}N_2O_3S$ $(M + H)^+$ 371.1429, found $(M + H)^+$ 371.1422. Anal. Calcd for $C_{20}H_{22}N_2O_3S$: C, 64.84; H, 5.99; N, 7.56. Found: C, 64.85; H, 5.82; N, 7.53.

General Procedure for the Cross-Metathesis of β-lactams with Styrenes Using Molybdenum Carbene 1 Furnishing 15−26. Substrates **5**−**7**, **10**, **11**, or **13** (0.21 mmol) were added to a stirred solution of the appropriate styrene (0.83 mmol) (Table 1) and molybdenum carbene (1.6 mg, 0.0021 mmol) in CH_2Cl_2 (1 mL). After 6 h, the solution was exposed to air, the dark solution was concentrated, and the crude oil was chromatographed.

1-(*tert*-Butyldimethylsilyl)-4-[3-(*E*)-phenyl-2-propenyl-1-oxy]azetidin-2-one (15): colorless oil (48 mg, 74%); TLC R_f 0.54 (1:1 Et_2O/hexanes); IR (film) 1755 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.42−7.28 (m, 5H), 6.62 (d, J = 15.0 Hz, 1H), 6.31−6.22 (m, 1H), 5.06−5.04 (m, 1H), 4.18 (d, J = 6.0 Hz, 2H), 3.13 (dd, J = 4.0, 15.0 Hz, 1H), 2.93 (dd, J = 1.0, 15.0 Hz, 1H), 0.98 (s, 9H), 0.27 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.8, 136.4, 132.6, 128.7, 127.9, 126.5, 125.0, 79.4, 67.2, 46.1, 26.1, 18.2, −5.7, −6.0; MS (CI, NH_3) m/z 335 $(M + NH_4)^+$, 318 $(M + H)^+$; HRMS (CI) calcd for $C_{18}H_{28}NO_2Si$ $(M + H)^+$ 318.1889, found $(M + H)^+$ 318.1884. Anal. Calcd for $C_{18}H_{27}NO_2Si$: C, 68.10; H, 8.58; N, 4.41. Found: C, 68.33; H, 8.43; N, 4.61.

1-(*tert*-Butyldimethylsilyl)-4-[3-(*E*)-(4-chlorophenyl)-2-propenyl-1-oxy]azetidin-2-one (16): colorless oil (53%); TLC R_f 0.26 (1:1 Et_2O/hexanes); IR (film) 1755 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 7.29 (s, 4H), 6.57 (d, J = 16.0 Hz, 1H), 6.25−6.19 (m, 1H), 5.02 (dd, J = 1.5, 4.0 Hz, 1H), 4.16 (dd, J = 1.5, 6.0 Hz, 2H), 3.14 (dd, J = 4.0, 15.5 Hz, 1H), 2.93 (dd, J = 1.5, 15.5 Hz, 1H), 0.98 (s, 9H), 0.27 (s, 3H), 0.26 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.6, 134.9, 133.6, 131.2, 128.8, 127.7, 125.7, 79.5, 66.9, 46.1, 26.1, 18.2, −5.7, −6.0; MS (CI, NH_3) m/z 352 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{18}H_{27}NO_2SiCl$ $(M + H)^+$ 352.1500, found $(M + H)^+$ 352.1502.

1-(*tert*-Butyldimethylsilyl)-4-[3-(*E*)-(4-methylphenyl)-2-propenyl-1-oxy]azetidin-2-one (17): colorless oil (66%); TLC R_f 0.54 (1:1 Et_2O/hexanes); IR (film) 1756 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 7.29−7.25 (m, 2H), 7.15−7.12 (m, 2H), 6.57 (d, J = 16.0 Hz, 1H), 6.23−6.17 (m, 1H), 5.02 (dd, J = 1.5, 4.0 Hz, 1H), 4.17 (dd, J = 1.5, 6.0 Hz, 2H), 3.15 (dd, J = 4.0, 15.5 Hz, 1H), 2.93 (dd, J = 1.5, 15.5 Hz, 1H), 2.35 (s, 3H), 0.99 (s, 9H), 0.28 (s, 3H), 0.27 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.8, 137.8, 133.6, 132.7, 129.4, 126.4, 123.9, 79.3, 67.4, 46.2, 26.1, 21.2, 18.2, −5.7, −6.0; MS (CI, NH_3) m/z 332 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{19}H_{30}NO_2Si$ $(M + H)^+$ 332.2045, found $(M + H)^+$ 332.2045.

1-(*tert*-Butyldimethylsilyl)-4-[3-(*E*)-(4-methoxyphenyl)-2-propenyl-1-oxy]azetidin-2-one (18): colorless oil (52%); TLC R_f 0.27 (1:1 Et_2O/hexanes); IR (film) 1755 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 7.32−7.26 (m, 2H), 6.88−6.84 (m, 2H), 6.56 (d, J = 16.0 Hz, 1H), 6.14−6.08 (m, 1H), 5.01 (dd, J = 1.5, 4.0 Hz, 1H), 4.15 (dd, J = 1.5, 6.0 Hz, 2H), 3.81 (s, 3H), 3.14 (dd, J = 4.0, 15.5 Hz, 1H), 2.93 (dd, J = 1.5, 15.5 Hz, 1H), 0.97 (s, 9H), 0.27 (s, 3H), 0.26 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.8, 159.5, 132.4, 129.2, 127.7, 122.7, 114.1, 79.3, 67.5, 55.3, 46.2, 26.1, 18.2, −5.7, −6.0; MS (CI, NH_3) m/z 348 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{19}H_{30}NO_3Si$ $(M + H)^+$ 348.1995, found $(M + H)^+$ 348.1989.

1-Benzyl-4-[3-(*E*)-phenyl-2-propenyl-1-oxy]azetidin-2-one (19): colorless oil (81%); TLC R_f 0.53 (Et_2O); IR (film) 1760 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.39−7.25 (m, 10H), 6.45 (dt, J = 1.5, 16.0 Hz, 1H), 6.11 (dt, J = 6.0, 16.0 Hz, 1H), 4.98 (dd, J = 4.0, 6.0 Hz, 1H), 4.60 (d, J = 15.5 Hz, 1H), 4.26 (d, J = 15.5 Hz, 1H), 4.11−4.06 (m, 2H), 3.08 (dd, J = 4.0, 15.0 Hz, 1H), 2.93 (d, J = 15.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.0, 136.2, 135.8, 133.2, 128.9, 128.6, 128.3, 128.0, 127.8, 126.6, 124.6, 80.7, 68.4, 44.8, 44.6; MS (CI) m/z 311 $(M + NH_4)^+$, 294 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{19}H_{20}NO_2$ $(M + H)^+$ 294.1494, found $(M + H)^+$ 294.1495. Anal. Calcd for $C_{19}H_{19}NO_2$: C, 77.79; H, 6.53; N, 4.77. Found: C, 77.51; H, 6.45; N, 5.05.

1-Benzyl-4-[3-(*E*)-(4-chlorophenyl)-2-propenyl-1-oxy]-azetidin-2-one (20): colorless oil (42%); TLC R_f 0.47 (Et_2O); IR (film) 1760 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 7.31−7.15 (m, 9H), 6.28 (d, J = 16.0 Hz, 1H), 6.05−5.96 (m, 1H), 4.91

(dd, J = 1.5, 4.0 Hz, 1H), 4.53 (d, J = 15.0 Hz, 1H), 4.18 (d, J = 15.5 Hz, 1H), 4.00−3.95 (m, 2H), 3.01 (dd, J = 4.0, 15.0 Hz, 1H), 2.84 (d, J = 14.5 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.0, 135.8, 134.8, 133.7, 131.8, 128.9, 128.8, 128.4, 127.9, 127.8, 125.3, 80.9, 68.1, 44.8, 44.5; MS (CI, NH_3) m/z 328 (M + H)$^+$; HRMS (CI, NH_3) calcd for $C_{19}H_{19}NO_2Cl$ (M + H)$^+$ 328.1104, found (M + H)$^+$ 328.1097. Anal. Calcd for $C_{19}H_{18}NO_2Cl$: C, 69.70; H, 5.55; N, 4.28. Found: C, 69.63; H, 5.80; N, 4.44.

1-Benzyl-4-[3-(*E*)-(4-methylphenyl)-2-propenyl-1-oxy]-azetidin-2-one (21). Benzyl lactam **7** and lactam **21** were found to coelute and thus were isolated as a mixture (51% yield by ^{1}H NMR integration): TLC R_f 0.63 (Et_2O); ^{1}H NMR (270 MHz, $CDCl_3$) δ 7.37−7.09 (m, 9H), 6.39 (d, J = 16.0 Hz, 1H), 6.06 (dt, J = 6.0, 16.0 Hz, 1H), 4.97−4.94 (m, 1H), 4.59 (d, J = 15.0 Hz, 1H), 4.23 (d, J = 15.0 Hz, 1H), 4.11−3.87 (m, 2H), 3.09−2.84 (m, 2H), 2.32 (s, 3H); MS (CI, NH_3) m/z 308 (M + H)$^+$; HRMS (CI, NH_3) calcd for $C_{20}H_{22}NO_2$ (M + H)$^+$ 308.1651, found (M + H)$^+$ 308.1652.

1-Benzyl-4-[3-(*E*)-(4-methoxyphenyl)-2-propenyl-1-oxy]-azetidin-2-one (22): colorless oil (53%), TLC R_f 0.52 (Et_2O); IR (film) 1759 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 7.31−7.17 (m, 7H), 6.80−6.75 (m, 2H), 6.30 (d, J = 16.0 Hz, 1H), 5.96−5.86 (m, 1H), 4.90 (dd, J = 1.5, 4.0 Hz, 1H), 4.53 (d, J = 15.5 Hz, 1H), 4.17 (d, J = 15.5 Hz, 1H), 4.01−3.96 (m, 2H), 3.74 (s, 3H), 3.01 (dd, J = 4.0, 15.0 Hz, 1H), 2.85 (dd, J = 1.0, 15.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.1, 159.6, 135.9, 133.0, 129.0, 128.9, 128.4, 127.8, 122.3, 114.1, 80.7, 68.8, 55.4, 44.8, 44.6; MS (CI, NH_3) m/z 324 (M + H)$^+$; HRMS (CI, NH_3) calcd for $C_{20}H_{22}NO_3$ (M + H)$^+$ 324.1600, found (M + H)$^+$ 324.1634. Anal. Calcd for $C_{20}H_{21}NO_3$: C, 74.27; H, 6.55; N, 4.33. Found: C, 74.53; H, 6.42; N, 4.31.

Ethyl 2-(*tert*-Butyldimethylsilyl)oxy-2-{2-oxo-4-[3-(*E*)-phenyl-2-propenyl-1-oxy]azetidinyl}acetate (23): colorless oil (67%); TLC R_f 0.45 (1:1 Et_2O/hexanes); IR (film) 1777, 1758 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.42−7.27 (m, 5H), 6.64 (d, J = 16.0 Hz, 1H), 6.32−6.22 (m, 1H), 5.64 (s, 1H), 5.42 (dd, J = 1.5, 4.5 Hz, 1H), 4.54 (dd, J = 1.5, 13.0 Hz, 1H), 4.29 (dd, J = 1.5, 6.0 Hz, 1H), 4.25 (q, J = 7.0 Hz, 2H), 3.20 (dd, J = 4.5, 15.0 Hz, 1H), 2.95 (dd, J = 1.5, 15.0 Hz, 1H), 1.32 (t, J = 7.0 Hz, 3H), 0.96 (s, 9H), 0.22 (s, 3H), 0.21 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.8, 166.0, 136.6, 133.0, 128.6, 127.8, 126.5, 124.9, 81.7, 72.0, 70.0, 61.9, 45.2, 25.6, 18.2, 14.2, −4.9, −5.4; MS (CI, NH_3) m/z 437 (M + NH_4)$^+$, 420 (M + H)$^+$; HRMS (CI, NH_3) calcd for $C_{22}H_{37}N_2O_5Si$ (M + NH_4)$^+$ 437.2471, found (M + NH_4)$^+$ 437.2464.

Ethyl 2-(*tert*-Butyldimethylsilyl)oxy-2-{2-oxo-4-[3-(*E*)-phenyl-2-propenyl-1-oxy]azetidinyl}acetate (24): colorless oil (79%); TLC R_f 0.41 (1:1 Et_2O/hexanes); IR (film) 1779, 1759 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.32−7.17 (m, 5H), 6.54 (d, J = 16.0, 1H), 6.19−6.10 (m, 1H), 5.49 (s, 1H), 5.21 (dd, J = 2.0, 4.0 Hz, 1H), 4.22 (dd, J = 1.5, 6.0 Hz, 1H), 4.15 (dd, J = 1.0, 5.0 Hz, 1H), 4.14 (q, J = 7.0 Hz, 2H), 3.00 (dd, J = 4.0, 15.5 Hz, 1H), 2.90 (dd, J = 2.0, 15.5 Hz, 1H), 1.21 (t, J = 7.0 Hz, 3H), 0.85 (s, 9H), 0.09 (s, 3H), 0.04 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.2, 165.4, 136.4, 132.9, 128.6, 127.9, 126.5, 124.8, 78.7, 71.8, 69.3, 61.9, 44.6, 25.6, 18.2, 14.1, −5.1, −5.2; MS (CI, NH_3) m/z 437 (M + NH_4)$^+$, 420 (M + H)$^+$; HRMS (CI, NH_3) calcd for $C_{22}H_{34}NO_5Si$ (M + H)$^+$ 420.2206, found (M + H)$^+$ 420.2215.

1-Benzyl-4-{*N*-[3-(*E*)-phenyl-2-propenyl]-4-toluenesulfonamido}azetidin-2-one (25). Benzyl lactam **13** and lactam **25** were found to coelute and thus were isolated as a mixture (35% yield by ^{1}H NMR integration): TLC R_f 0.68 (Et_2O); ^{1}H NMR (270 MHz, $CDCl_3$) δ 7.65−7.61 (m, 4H), 7.37−7.17 (m, 10H), 6.40 (d, J = 16.0 Hz, 1H), 6.00 (m, 1H), 5.55−5.53 (m, 1H), 4.43 (d, J = 15.5 Hz, 1H), 3.85 (d, J = 15.5 Hz, 1H), 3.75−3.68 (m, 2H), 3.06−2.87 (m, 2H), 2.42 (s, 3H); MS (CI, NH_3) m/z 447 (M + NH_4)$^+$; HRMS (CI, NH_3) calcd for $C_{26}H_{27}N_2O_3S$ (M + NH_4)$^+$ 447.1742, found (M + NH_4)$^+$ 447.1745.

1-Benzyl-4-{*N*-[3-(*E*)-(4-chlorophenyl)-2-propenyl]-4-toluenesulfonamido}azetidin-2-one (26). Benzyl lactam **13** and lactam **26** were found to coelute and thus were isolated as a mixture (43% yield by ^{1}H NMR integration): TLC R_f 0.68 (Et_2O); ^{1}H NMR (270 MHz, $CDCl_3$) δ 7.64−7.61 (m, 4H), 7.36−7.09 (m, 9H), 6.36 (d, J = 16.0 Hz, 1H), 5.97 (m, 1H), 5.60−5.53 (m, 1H), 4.43 (d, J = 15.0 Hz, 1H), 3.93−3.68 (m, 3H), 3.06−2.88 (m, 2H), 2.42 (s, 3H); MS (CI, NH_3) m/z 481 (M + H)$^+$; HRMS (CI, NH_3) calcd for $C_{26}H_{26}N_2O_3ClS$ (M + H)$^+$ 481.1353, found (M + H)$^+$ 481.1348.

General Procedure for the Zinc Acetate Preparation of 27a−d. Zinc acetate dihydrate (0.43 g, 1.94 mmol) was refluxed in PhH (60 mL) with azeotropic removal of water for 1 h, followed by the addition of lactam **4** (0.50 g, 3.87 mmol) and the respective unsaturated alcohol (8.5 mmol). The suspension was heated at reflux for 4 h, cooled, filtered through Celite, and concentrated. The resulting oil was chromatographed.

4-(3-Butenyl-2-oxy)azetidin-2-one (27a): from 3-buten-2-ol, inseparable mixture of diastereoisomers as a colorless oil (67%); TLC R_f 0.56 (Et_2O); IR (film) 3267, 1768 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 7.87 (br s, 1H), 7.53 (br s, 1H), 5.84−5.66 (m, 2H), 5.29−5.07 (m, 6H), 4.02 (m, 2H), 3.13−3.03 (m, 2H), 2.84−2.77 (m, 2H), 1.28−1.27 (m, 6H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 168.0, 167.7, 140.1, 139.1, 116.9, 16.6, 77.1, 76.7, 75.7, 75.5, 45.9, 45.7, 21.4, 21.2; MS (CI, NH_3) m/z 159 (M + NH_4)$^+$, 142 (M + H)$^+$; HRMS (CI, NH_3) calcd for $C_7H_{12}NO_2$ (M + H)$^+$ 142.0868, found (M + H)$^+$ 142.0866.

4-(3-Butenyl-1-oxy)azetidin-2-one (27b): from 3-buten-1-ol, colorless oil (83%); TLC R_f 0.34 (Et_2O); IR (film) 3276, 1764 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.13 (br s, 1H), 5.85−5.69 (m, 1H), 5.12−5.01 (m, 3H), 3.59−3.43 (m, 2H), 3.05 (ddd, J = 3.0, 4.0, 15.0 Hz, 1H), 2.83 (ddd, J = 0.5, 1.5, 15.0 Hz, 1H), 2.37−2.28 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.6, 134.5, 116.9, 77.9, 67.2, 45.2, 33.9; MS (CI, NH_3) m/z 159 (M + NH_4)$^+$, 142 (M + H)$^+$; HRMS (CI, NH_3) calcd for $C_7H_{15}N_2O_2$ (M + NH_4)$^+$ 159.1134, found (M + NH_4)$^+$ 159.1143. Anal. Calcd for $C_7H_{11}NO_2$: C, 59.56; H, 7.85; N, 9.92. Found: C, 59.29; H, 7.67; N, 9.76.

4-(4-Pentenyl-1-oxy)azetidin-2-one (27c): from 4-penten-1-ol, colorless oil (65%); TLC R_f 0.34 (Et_2O); IR (film) 3246, 1765 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.33 (br s, 1H), 5.78−5.68 (m, 1H), 5.00−4.89 (m, 3H), 3.50−3.34 (m, 2H), 3.00 (ddd, J = 3.0, 4.0, 15.0 Hz, 1H), 2.77 (dd, J = 1.5, 15.0 Hz, 1H), 2.10−2.02 (m, 2H), 1.68−1.57 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.4, 137.3, 114.7, 77.5, 66.7, 44.7, 29.6, 28.1; MS (CI, NH_3) m/z 173 (M + NH_4)$^+$, 156 (M + H)$^+$; HRMS (CI, NH_3) calcd for $C_8H_{17}N_2O_2$ (M + NH_4)$^+$ 173.1290, found (M + NH_4)$^+$ 173.1297. Anal. Calcd for $C_8H_{13}NO_2$: C, 61.90; H, 8.45; N, 9.03. Found: C, 61.81; H, 8.44; N, 9.31.

4-(5-Hexenyl-1-oxy)azetidin-2-one (27d): from 5-hexen-1-ol, colorless oil (73%); TLC R_f 0.34 (Et_2O); IR (film) 3279, 1766 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.99 (br s, 1H), 5.84−5.70 (m, 1H), 5.03−4.91 (m, 3H), 3.53−3.38 (m, 2H), 3.06 (ddd, J = 2.5, 3.0, 15.0 Hz, 1H), 2.84 (ddd, J = 0.5, 1.0, 15.0 Hz, 1H), 2.10−2.01 (m, 2H), 1.64−1.54 (m, 2H), 1.49−1.40 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.8, 138.4, 114.7, 73.0, 67.8, 45.1, 33.3, 28.9, 25.2; MS (CI, NH_3) m/z 187 (M + NH_4)$^+$, 170 (M + H)$^+$; HRMS (CI, NH_3) calcd for $C_9H_{16}NO_2$ (M + H)$^+$ 170.1181, found (M + H)$^+$ 170.1182. Anal. Calcd for $C_9H_{15}NO_2$: C, 63.88; H 8.93; N 8.28. Found: C, 63.67; H, 8.78; N, 8.55.

General Procedure for *N*-Alkylation of Azetidinones. Method A. NaH (0.062 g as a 60% dispersion in oil, 1.56 mmol) was added in one portion to a stirred solution of azetidinone (1.42 mmol) in DMF (10 mL) at 0 °C, followed after 1 min by allyl bromide (0.61 mL, 7.08 mmol). After 1 h, the solution was diluted with Et_2O (40 mL), washed with water (3 × 20 mL), dried ($MgSO_4$), concentrated, and chromatographed. **Method B.** To a solution of azetidinone (0.90 mmol) in PhH (2 mL) was added powdered KOH (0.06 g, 1.07 mmol), 18-crown-6 (0.01 g, 0.004 mmol), and an alkyl halide (1.49 mmol). The mixture was stirred for 2 h, filtered, concentrated, and chromatographed. **Method C.** Powdered KOH (0.14 g, 2.48 mmol) and tetrabutylammonium bromide (0.13 g, 0.41 mmol) were added to a solution of azetidinone (2.25 mmol) and an appropriate propargylic bromide (2.48 mmol) in THF (5 mL) at 0 °C. The reaction was allowed to stir for 2 h, filtered, concentrated, and chromatographed.

1-(2-Propenyl)-4-(2-propenyl-1-oxy)azetidin-2-one (28): from **5** following the general procedure for *N*-alkylation (method A); colorless oil (0.18 g, 76%); TLC R_f 0.50 (Et_2O); IR (film) 1761 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 5.91−5.68 (m, 2H), 5.29−5.15 (m, 4H), 5.02 (dd, J = 1.5, 4.0 Hz, 1H), 4.10−3.89 (m, 3H), 3.71 (dd, J = 7.0, 16.0 Hz, 1H), 3.05 (dd, J = 3.0, 16.0 Hz, 1H), 2.84 (d, J = 14.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.9, 133.7, 132.0, 118.3, 117.6, 81.0, 68.8, 44.5, 43.3; MS (CI, NH_3) m/z 185 $(M + NH_4)^+$, 168 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_9H_{17}N_2O_2$ $(M + NH_4)^+$ 185.1300, found $(M + NH_4)^+$ 185.1290.

4-(3-Butenyl-2-oxy)-1-(2-propenyl)azetidin-2-one (29a): from **27a** following the general procedure for *N*-alkylation (method A); inseparable mixture of diastereoisomers as a colorless oil (79%); TLC R_f 0.83 (Et_2O); IR (film) 1764 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 5.85−5.67 (m, 4H), 5.30−5.12 (m, 8H), 5.06−5.02 (m, 2H), 4.08−3.92 (m, 4H), 3.76−3.60 (m, 2H), 3.09−3.20 (m, 2H), 2.81−2.79 (m, 2H), 1.29−1.27 (m, 6H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 165.5, 165.3, 138.9, 131.8, 131.5, 117.4, 117.4, 115.9, 115.4, 79.2, 78.8, 75.9, 74.4, 45.0, 44.8, 42.5, 42.2, 20.8; MS (CI, NH_3) m/z 199 $(M + NH_4)^+$, 182 $(M + H)^+$; HRMS (CI, NH_3) calc for $C_{10}H_{16}NO_2$ $(M + H)^+$ 182.1181, found $(M + H)^+$ 182.1179.

4-(3-Butenyl-1-oxy)-1-(2-propenyl)azetidin-2-one (29b): from **27b** following the general procedure for *N*-alkylation (method A); colorless oil (78%); TLC R_f 0.50 (Et_2O); IR (film) 1764 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 5.79−5.65 (m, 2H), 5.21−4.92 (m, 5H), 3.92−3.85 (m, 1H), 3.69 (dd, J = 7.0, 16.0 Hz, 1H), 3.58−3.42 (m, 2H), 2.96 (dd, J = 4.0, 15.0 Hz, 1H), 2.75 (d, J = 15.0 Hz, 1H), 2.20−2.19 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.9, 134.5, 132.0, 118.3, 117.0, 81.2, 67.0, 44.2, 43.4, 34.1; MS (CI, NH_3) m/z 199 $(M + NH_4)^+$, 182 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{10}H_{16}NO_2$ $(M + H)^+$ 182.1181, found $(M + H)^+$ 182.1188.

4-(4-Pentenyl-1-oxy)-1-(2-propenyl)azetidin-2-one (29c): from **27c** following the general procedure for *N*-alkylation (method A); colorless oil (72%); TLC R_f 0.49 (Et_2O); IR (film) 1766 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 5.79−5.63 (m, 2H), 5.19−4.89 (m, 5H), 3.88 (dd, J = 5.0, 15.5 Hz, 1H), 3.63 (dd, J = 6.5, 15.5 Hz, 1H), 3.52−3.36 (m, 2H), 2.96 (dd, J = 4.0, 15.0 Hz, 1H), 2.73 (d, J = 15.0 Hz, 1H), 2.09−2.00 (m, 2H), 1.65−1.55 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.0, 137.7, 132.0, 118.2, 115.1, 81.2, 67.0, 44.2, 43.3, 30.0, 28.7; MS (CI, NH_3) m/z 213 $(M + NH_4)^+$, 196 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{11}H_{21}N_2O_2$ $(M + NH_4)^+$ 213.1603, found $(M + NH_4)^+$ 213.1597. Anal. Calcd for $C_{11}H_{17}NO_2$: C, 67.95; H, 8.56; N, 7.18. Found: C, 67.66; H, 8.78; N, 7.17.

4-(5-Hexenyl-1-oxy)-1-(2-propenyl)azetidin-2-one (29d): from **27d** following the general procedure for *N*-alkylation (method A); colorless oil (70%); TLC R_f 0.56 (Et_2O); IR (film) 1765 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 5.75−5.61 (m, 2H), 5.17−5.07 (m, 2H), 4.93−4.82 (m, 3H), 3.89−3.81 (m, 1H), 3.61 (dd, J = 7.0, 16.0 Hz, 1H), 3.49−3.34 (m, 2H), 2.93 (dd, J = 4.0, 14.5 Hz, 1H), 2.70 (d, J = 14.5 Hz, 1H), 2.00−1.92 (m, 2H), 1.54−1.51 (m, 2H), 1.49−1.29 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.9, 138.3, 132.0, 118.1, 114.7, 81.2, 67.6, 44.2, 43.3, 33.3, 29.0, 25.2; MS (CI, NH_3) m/z 227 $(M + NH_4)^+$, 210 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{12}H_{20}NO_2$ $(M + H)^+$ 210.1494, found $(M + H)^+$ 210.1489. Anal. Calcd for $C_{12}H_{19}NO_2$: C, 68.87; H, 9.15; N, 6.69. Found: C, 68.81; H, 9.32; N, 6.92.

4-(2-Propenyl-1-thio)azetidin-2-one (30a). To a −5 °C solution of allyl mercaptan (0.79 g, 10.70 mmol) in MeOH (7 mL) was added NaOMe (1.67 mL of a 25% solution in MeOH, 7.75 mmol). The solution was allowed to warm to 10 °C over 10 min, followed by cooling to −15 °C. A solution of **4** (1.00 g, 7.75 mmol) in MeOH (5 mL) was added, and the solution was allowed to warm to 25 °C. After 1.5 h, the mixture was poured into pH 7 buffer solution (50 mL), washed with Et_2O (3 × 50 mL), dried ($MgSO_4$), and concentrated. The resulting oil was chromatographed (4:1 Et_2O/hexanes) to produce **30a** as a colorless oil (0.76 g, 69%): TLC R_f 0.32 (3:1 Et_2O/hexanes); IR (KBr) 3257, 1756 cm^{-1}; ^1H NMR (250 MHz, $CDCl_3$) δ 6.72 (br s, 1H), 5.96−5.80 (m, 1H), 5.26−5.12 (m, 2H), 4.75 (dd, J = 2.0, 6.0 Hz, 1H), 3.38 (ddd, J = 2.0, 5.0, 15.0 Hz, 1H), 3.30 (d, J = 2.0 Hz, 2H), 2.95 (ddd, J = 2.0, 2.0, 15.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.5, 134.5, 117.7, 52.0, 45.9, 34.3; MS (CI, NH_3) m/z 161 $(M + NH_4)^+$, 144 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_6H_{10}NOS$ $(M + H)^+$ 144.0483, found $(M + H)^+$ 144.0497.

4-(2-Propenyl)azetidin-2-one (30b).[17] Et_3N (3.71 g, 36.6 mmol) was added dropwise to a solution of **4** (4.30 g, 33.3 mmol) and allylchlorodimethylsilane (4.91 g, 36.6 mmol) in CH_2Cl_2 (20 mL). The mixture was stirred for 15 h, concentrated, diluted with Et_2O (200 mL), washed with water (3 × 150 mL), dried ($MgSO_4$), and concentrated. This oil was dissolved in CH_2Cl_2 (15 mL) and cooled to 0 °C, and TMSOTf (2.58 mL, 13.32 mmol) was added. After 2 h of stirring at 0 °C, Et_3N (10.11 g, 99.9 mmol) was added, followed by MeOH (10 mL), and this mixture was stirred for a further 2 h at 0 °C. The solution was concentrated, diluted with Et_2O (200 mL), washed with water (3 × 150 mL), dried ($MgSO_4$), and concentrated. The resulting oil was chromatographed (Et_2O) to produce **30b** as a colorless oil (2.57 g, 69%): TLC R_f 0.27 (Et_2O); IR (KBr) 3257, 1754 cm^{-1}; ^1H NMR (250 MHz, $CDCl_3$) δ 6.15 (br s, 1H), 5.86−5.70 (m, 1H), 5.18−5.08 (m, 2H), 3.75−3.65 (m, 1H), 3.07 (ddd, J = 2.0, 5.0, 14.5 Hz, 1H), 2.63 (dt, J = 2.0, 14.5 Hz, 1H), 2.47−2.28 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.0, 133.2, 118.0, 47.0, 42.9, 39.4; MS (CI, NH_3) m/z 129 $(M + NH_4)^+$, 112 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_6H_{13}N_2O$ $(M + NH_4)^+$ 129.1028, found $(M + H)^+$ 129.1031.

1-(2-Propenyl)-4-[*N*-(2-propenyl)-4-toluenesulfonamido]azetidin-2-one (31): from **12** following the general procedure for *N*-alkylation of azetidinones (method A); white solid (89%); mp 48 °C; TLC R_f 0.71 (Et_2O); IR (KBr) 1766 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 7.70 (d, J = 8.0 Hz, 2H), 7.32 (d, J = 8.0 Hz, 2H), 5.88−5.60 (m, 2H), 5.63 (dd, J = 2.0, 5.0 Hz, 1H), 5.23−5.07 (m, 4H), 3.92−3.68 (m, 3H), 3.27 (dd, J = 6.0, 15.0 Hz, 1H), 3.08 (dd, J = 5.0, 15.0 Hz, 1H), 2.92 (dd, J = 2.0, 15.0 Hz, 1H), 2.43 (s, 3H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 165.3, 144.1, 137.1, 134.5, 131.0, 129.9, 127.1, 118.7, 118.0, 63.4, 44.4, 43.3, 42.3, 21.6; MS (CI, NH_3) m/z 338 $(M + NH_4)^+$, 321 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{16}H_{21}N_2O_3S$ $(M + H)^+$ 321.1273, found $(M + H)^+$ 321.1276. Anal. Calcd for $C_{16}H_{20}N_2O_3S$: C, 59.98; H, 6.29; N 8.74. Found: C, 60.15; H, 6.12; N, 8.67.

1-(2-Propenyl)-4-(2-propenyl-1-thio)azetidin-2-one (32a): from **30a** following the general procedure for *N*-alkylation of azetidinones (method A); colorless oil (81%); TLC R_f 0.56 (3:1 Et_2O/hexanes); IR (film) 1766 cm^{-1}; ^1H NMR (250 MHz, $CDCl_3$) δ 5.91−5.72 (m, 2H), 5.28−5.12 (m, 4H), 4.66 (dd, J = 2.0, 5.0 Hz, 1H), 4.07 (dd, J = 5.0, 15.0 Hz, 1H), 3.62 (dd, J = 7.0, 15.0 Hz, 1H), 3.36 (dd, J = 5.0, 15.0 Hz, 1H), 3.21 (d, J = 7.0 Hz, 2H), 2.96 (dd, J = 2.0, 16.0 Hz, 1H); ^{13}C NMR (62.5 MHz, $CDCl_3$) δ 165.4, 134.0, 131.8, 118.5, 117.9, 56.0, 45.2, 42.8, 32.8; MS (CI, NH_3) m/z 201 $(M + NH_4)^+$, 184 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_9H_{17}N_2OS$ $(M + NH_4)^+$ 201.1061, found $(M + NH_4)^+$ 201.1058.

1,4-Di(2-propenyl)azetidin-2-one (32b): from **30b** following the general procedure for *N*-alkylation employing allyl bromide (method B); colorless oil (0.10 g, 77%); TLC R_f 0.54 (Et_2O); IR (film) 1741 cm^{-1}; ^1H NMR (250 MHz, $CDCl_3$) δ 5.85−5.68 (m, 2H), 5.26−5.11 (m, 4H), 4.02 (dd, J = 6.0, 16.0 Hz, 1H), 3.71−3.60 (m, 2H), 3.02 (dd, J = 5.0, 15.0 Hz, 1H), 2.63 (dd, J = 2.0, 15.0 Hz, 1H), 2.56−2.47 (m, 1H), 2.33−2.22 (m, 1H); ^{13}C NMR (62.5 MHz, $CDCl_3$) δ 166.7, 132.6, 132.1, 118.2, 50.4, 43.3, 41.7, 37.1; MS (CI, NH_3) m/z 169 $(M + NH_4)^+$, 152 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_9H_{14}NO$ $(M + H)^+$ 152.1075, found $(M + H)^+$ 152.1069.

(3*S*,4*R*)-(−)-3-{(1*R*)-[(*tert*-Butyldimethylsilyl)oxy]ethyl}-4-(2-propenyl)azetidin-2-one (33).[27] Following the procedure for the preparation of **30b**, lactam **33** was prepared in an analogous manner from (3*R*,4*R*)-(+)-4-acetoxy-3-[1-(*R*)-(*tert*-butyldimethylsilyl)oxyethyl]azetidin-2-one as a white solid (81%): mp 75−77 °C; TLC R_f 0.38 1:1 (Et_2O/hexanes); $[\alpha]^{23}_D$ = −19.0 (*c* 1.00, $CHCl_3$); IR (KBr) 3240, 1755 cm^{-1}; ^1H NMR

(27) Hannesian, S.; Desilets, D.; Bennanai, Y. L. *J. Org. Chem.* **1990**, *55*, 3098.

(250 MHz, $CDCl_3$) δ 5.97 (br s, 1H), 5.86−5.70 (m, 1H), 5.16−5.08 (m, 2H), 4.22−4.12 (m, 1H), 3.73−3.66 (m, 1H), 2.80−2.76 (m, 1H), 2.48−2.26 (m, 2H), 1.20 (d, J = 6.0 Hz, 3H), 0.87 (s, 9H), 0.07 (s, 6H); ^{13}C NMR (62.5 MHz, $CDCl_3$) δ 168.5, 133.8, 118.0, 65.5, 63.7, 50.1, 39.4, 25.7, 22.7, 17.9, −4.2, −4.9; MS (CI, NH_3) m/z 287 $(M + NH_4)^+$, 270 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{14}H_{28}NO_2Si$ $(M + H)^+$ 270.1889, found $(M + H)^+$ 270.1878. Anal. Calcd for $C_{14}H_{27}NO_2Si$: C, 62.40; H 10.10; N 5.20. Found: C, 62.47; H, 10.05; N, 5.25.

(3*S*,4*R*)-(+)-3-{(1*R*)-[(*tert*-Butyldimethylsilyl)oxyethyl}-1,4-di(2-propenyl)azetidin-2-one (34): from **33** following the general procedure for *N*-alkylation employing allyl bromide (method B); colorless oil (92%); TLC R_f 0.52 (1:1 Et_2O/hexanes); $[\alpha]^{21}_D$ = +4.0 (*c* 1.00, $CHCl_3$); IR (film) 1746 cm^{-1}; ^{1}H NMR (250 MHz, $CDCl_3$) δ 5.88−5.68 (m, 2H), 5.27−5.08 (m, 4H), 4.22−4.12 (m, 1H), 4.01 (dd, J = 5.0, 15.0 Hz, 1H), 3.73−3.67 (m, 1H), 3.66−3.56 (m, 1H), 2.80−2.76 (m, 1H), 2.54−2.25 (m, 2H), 1.17 (d, J = 6.0 Hz, 3H), 0.87 (s, 9H), 0.07 (s, 3H), 0.05 (s, 3H); ^{13}C NMR (62.5 MHz, $CDCl_3$) δ 167.6, 133.5, 132.4, 115.2, 65.3, 62.5, 53.3, 43.2, 37.3, 25.8, 22.9, 17.9, −4.4, −4.8; MS (CI, NH_3) m/z 327 $(M + NH_4)^+$, 310 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{17}H_{32}NO_2Si$ $(M + H)^+$ 310.2202, found $(M + H)^+$ 310.2227. Anal. Calcd for $C_{17}H_{31}NO_2Si$: C, 65.97; H 10.09; N 4.53. Found: C, 65.84; H, 10.01; N, 4.34.

1-(2-Propenyl)-4-vinylazetidin-2-one (38a): from **36** following the general procedure for *N*-alkylation employing allyl bromide (method B); colorless oil (68%); TLC R_f 0.59 (Et_2O); IR (film) 1755 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 5.85−5.68 (m, 2H), 5.38−5.14 (m, 4H), 4.06−3.97 (m, 2H), 3.52 (ddd, J = 0.5, 7.0, 15.5 Hz, 1H), 3.18 (dd, J = 5.0, 15.0 Hz, 1H), 2.69 (dd, J = 2.5, 14.5 Hz, 1H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 166.6, 136.4, 131.9, 119.4, 118.3, 53.4, 43.8, 43.4; MS (CI, NH_3) m/z 155 $(M + NH_4)^+$, 138 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_8H_{15}N_2O$ $(M + NH_4)^+$ 155.1182, found $(M + NH_4)^+$ 155.1184. Anal. Calcd for $C_8H_{11}NO$: C, 70.04; H, 8.08; N, 10.21. Found: C, 69.92; H, 7.89; N, 10.25.

1-(2-Butenyl)-4-vinylazetidin-2-one (38b): from **36** following the general procedure for *N*-alkylation employing crotyl bromide (method B); colorless oil (82%) as a mixture of *E* and *Z* geometrical isomers; TLC R_f 0.63 (Et_2O); IR (film) 1753 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 5.85−5.23 (m, 5H), 4.04−3.90 (m, 1H), 3.64 (dd, J = 8.0 Hz, 15.0, 1H), 3.46 (dd, J = 7.0, 15.5 Hz, 1H), 3.14 (ddd, J = 1.5, 5.0, 14.5 Hz, 1H), 2.65 (d, J = 14.5 Hz, 1H), 1.68 (d, J = 6.0 Hz, 3H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 166.3, 136.4, 129.7, 128.4, 124.5, 123.4, 119.0, 52.9, 43.5, 42.5, 37.0, 17.5, 12.7; MS (CI, NH_3) m/z 169 $(M + NH_4)^+$, 152 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_9H_{14}NO$ $(M + H)^+$ 152.1075, found $(M + H)^+$ 152.1069.

1-(3-Butenyl)-4-vinylazetidin-2-one (38c): from **36** following the general procedure for *N*-alkylation employing 4-bromo-1-butene (method B); colorless oil (32%); TLC R_f 0.60 (Et_2O); IR (film) 1752 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 5.87−5.68 (m, 2H), 5.41−5.04 (m, 4H), 4.06−4.00 (m, 1H), 3.46−3.36 (m, 1H), 3.14 (dd, J = 5.0, 14.5 Hz, 1H), 3.08−3.01 (m, 1H), 2.65 (dd, J = 0.5, 14.5 Hz, 1H), 2.34−2.25 (m, 2H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 166.8, 136.7, 135.1, 119.2, 117.0, 53.7, 43.7, 40.1, 32.3; MS (CI, NH_3) m/z 169 $(M + NH_4)^+$, 152 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_9H_{14}NO$ $(M + H)^+$ 152.1075, found $(M + H)^+$ 152.1069.

4-(3-Butenyl)-1-(2-propenyl)azetidin-2-one (39): from **37** following the general procedure for *N*-alkylation employing allyl bromide (method B); colorless oil (75%); TLC R_f 0.70 (Et_2O); IR (film) 1751 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 5.84−5.72 (m, 2H), 5.26−4.96 (m, 4H), 4.00 (dd, J = 4.0, 15.5 Hz, 1H), 3.65 (dd, J = 6.5, 15.5 Hz, 1H), 3.60−3.56 (m, 1H), 2.99 (dd, J = 3.0, 14.5 Hz, 1H), 2.55 (dd, J = 2.5, 14.5 Hz, 1H), 2.13−2.05 (m, 2H), 1.97−1.88 (m, 1H), 1.55−1.47 (m, 1H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 166.8, 137.2, 132.4, 118.1, 113.4, 51.3, 43.3, 42.4, 32.2, 29.8; MS (CI, NH_3) m/z 183 $(M + NH_4)^+$, 166 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{10}H_{16}NO$ $(M + H)^+$ 166.1232, found $(M + H)^+$ 166.1232.

Ethyl 2-Hydroxy-2-[2-oxo-4-(2-propenyl)azetidinyl] acetate (40). From **30b**, the same method as for the preparation of **8** furnished carbinolamine **40** (1:1 inseparable mixture of diastereomers) as a viscous liquid (89%): TLC R_f 0.67 (Et_2O); IR (film) 3393, 1753, 1731 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 5.87−5.67 (m, 2H), 5.44 (d, J = 6.0 Hz, 1H), 5.42 (d, J = 7.0 Hz, 1H), 5.20−5.09 (m, 4H), 4.75 (d, J = 6.0 Hz, 1H), 4.67 (d, J = 7.0 Hz, 1H), 4.31 (q, J = 7.0 Hz, 2H), 4.29 (q, J = 7.0 Hz, 2H), 4.00−3.94 (m, 1H), 3.87−3.80 (m, 1H), 3.15−2.95 (m, 2H), 2.68−2.19 (m, 6H), 1.40−1.33 (m, 6H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 169.5, 168.5, 167.5, 166.9, 133.0, 132.5, 118.5, 72.1, 71.6, 62.7, 62.6, 51.1, 49.6, 42.1, 41.9, 37.9, 37.0, 14.1, 14.0; MS (CI, NH_3) m/z 231 $(M + NH_4)^+$, 214 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{10}H_{16}NO_4$ $(M + H)^+$ 214.1079, found $(M + H)^+$ 214.1090.

Ethyl 2-[2-Oxo-4-(2-propenyl)azetidinyl]-2-(2-propenyl-1-oxy)acetate (41): from **40** following the general procedure for *N*-alkylation (method A); 1:1 inseparable mixture of diastereomers as a colorless oil (0.094 g, 69%); TLC R_f 0.64 (Et_2O); IR (film) 1755 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 5.97−5.65 (m, 4H), 5.43−5.08 (m, 10H), 4.25−3.88 (m, 10H), 3.07 (m, 2H), 2.78−2.54 (m, 4H), 2.35−2.24 (m, 2H), 1.33−1.31 (m, 6H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 167.9, 167.6, 166.8, 166.0, 132.9, 132.8, 132.7, 132.3, 119.2, 118.9, 118.5, 118.3, 76.7, 76.5, 69.8, 69.5, 62.0, 51.7, 49.7, 42.2, 42.0, 38.2, 36.8, 14.1, 14.0; MS (CI, NH_3) m/z 271 $(M + NH_4)^+$, 254 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{13}H_{20}NO_4$ $(M + H)^+$ 254.1392, found $(M + H)^+$ 254.1399. Anal. Calcd for $C_{13}H_{19}NO_4$: C, 61.64; H, 7.56; N, 5.53. Found: C, 61.54; H, 7.26; N, 5.82.

(2*S*)-Ethyl 2-Hydroxy-2-{(3*S*,4*R*)-3-[(*tert*-butyldimethylsilyl)oxy-1-ethyl]-2-oxo-4-(2-propenyl)azetidinyl}-acetate (42) and (2*R*)-Ethyl 2-Hydroxy-2-{(3*S*,4*R*)-3-[(*tert*-butyldimethylsilyl)oxy-1-ethyl]-2-oxo-4-(2-propenyl)-azetidinyl}acetate (43). From **33**, the same method as for the preparation of **8** furnished carbinolamines **42** and **43** as chromatographically separable diastereoisomers. **42:** white solid (42%); mp 60−61 °C; TLC R_f 0.69 (3:1 Et_2O/hexanes); $[\alpha]^{27}_D$ = −70.8 (*c* 1.08, $CHCl_3$); IR (KBr) 3355, 1764, 1749 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 5.77−5.71 (m, 1H), 5.42 (s, 1H), 5.17−5.08 (m, 2H), 4.31−4.13 (m, 3H), 3.98−3.92 (m, 1H), 2.86 (dd, J = 2.5, 15.5 Hz, 1H), 2.45−2.41 (m, 1H), 2.34−2.28 (m, 1H), 1.33 (t, J = 7.0 Hz, 3H), 1.19 (d, J = 6.0, 3H), 0.88 (s, 9H), 0.07 (s, 3H), 0.06 (s, 3H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 170.1, 168.0, 133.2, 118.5, 71.3, 65.6, 62.5, 52.9, 37.1, 29.7, 25.8, 22.9, 17.9, 14.0, −4.4, −4.8; MS (CI, NH_3) m/z 389 $(M + NH_4)^+$, 372 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{18}H_{34}NO_5Si$ $(M + H)^+$ 372.2206, found $(M + H)^+$ 372.2215. **43:** viscous oil (43%); TLC R_f 0.64 (3:1 Et_2O/hexanes); $[\alpha]^{29}_D$ = −4.0 (*c* 1.10, $CHCl_3$); IR (film) 3409, 1768, 1742 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 5.90−5.80 (m, 1H), 5.47 (s, 1H), 5.19−5.09 (m, 2H), 4.84 (br s, 1H), 4.29 (q, J = 7.0 Hz, 2H), 4.13 (m, 1H), 3.87−3.82 (m, 1H), 2.84 (dd, J = 2.5, 15.5 Hz, 1H), 2.63−2.58 (m, 1H), 2.52−2.44 (m, 1H), 1.33 (t, J = 7.0 Hz, 3H), 1.19 (d, J = 6.0 Hz, 3H), 0.87 (s, 9H), 0.07 (s, 3H), 0.05 (s, 3H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 168.1, 167.4, 133.6, 118.1, 71.5, 65.4, 62.5, 62.1, 54.6, 37.8, 25.6, 22.6, 17.7, 13.9, −4.6, −5.1; MS (CI, NH_3) m/z 389 $(M + NH_4)^+$, 372 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{18}H_{34}NO_5Si$ $(M + H)^+$ 372.2206, found $(M + H)^+$ 372.2218. Anal. Calcd for $C_{18}H_{33}NO_5Si$: C, 58.19; H, 8.95; N, 3.77. Found: C, 58.10; H, 8.65; N, 3.54.

(2*S*)-Ethyl 2-{(3*S*,4*R*)-3-[(*tert*-Butyldimethylsilyl)oxy-1-ethyl]-2-oxo-4-(2-propenyl)azetidinyl}-2-(2-propenyl-1-oxy)acetate (44) and (2*R*)-Ethyl 2-{(3*S*,4*R*)-3-[(*tert*-Butyldimethylsilyl)oxy-1-ethyl]-2-oxo-4-(2-propenyl)azetidinyl}-2-(2-propenyl-1-oxy)acetate (45): from **43** and **45**, respectively, following the general procedure for *N*-alkylation of azetidinones (method A). **45:** colorless oil (82%); TLC R_f 0.69 (1:1 Et_2O/hexanes); $[\alpha]^{32}_D$ = +22.0 (*c* 1.02, $CHCl_3$); IR (film) 1764, 1754 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 5.95−5.76 (m, 2H), 5.39 (s, 1H), 5.42−5.08 (m, 4H), 4.31−4.14 (m, 4H), 4.06 (dd, J = 6.0, 12.5 Hz, 1H), 3.96−3.91 (m, 1H), 2.88 (dd, J = 2.5, 15.0 Hz, 1H), 2.75−2.70 (m, 1H), 2.39−2.34 (m, 1H), 1.32 (t, J = 7.0 Hz, 3H), 1.19 (d, J = 6.0 Hz, 3H), 0.85 (s, 9H), 0.06 (s, 3H), 0.04 (s, 3H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 168.5, 165.9, 133.6, 132.9, 118.8, 118.2, 76.5, 69.3, 65.4, 62.8, 61.8, 55.1, 38.2, 25.7, 22.8, 17.9, 14.1, −4.4, −4.9; MS (CI, NH_3) m/z 429 $(M + NH_4)^+$, 412 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{21}H_{38}NO_5Si$ $(M + H)^+$ 412.2519, found $(M + H)^+$ 412.2524. **44:** colorless oil (86%); TLC R_f 0.60 (1:1 Et_2O/hexanes); $[\alpha]^{29}_D$

= −112.0 (*c* 1.09, $CHCl_3$); IR (film) 1768, 1743 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 5.96−5.68 (m, 2H), 5.40 (s, 1H), 5.34−5.08 (m, 4H), 4.28−4.14 (m, 4H), 4.00 (dd, *J* = 6.5, 12.5 Hz, 1H), 3.93−3.88 (m, 1H), 2.94 (dd, *J* = 3.0, 16.0 Hz, 1H), 2.52−2.48 (m, 1H), 2.33−2.28 (m, 1H), 1.31 (t, *J* = 7.0 Hz, 3H), 1.21 (d, *J* = 6.0 Hz, 3H), 0.88 (s, 9H), 0.09 (s, 3H), 0.05 (s, 3H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 168.6, 167.1, 133.1, 132.7, 119.2, 118.5, 76.1, 69.5, 65.9, 62.6, 62.0, 53.3, 36.8, 25.9, 23.1, 18.1, 14.1, −4.3, −4.6; MS (CI, NH_3) *m/z* 429 $(M + NH_4)^+$, 412 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{21}H_{38}NO_5Si$ $(M + H)^+$ 412.2519, found $(M + H)^+$ 412.2531.

General Procedure for the Ring-Closing Metathesis of Dienes 28, 29a−d, 31, 32a−b, and 39. A solution of carbene **1** or **3** in CH_2Cl_2 (6 mL) was added to the corresponding diene (50 mg) (Table 2), stirred for 2 h, and exposed to air. The dark solution was concentrated and chromatographed.

1-Aza-6-oxabicyclo[5.2.0]non-3-en-9-one (46): colorless oil (84%); TLC R_f 0.33 (Et_2O); IR (film) 1756 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 5.69−5.56 (m, 2H), 5.26 (dd, *J* = 1.0, 3.5 Hz, 1H), 4.48−4.33 (m, 2H), 4.23−4.15 (m, 1H), 3.75−3.72 (m, 1H), 3.18−3.11 (m, 1H), 2.91 (d, *J* = 15.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.3, 128.5, 125.0, 82.6, 65.3, 44.1, 42.1; MS (CI, NH_3) *m/z* 157 $(M + NH_4)^+$; HRMS (CI, NH_3) calcd for $C_7H_{13}N_2O_2$ $(M + NH_4)^+$ 157.0982, found $(M + NH_4)^+$ 157.0977. Anal. Calcd for $C_7H_9NO_2$: C, 60.42; H, 6.52; N, 10.07. Found: C, 60.52; H, 6.25; N, 10.35.

5-Methyl-1-aza-6-oxabicyclo[5.2.0]non-3-en-9-one (47): separable diastereoisomers both as colorless oils (60% combined yield). **47a:** TLC R_f 0.42 (Et_2O); IR (film) 1758 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.56−5.48 (m, 2H), 5.15 (dd, *J* = 1.0, 4.0 Hz, 1H), 4.46−4.40 (m, 1H), 4.31 (d, *J* = 19.0 Hz, 1H), 3.75 (d, *J* = 18.0 Hz, 1H), 3.15 (ddd, *J* = 2.5, 3.5, 14.5 Hz, 1H), 2.84 (d, *J* = 14.5 Hz, 1H), 1.35 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.2, 134.1, 123.6, 81.8, 75.3, 45.0, 41.7, 22.2; MS (CI, NH_3) *m/z* 171 $(M + NH_4)^+$, 154 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_8H_{12}NO_2$ $(M + H)^+$ 154.0868, found $(M + H)^+$ 154.0867. **47b:** TLC R_f 0.52 (Et_2O); IR (film) 1760 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.51 (s, 2H), 5.40 (d, *J* = 3.5 Hz, 1H), 4.75−4.70 (m, 1H), 4.63 (dd, *J* = 2.0, 16.5 Hz, 1H), 3.65 (dd, *J* = 2.5, 15.5 Hz, 1H), 3.12 (ddd, *J* = 1.0, 3.5, 15.5 Hz, 1H), 2.92 (d, *J* = 15.0 Hz, 1H), 1.33 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.4, 134.1, 126.6, 79.9, 64.9, 43.3, 42.5, 21.6; MS (CI, NH_3) *m/z* 171 $(M + NH_4)^+$, 154 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_8H_{12}NO_2$ $(M + H)^+$ 154.0868, found $(M + H)^+$ 154.0868.

1-Aza-7-oxabicyclo[6.2.0]dec-3-en-10-one (48): colorless oil (76%); TLC R_f 0.31 (Et_2O); IR (film) 1754 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 6.02−5.82 (m, 2H), 4.88 (dd, *J* = 2.5, 4.0 Hz, 1H), 4.05−3.97 (m, 1H), 3.86 (dd, *J* = 7.0, 14.0 Hz, 1H), 3.55 (dd, *J* = 7.5, 14.0 Hz, 1H), 3.59−3.51 (m, 1H), 3.01 (dd, *J* = 4.0, 15.0 Hz, 1H), 2.72 (dd, *J* = 2.5, 15.0 Hz, 1H), 2.48−2.30 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.2, 133.8, 124.9, 82.0, 69.3, 43.0, 35.5, 29.3; MS (CI, NH_3) *m/z* 171 $(M + NH_4)^+$, 154 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_8H_{15}N_2O_2$ $(M + NH_4)^+$ 171.1134, found $(M + NH_4)^+$ 171.1145.

1-Aza-8-oxabicyclo[7.2.0]undec-3-en-11-one (49): colorless oil (23%); TLC R_f 0.31 (Et_2O); IR (film) 1761 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.86 (dt, *J* = 7.5, 10.5 Hz, 1H), 5.69 (dt, *J* 7= 0.5, 10.5 Hz, 1H), 5.14 (dd, *J* = 1.5, 4.0 Hz, 1H), 3.90 (dd, *J* = 7.0, 14.5 Hz, 1H), 3.83−3.78 (m, 1H), 3.65 (dd, *J* = 8.0, 14.5 Hz, 1H), 3.50−3.44 (m, 1H), 2.94 (dd, *J* = 4.0, 15.0 Hz, 1H), 2.79 (d, *J* = 15.0 Hz, 1H), 2.44−2.26 (m, 2H), 1.71−1.53 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.7, 135.0, 124.3, 83.8, 62.7, 42.8, 36.7, 26.1, 22.4; MS (CI, NH_3) *m/z* 185 $(M + NH_4)^+$, 168 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_9H_{14}NO_2$ $(M + H)^+$ 168.1025, found $(M + H)^+$ 168.1030.

1-Aza-9-oxabicyclo[8.2.0]dodec-3-en-12-one (50): white solid (10%); mp 88−89 °C; TLC R_f 0.31 (Et_2O); IR (KBr) 1756 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.71 (dt, *J* = 4.0, 11.0 Hz, 1H), 5.57−5.51 (m, 1H), 4.94 (d, *J* = 3.0 Hz, 1H), 3.94−3.89 (m, 1H), 3.81−3.65 (m, 3H), 2.97 (dd, *J* = 2.5, 14.5 Hz, 1H), 2.87−2.77 (m, 1H), 2.66 (dd, *J* = 1.5, 14.0 Hz, 1H), 2.11−2.07 (m, 1H), 2.01−1.91 (m, 2H), 1.82−1.74 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.8, 138.3, 121.0, 77.9, 69.8, 44.6, 35.8, 26.8, 25.9, 23.5; MS (CI, NH_3) *m/z* 199 $(M + NH_4)^+$, 182 $(M + H)^+$; HRMS (CI) calcd for $C_{10}H_{16}NO_2$ $(M + H)^+$ 182.1181, found $(M + H)^+$ 182.1183.

6-(4-Toluenesulfonyl)-1,6-diazabicyclo[5.2.0]non-3-en-9-one (51): white solid (92%); mp 141 °C; TLC R_f 0.43 (Et_2O); IR (KBr) 1763 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 7.70 (d, *J* = 8.0 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 5.65 (d, *J* = 4.0 Hz, 1H), 5.63−5.52 (m, 1H), 5.37−5.28 (m, 1H), 4.32−4.20 (m, 2H), 3.86−3.74 (m, 1H), 3.27 (dd, *J* = 4.0, 15.0 Hz, 1H), 3.14−3.03 (m, 1H), 2.78 (dd, *J* = 2.0, 15.0 Hz, 1H), 2.43 (s, 3H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 165.3, 144.1, 137.0, 129.8, 128.1, 127.3, 125.6, 64.9, 43.4, 42.2, 39.3, 21.6; MS (CI, NH_3) *m/z* 310 $(M + NH_4)^+$, 293 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{14}H_{17}N_2O_3S$ $(M + H)^+$ 293.0960, found $(M + H)^+$ 293.0964. Anal. Calcd for $C_{14}H_{16}N_2O_3S$: C, 57.32; H, 5.52; N 9.58. Found: C, 57.49; H, 5.26; N, 9.48.

6-Thia-1-azabicyclo[5.2.0]non-3-en-9-one (52): colorless oil (79%); TLC R_f 0.33 (3:1 Et_2O/hexanes); IR (film) 1754 cm^{-1}; ^1H NMR (250 MHz, $CDCl_3$) δ 6.02−5.90 (m, 1H), 5.61−5.52 (m, 1H), 4.88 (dd, *J* = 2.0, 5.0 Hz, 1H), 4.40 (dd, *J* = 5.0, 18.0 Hz, 1H), 3.70 (dd, *J* = 5.0, 18.0 Hz, 1H), 3.57−3.48 (m, 1H), 3.43 (dd, *J* = 5.0, 15.0 Hz, 1H), 3.02 (dd, *J* = 2.0, 15.0 Hz, 1H), 2.97 (d, *J* = 15.0 Hz, 1H); ^{13}C NMR (62.5 MHz, $CDCl_3$) δ 165.5, 129.8, 127.5, 55.9, 46.0, 41.4, 23.6; MS (CI, NH_3) *m/z* 173 $(M + NH_4)^+$, 156 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_7H_{10}NOS$ $(M + H)^+$ 156.0483, found $(M + H)^+$ 156.0494.

1-Azabicyclo[4.2.0]oct-3-en-8-one (53): colorless oil (81%); TLC R_f 0.34 (Et_2O); IR (film) 1754 cm^{-1}; ^1H NMR (250 MHz, $CDCl_3$) δ 5.90−5.68 (m, 2H), 4.07 (dd, *J* = 4.0, 15.0 Hz, 1H), 3.57−3.42 (m, 2H), 3.21 (ddd, *J* = 2.0, 5.0, 15.0 Hz, 1H), 2.56 (dd, *J* = 1.0, 15.0 Hz, 1H), 2.52−2.41 (m, 1H), 2.18−2.03 (m, 1H); ^{13}C NMR (62.5 MHz, $CDCl_3$) δ 167.0, 123.8, 122.6, 45.3, 43.1, 38.5, 28.7; MS (CI, NH_3) *m/z* 141 $(M + NH_4)^+$, 124 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_7H_{10}NO$ $(M + H)^+$ 124.0762, found $(M + H)$ 124.0761.

1-Azabicyclo[5.2.0]non-3-en-9-one (54): colorless oil (83%); TLC R_f 0.28 (Et_2O); IR (film) 1745 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 5.80−5.72 (m, 1H), 5.55−5.48 (m, 1H), 4.35 (ddd, *J* = 1.0, 5.5, 18.5 Hz, 1H), 3.82−3.76 (m, 1H), 3.57−3.50 (m, 1H), 3.10 (ddd, *J* = 1.5, 5.0, 14.5 Hz, 1H), 2.60 (dd, *J* = 2.0, 14.5 Hz, 1H), 2.42−2.34 (m, 1H), 2.20−2.07 (m, 2H), 1.76−1.64 (m, 1H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 166.2, 130.7, 126.0, 53.2, 42.8, 41.2, 32.9, 24.7; MS (CI, NH_3) *m/z* 138 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_8H_{12}NO$ $(M + H)^+$ 138.0919, found $(M + H)^+$ 138.0918.

(6*R*,7*S*)-(+)-7-[1-(*R*)-(*tert*-Butyldimethylsilyl)oxyethyl]-1-azabicyclo[4.2.0]oct-3-en-8-one (55): colorless oil (78%); TLC R_f 0.34 (1:1 Et_2O/hexanes); $[\alpha]^{21}_D = +60.0$ (*c* 1.00, $CHCl_3$); IR (film) 1758, 1748, 1732 cm^{-1}; ^1H NMR (250 MHz, $CDCl_3$) δ 5.88−5.64 (m, 2H), 4.17 (m, 1H), 4.06 (dd, *J* = 3.0, 18.0 Hz, 1H), 3.54−3.42 (m, 2H), 2.74 (dd, *J* = 1.0, 15.0 Hz, 1H), 2.48−2.34 (m, 1H), 2.22−2.06 (m, 1H), 1.25 (d, *J* = 6.0 Hz, 3H), 0.86 (s, 9H), 0.07 (s, 6H); ^{13}C NMR (62.5 MHz, $CDCl_3$) δ 167.2, 124.0, 122.4, 66.9, 65.7, 45.9, 38.1, 28.1, 25.7, 22.8, 17.9, −4.2, −5.1; MS (CI, NH_3) *m/z* 282 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{15}H_{28}NO_2Si$ $(M + H)^+$ 282.1889, found $(M + H)$ 282.1871.

1-Methyl-4-vinylazetidin-2-one (58): from **36** following the general procedure for *N*-alkylation employing methyl iodide (method B); colorless oil (0.22 g, 65%); TLC R_f 0.34 (Et_2O); IR (film) 1747 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 5.86−5.73 (m, 1H), 5.41−5.27 (m, 2H), 3.97−3.91 (m, 1H), 3.17 (ddd, *J* = 0.5, 3.0, 14.5 Hz, 1H), 2.76 (s, 3H), 2.68 (ddd, *J* = 1.0, 2.5, 14.5 Hz, 1H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 167.1, 136.1, 119.4, 54.8, 44.2, 26.9; MS (CI, NH_3) *m/z* 129 $(M + NH_4)^+$, 112 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_6H_{13}N_2O$ $(M + NH_4)^+$ 129.1027, found $(M + NH_4)^+$ 129.1026.

1-Methyl-4-(2-propenyl)azetidin-2-one (59): from **30b** following the general procedure for *N*-alkylation employing MeI (method B); colorless oil (63%); TLC R_f 0.24 (Et_2O); IR (film) 1746 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 5.83−5.71 (m, 1H), 5.20−5.11 (m, 2H), 3.61−3.55 (m, 1H), 2.99 (ddd, *J* = 0.5, 5.0, 14.5 Hz, 1H), 2.80 (s, 3H), 2.60 (ddd, *J* = 1.0, 2.0, 14.5 Hz, 1H), 2.55−2.25 (m, 2H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 167.0, 132.7, 118.4, 51.9, 42.1, 36.8, 27.0; MS (CI, NH_3) *m/z* 143 $(M + NH_4)^+$, 126 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_7H_{15}N_2O$ $(M + NH_4)^+$ 143.1184, found $(M + NH_4)^+$ 143.1184.

1-Benzyl-4-(2-propenyl)azetidin-2-one (60): from **30b** following the general procedure for *N*-alkylation employing benzyl bromide (method B); colorless oil (70%); TLC R_f 0.56 (Et_2O); IR (film) 1744 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 7.36−7.22 (m, 5H), 5.69−5.57 (m, 1H), 5.09−5.01 (m, 2H), 4.58 (d, J = 15.5 Hz, 1H), 4.13 (d, J = 15.5 Hz, 1H), 3.57−3.50 (m, 1H), 2.96 (dd, J = 5.0, 14.5 Hz, 1H), 2.61 (dd, J = 2.5, 14.5 Hz, 1H), 2.43−2.33 (m, 1H), 2.22−2.12 (m, 1H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 166.7, 136.1, 132.7, 128.7, 128.1, 127.6, 118.2, 50.3, 44.7, 41.8, 37.0; MS (CI, NH_3) m/z 219 $(M + NH_4)^+$, 202 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{13}H_{16}NO$ $(M + H)^+$ 202.1231, found $(M + H)^+$ 202.1230. Anal. Calcd for $C_{13}H_{15}NO$: C, 77.58; H, 7.51; N, 6.96. Found: C, 77.39; H, 7.55; N, 6.79.

4-(3-Butenyl)-1-methylazetidin-2-one (61): from **37** following the general procedure for *N*-alkylation employing MeI (method B); colorless oil (85%); TLC R_f 0.33 (Et_2O); IR (film) 1746 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 5.86−5.74 (m, 1H), 5.08−4.97 (m, 2H), 3.53−3.47 (m, 1H), 3.00 (dd, J = 7.0, 14.5 Hz, 1H), 2.79 (s, 3H), 2.53 (dd, J = 0.5, 14.5 Hz, 1H), 2.16−2.11 (m, 2H), 1.99−1.90 (m, 1H), 1.58−1.50 (m, 1H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 167.1, 137.2, 115.5, 52.4, 42.6, 31.8, 29.8, 26.8; MS (CI, NH_3) m/z 157 $(M + NH_4)^+$, 140 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_8H_{14}NO$ $(M + H)^+$ 140.1075, found $(M + H)^+$ 140.1070.

1-Benzyl-4-(3-butenyl)azetidin-2-one (62): from **37** following the general procedure for *N*-alkylation employing benzyl bromide (method B); colorless oil (86%); TLC R_f 0.68 (Et_2O); IR (film) 1748 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 7.36−7.16 (m, 5H), 5.76−5.62 (m, 1H), 4.96−4.91 (m, 2H), 4.57 (d, J = 15.5 Hz, 1H), 4.15 (d, J = 15.5 Hz, 1H), 3.49−3.45 (m, 1H), 2.99 (ddd, J = 1.5, 5.0, 14.5 Hz, 1H), 2.58 (d, J = 14.5 Hz, 1H), 2.02−1.94 (m, 2H), 1.84−1.74 (m, 1H), 1.51−1.38 (m, 1H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 167.0, 137.1, 136.2, 129.3, 128.1, 127.6, 115.4, 51.0, 44.6, 42.4, 32.0, 29.6; MS (CI, NH_3) m/z 233 $(M + NH_4)^+$, 216 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{14}H_{18}NO$ $(M + H)^+$ 216.1388, found $(M + H)^+$ 216.1383. Anal. Calcd for $C_{14}H_{17}NO$: C, 78.10; H, 7.96; N, 6.51. Found: C, 77.81; H, 7.67; N, 6.49.

1-Benzyl-4-[4-(*E*)-phenyl-3-butenyl]azetidin-2-one (63): from **62** following the general procedure for cross-metathesis; colorless oil (72%); TLC R_f 0.53 (Et_2O); IR (film) 1746 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 7.38−7.18 (m, 10H), 6.27 (d, J = 16.0 Hz, 1H), 6.04 (dt, J = 7.0, 15.5 Hz, 1H), 4.60 (d, J = 15.5 Hz, 1H), 4.17 (d, J = 15.5 Hz, 1H), 3.54−3.51 (m, 1H), 3.04 (dd, J = 5.0, 14.5 Hz, 1H), 2.63 (d, J = 2.5, 14.5 Hz, 1H), 2.17−2.11 (m, 2H), 1.88−1.84 (m, 1H), 1.58−1.50 (m, 1H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 167.0, 137.3, 136.2, 130.8, 128.8, 128.5, 128.2, 127.7, 127.2, 125.9, 51.1, 44.8, 42.5, 32.6, 29.0; MS (CI, NH_3) m/z 309 $(M + NH_4)^+$, 292 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{20}H_{22}NO$ $(M + H)^+$ 292.1701, found $(M + H)^+$ 292.1699. Anal. Calcd for $C_{20}H_{21}NO$: C, 82.44; H, 7.26; N, 4.81. Found: C, 82.38; H, 7.27; N, 4.75.

(2*S,8*R**)-Ethyl 10-Oxo-3-oxa-1-azabicyclo[6.2.0]dec-5-ene-2-carboxylate (64) and (2*R**,8*R**)-10-Oxo-3-oxa-1-azabicyclo[6.2.0]dec-5-ene-2-carboxylate (65).** From **41** (Scheme 4), the general procedure for ring-closing metathesis afforded compounds **64** and **65** as white solids (75% combined yield). **64:** mp 76−77 °C; TLC R_f 0.35 (Et_2O); IR (KBr) 1754, 1743 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 6.05−5.98 (m, 1H), 5.88−5.81 (m, 1H), 4.74 (s, 1H), 4.45−4.21 (m, 4H), 3.79−3.73 (m, 1H), 3.12 (dd, J = 5.0, 15.0 Hz, 1H), 2.70−2.63 (m, 2H), 2.51−2.43 (m, 1H), 1.32 (t, J = 7.0 Hz, 3H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 165.9, 165.6, 129.8, 129.1, 78.0, 62.3, 51.4, 41.9, 30.3, 14.1; MS (CI, NH_3) m/z 243 $(M + NH_4)^+$, 226 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{11}H_{16}NO_4$ $(M + H)^+$ 226.1079, found $(M + H)^+$ 226.1084. Anal. Calcd for $C_{11}H_{15}NO_4$: C, 58.66; H, 6.71; N, 6.22. Found: C, 58.53; H, 6.51; N, 6.16. **65**: mp 76−77 °C; TLC R_f 0.67 (Et_2O); IR (KBr) 1748 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 5.68−5.57 (m, 2H), 5.50 (s, 1H), 4.63 (br d, J = 15.5 Hz, 1H), 4.31−4.05 (m, 4H), 3.24−3.17 (m, 1H), 2.99 (dd, J = 5.0, 15.0 Hz, 1H), 2.64 (dd, J = 2.5, 15.0 Hz, 1H), 2.28−2.18 (m, 1H), 1.33 (t, J = 7.0 Hz, 3H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 167.6, 166.1, 132.2, 120.2, 79.5, 70.3, 62.1, 49.1, 48.4, 28.0, 14.2; MS (CI, NH_3) m/z 243 $(M + NH_4)^+$, 226 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{11}H_{16}NO_4$ $(M + H)^+$ 226.1079, found $(M + H)^+$ 226.1082. Anal. Calcd for $C_{11}H_{15}NO_4$: C, 58.66; H, 6.71; N, 6.22. Found: C, 58.73; H, 6.73; N, 6.22.

(2*S*,8*R*,9*S*)-(−)-Ethyl 9-[(1*R*)−(*tert*-Butyldimethylsilyl)-oxyethyl]-10-oxo-3-oxa-1-azabicyclo[6.2.0]dec-5-ene-2-carboxylate (66): from **44** (Scheme 5) using the general procedure for ring closing metathesis; colorless oil (76%); TLC R_f 0.32 (1:1 Et_2O/hexanes); $[\alpha]^{29}_D = -54.0$ (*c* 0.50, $CHCl_3$); IR (film) 1769 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 6.04−5.97 (m, 1H), 5.86−5.80 (m, 1H), 4.73 (s, 1H), 4.40−4.10 (m, 5H), 3.73−3.68 (m, 1H), 2.81 (dd, J = 2.5, 6.5 Hz, 1H), 2.67−2.63 (m, 1H), 2.49−2.44 (m, 1H), 1.31 (t, J = 7.0 Hz, 3H), 1.24 (d, J = 6.0 Hz, 3H), 0.90 (s, 9H), 0.08 (s, 3H), 0.07 (s, 3H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 166.2, 165.8, 129.9, 129.1, 78.3, 66.2, 62.6, 62.2, 55.3, 30.1, 29.7, 25.8, 22.8, 17.9, 14.1, −4.3, −4.8; MS (CI, NH_3) m/z 384 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{19}H_{34}NO_5Si$ $(M + H)^+$ 384.2206, found $(M + H)^+$ 384.2202.

(2*R*,8*R*,9*S*)-(−)-Ethyl 9-[(1*R*)-(*tert*-Butyldimethylsilyl)-oxyethyl]-10-oxo-3-oxa-1-azabicyclo[6.2.0]dec-5-ene-2-carboxylate (67): from **45** (Scheme 5) using the general procedure for ring closing metathesis; colorless oil (79%); TLC R_f 0.48 (1:1 Et_2O/hexanes); $[\alpha]^{29}_D = -32.0$ (*c* 0.96, $CHCl_3$); IR (film) 1756 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 5.63−5.52 (m, 2H), 5.48 (s, 1H), 4.59 (d, J = 17.5 Hz, 1H), 4.23 (q, J = 7.0 Hz, 2H), 4.19−4.06 (m, 3H), 3.25−3.19 (m, 1H), 2.84−2.82 (m, 1H), 2.25−2.18 (m, 1H), 1.32 (t, J = 7.0 Hz, 3H), 1.21 (d, J = 6.0 Hz, 3H), 0.86 (s, 9H), 0.07 (s, 3H), 0.05 (s, 3H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 168.0, 166.0, 131.9, 121.1, 79.2, 70.0, 65.6, 61.9, 61.1, 52.4, 28.0, 25.7, 22.6, 17.9, 14.1, −4.3, −5.0; MS (CI, NH_3) m/z 401 $(M + NH_4)^+$, 384 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{19}H_{34}NO_5Si$ $(M + H)^+$ 384.2206, found $(M + H)^+$ 384.2216.

1,1-Bis(2-propenyl-1-oxy)propionamide (68): isolated as a byproduct in the preparation of lactam **5** from **4**; colorless oil; TLC R_f 0.44 (EtOAc); IR (film) 3409, 1671 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 6.34 (m, 2H), 5.91−5.77 (m, 2H), 5.27−5.09 (m, 4H), 4.87 (t, J = 5.0 Hz, 1H), 4.13−3.96 (m, 4H), 2.53 (d, J = 5.5 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.7, 133.9, 117.5, 99.3, 67.8, 41.3; MS (CI, NH_3) m/z 203 $(M + NH_4)^+$, 186 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_9H_{16}NO_3$ $(M + H)^+$ 186.1130, found $(M + H)^+$ 186.1135. Anal. Calcd for $C_9H_{15}NO_3$: C, 58.36; H, 8.16; N, 7.56. Found: C, 58.66; H, 8.17; N, 7.43.

(4,7-Dihydro-1,3-dioxepin-2-yl)acetamide (69): from **68** (Scheme 6) using the general procedure for ring-closing metathesis; white solid (84%); mp 92−93 °C; TLC R_f 0.18 (EtOAc); IR (KBr) 3506, 1683 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 6.15 (br s, 1H), 5.73 (s, 2H), 5.57 (br s, 1H), 5.12 (t, J = 5.5 Hz, 1H), 4.43 (d, J = 15.0 Hz, 2H), 4.24 (d, J = 15.0 Hz, 2H), 2.64 (d, J = 5.5 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.5, 129.3, 101.2, 66.1, 41.5; MS (CI, NH_3) m/z 175 $(M + NH_4)^+$, 158 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_7H_{12}NO_3$ $(M + H)^+$ 158.0817, found $(M + H)^+$ 158.0806. Anal. Calcd for $C_7H_{11}NO_3$: C, 53.49; H, 7.05; N, 8.91. Found: C, 53.27; H, 6.83; N, 8.75.

4-(2-Propenyl)-1-(2-propynyl)azetidin-2-one (70): from **30b** following the general procedure for *N*-alkylation employing 3-bromopropyne (80% solution in PhMe) (method C); colorless oil (0.25 g, 76%); TLC R_f 0.75 (Et_2O); IR (film) 1749 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 5.87−5.78 (m, 1H), 5.21−5.12 (m, 2H), 4.21−4.12 (m, 1H), 3.88−3.78 (m, 2H), 2.99 (ddd, J = 2.0, 5.5, 14.5 Hz, 1H), 2.62 (dd, J = 1.5, 14.5 Hz, 1H), 2.57−2.54 (m, 1H), 2.42−2.40 (m, 1H), 2.37 (t, J = 2.0 Hz, 1H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 166.2, 132.7, 118.4, 77.1 72.5, 50.7, 41.9, 37.0, 30.0; MS (CI, NH_3) m/z 167 $(M + NH_4)^+$, 150 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_9H_{12}NO$ $(M + H)^+$ 150.0919, found $(M + H)^+$ 150.0918.

1-(2-Butynyl)-4-(2-propenyl)azetidin-2-one (71): from **30b** following the general procedure for *N*-alkylation employing 1-bromo-2-butyne (method C); colorless oil (0.21 g, 58%); TLC R_f 0.76 (Et_2O); IR (film) 1754 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 5.88−5.75 (m, 1H), 5.20−5.13 (m, 2H), 4.18−4.10 (m, 1H), 3.83−3.73 (m, 2H), 2.99 (dd, J = 5.0, 14.5 Hz, 1H), 2.61 (dd, J = 2.0, 14.5 Hz, 1H), 2.56−2.54 (m, 1H), 2.40−2.30 (m,

1H), 1.82 (s, 3H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 166.4, 132.8, 118.4, 80.0, 72.3, 50.7, 41.7, 37.0, 30.5, 1.9; MS (CI, NH_3) *m/z* 164 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{10}H_{14}NO$ $(M + H)^+$ 164.1075, found $(M + H)^+$ 164.1074.

4-(3-Buten-1-yl)-1-(2-butynyl)azetidin-2-one (72): from **37** following the general procedure for *N*-alkylation employing 1-bromo-2-butyne (method C); colorless oil (83%); TLC R_f 0.71 (Et_2O); IR (film) 1754 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 5.90−5.75 (m, 1H), 5.11−4.98 (m, 2H), 4.10 (ddd, J = 2.5, 5.0, 17.5 Hz, 1H), 3.82−3.68 (m, 2H), 2.98 (dd, J = 3.0, 14.5 Hz, 1H), 2.55 (dd, J = 2.5, 14.5 Hz, 1H), 2.19−2.11 (m, 2H), 2.05−1.93 (m, 1H), 1.81 (s, 3H), 1.67−1.53 (m, 1H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 166.0, 136.9, 114.9, 79.5, 72.1, 50.8, 41.9, 31.7, 29.9, 29.4, 3.0; MS (CI, NH_3) *m/z* 195 $(M + NH_4)^+$, 178 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{11}H_{16}NO$ $(M + H)^+$ 178.1232, found $(M + H)^+$ 178.1228.

1-(2-Butynyl)-4-[*N*-(2-propenyl)-4-toluenesulfonamido]-azetidin-2-one (73): from **12** following the general procedure for *N*-alkylation employing 1-bromo-2-butyne (method C); viscous yellow oil (51%); TLC R_f 0.60 (3:1 Et_2O/hexanes); IR (film) 1768 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 7.76 (d, J = 8.5 Hz, 2H), 7.32 (d, J = 8.0 Hz, 2H), 5.80−5.77 (m, 1H), 5.75 (dd, J = 2.0, 5.0 Hz, 1H), 5.25−5.13 (m, 2H), 3.90−3.70 (m, 3H), 3.28 (dd, J = 2.5, 18.0 Hz, 1H), 3.10 (dd, J = 5.0, 15.5 Hz, 1H), 2.96 (dd, J = 2.0, 15.5 Hz, 1H), 2.44 (s, 3H), 1.80 (s, 3H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 165.1, 144.0, 137.0, 134.8, 129.9, 127.1, 117.8, 80.6, 71.8, 63.3, 44.5, 42.8, 30.7, 21.5, 3.4; MS (CI, NH_3) *m/z* 350 $(M + NH_4)^+$, 333 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{17}H_{21}N_2O_3S$ $(M + H)^+$ 333.1273, found $(M + H)^+$ 333.1267. Anal. Calcd for $C_{17}H_{20}N_2O_3S$: C, 61.42; H, 6.06; N, 8.43. Found: C, 61.34; H, 6.35; N, 8.29.

1-(2-Butynyl)-4-(2-propenyl-1-oxy)azetidin-2-one (74): from **5** following the general procedure for *N*-alkylation employing 1-bromo-2-butyne (method C); colorless oil (60%); TLC R_f 0.66 (Et_2O); IR (film) 1766 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 5.98−5.85 (m, 1H), 5.38−5.13 (m, 2H), 4.22−4.10 (m, 3H), 3.75 (d, J = 17.5 Hz, 2H), 3.08−2.99 (m, 1H), 2.86−2.78 (m, 1H), 1.82 (s, 3H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 165.3, 133.9, 117.5, 80.5, 72.1, 69.1, 44.4, 30.4, 3.4; MS (CI, NH_3) *m/z* 197 $(M + NH_4)^+$, 180 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{10}H_{14}NO_2$ $(M + H)^+$ 180.1025, found $(M + H)^+$ 180.1024.

4-(3-Butenyl-1-oxy)-1-(2-butynyl)azetidin-2-one (75): from **27b** following the general procedure for *N*-alkylation employing 1-bromo-2-butyne (method C); yellow oil (52%); TLC R_f 0.75 (Et_2O); IR (film) 1765 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 5.88−5.75 (m, 1H), 5.16−5.05 (m, 3H), 4.16 (dd, J = 2.5, 17.5 Hz, 1H), 3.80−3.59 (m, 3H), 3.03 (dd, J = 4.0, 15.0 Hz, 1H), 2.83 (dd, J = 1.0, 14.5 Hz, 1H), 2.42−2.33 (m, 2H), 1.82 (s, 3H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 165.4, 134.5, 116.9, 80.8, 80.3, 72.1, 67.4, 44.1, 34.1, 30.4, 3.4; MS (CI, NH_3) *m/z* 211 (M + $NH_4)^+$, 194 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{11}H_{16}NO_2$ $(M + H)^+$ 194.1181, found $(M + H)^+$ 194.1182.

4-(2-Butynyl-1-oxy)azetidin-2-one (76a): as for **27a−d** from 2-butyn-1-ol; white solid (77%); mp 64−65 °C; TLC R_f 0.47 (Et_2O); IR (KBr) 3250, 1751 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 6.88 (br s, 1H), 5.22−5.17 (m, 1H), 4.22−4.18 (m, 2H), 3.19−3.09 (m, 1H), 2.96−2.90 (m, 1H), 1.86 (s, 3H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 166.9, 83.7, 77.7, 74.1, 56.5, 45.7, 3.6; MS (CI, NH_3) *m/z* 157 $(M + NH_4^+)$, 140 (55, $M + H^+$); HRMS (CI, NH_3) calcd for $C_7H_{10}NO_2$ $(M + H)^+$ 140.0711, found $(M + H)^+$ 140.0713. Anal. Calcd for $C_7H_9NO_2$: C, 60.42; H, 6.52; N, 10.07. Found: C, 60.48; H, 6.31; N, 9.88.

4-[4-(*tert*-Butyldimethylsilyl)oxy-2-butynyl-1-oxy]azetidin-2-one (76b): as for **27a−d** from 1-(*tert*-butyldimethylsilyl)oxy-2-butyn-4-ol; white solid (56%); mp 38−39 °C; TLC R_f 0.56 (Et_2O); IR (KBr) 3286, 1777 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 7.06 (br s, 1H), 5.21 (dd, J = 1.5, 4.0 Hz, 1H), 4.35 (t, J = 2.0 Hz, 2H), 4.28 (t, J = 2.0 Hz, 2H), 3.14 (ddd, J = 3.0, 3.5, 15.5 Hz, 1H), 2.92 (dd, J = 1.0, 15.0 Hz, 1H), 0.91 (s, 9H), 0.12 (s, 6H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 166.8, 86.0, 79.7, 77.8, 56.2, 51.6, 45.7, 25.8, 18.3, −5.2; MS (CI, NH_3) *m/z* 287 $(M + NH_4)^+$, 270 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{13}H_{24}NO_3Si$ $(M + H)^+$ 270.1525, found $(M + H)^+$ 270.1517. Anal. Calcd for $C_{13}H_{23}NO_3Si$: C, 57.96; H, 8.60; N, 5.20. Found: C, 58.09; H, 8.34; N, 5.27.

4-(5-Oxa-oct-7-en-2-ynyl-1-oxy)azetidin-2-one (76c): as for **27a−d** from 4-(2-propenoxy)-2-butyn-1-ol; colorless oil (76%); TLC R_f 0.39 (Et_2O); IR (film) 3294, 1776 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 7.35−7.18 (br s, 1H), 5.95−5.82 (m, 1H), 5.35−5.17 (m, 3H), 4.42−4.29 (m, 2H), 4.26−4.16 (m, 2H), 4.07−4.01 (m, 2H), 3.20−3.09 (m, 1H), 2.96−2.85 (m, 1H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 167.1, 133.8, 118.1, 83.3, 81.2, 77.6, 70.8, 57.3, 56.0, 45.6; MS (CI, NH_3) *m/z* 213 $(M + NH_4)^+$, 196 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{10}H_{14}NO_3$ $(M + H)^+$ 196.0974, found $(M + H)^+$ 196.0969.

4-(2-Butynyl-1-oxy)-1-(2-propenyl)azetidin-2-one (77a): from **76a** following the general procedure for *N*-alkylation (method A); colorless oil (91%); TLC R_f 0.73 (Et_2O); IR (film) 1763 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 5.86−5.73 (m, 1H), 5.30−5.19 (m, 2H), 5.13 (dd, J = 1.5, 3.5 Hz, 1H), 4.21−4.18 (m, 2H), 4.00−3.92 (m, 1H), 3.74 (dd, J = 6.5, 16.0 Hz, 1H), 3.08 (dd, J = 3.5, 15.0 Hz, 1H), 2.89 (dd, J = 1.5, 15.0 Hz, 1H), 1.85 (s, 3H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 165.5, 131.8, 117.8, 83.2, 80.5, 74.1, 55.9, 44.3, 43.0, 3.2; MS (CI, NH_3) *m/z* 197 $(M + NH_4)^+$, 180 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{10}H_{14}NO_2$ $(M + H)^+$ 180.1025, found $(M + H)^+$ 180.1026. Anal. Calcd for $C_{10}H_{13}NO_2$: C, 67.02; H, 7.31; N, 7.82. Found: C, 66.80; H, 7.28; N, 7.61.

4-[4-(*tert*-Butyldimethylsilyl)oxy-2-butynyl-1-oxy]-1-(2-propenyl)azetidin-2-one (77b): from **76b** following the general procedure for *N*-alkylation (method A); colorless oil (82%); TLC R_f 0.56 (3:1 Et_2O/hexanes); IR (film) 1768 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 5.81−5.75 (m, 1H), 5.30−5.20 (m, 2H), 5.13 (dd, J = 1.5, 3.5 Hz, 1H), 4.34 (s, 2H), 4.27 (s, 2H), 4.00−3.92 (m, 1H), 3.77 (dd, J = 6.5, 15.5 Hz, 1H), 3.09 (dd, J = 3.5, 15.0 Hz, 1H), 2.89 (d, J = 15.0 Hz, 1H), 0.91 (s, 9H), 0.12 (s, 6H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 165.7, 132.0, 118.4, 85.9, 80.9, 79.8, 55.9, 51.6, 44.7, 43.3, 25.7, 18.3, −5.2; MS (CI, NH_3) *m/z* 327 $(M + NH_4)^+$, 310 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{16}H_{28}NO_3Si$ $(M + H)^+$ 310.1838, found $(M + H)^+$ 310.1838. Anal. Calcd for $C_{16}H_{27}NO_3Si$: C, 62.10; H, 8.79; N, 4.53. Found: C, 61.90; H, 8.70; N, 4.56.

4-(5-Oxa-oct-7-en-2-ynyl-1-oxy)-1-(2-propenyl)azetidin-2-one (77c): from **76c** following the general procedure for *N*-alkylation (method A); colorless oil (93%); TLC R_f 0.60 (Et_2O); IR (film) 1764 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 5.94−5.74 (m, 2H), 5.34−5.13 (m, 5H), 4.28 (s, 2H), 4.19 (s, 2H), 4.06−4.03 (m, 2H), 3.97 (dd, J = 4.0, 15.5 Hz, 1H), 3.72 (dd, J = 6.0, 15.5 Hz, 1H), 3.09 (dd, J = 3.0, 15.0 Hz, 1H), 2.94−2.89 (m, 1H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 165.7, 133.8, 131.9, 118.4, 117.9, 83.3, 81.4, 80.9, 70.8, 57.3, 55.8, 44.7, 43.4; MS (CI, NH_3) *m/z* 253 $(M + NH_4)^+$, 236 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{13}H_{18}NO_3$ $(M + H)^+$ 236.1287, found $(M + H)^+$ 236.1281.

4-(4-Hydroxy-2-butynyl-1-oxy)-1-(2-propenyl)azetidin-2-one (77d). To a solution of lactam **77b** (0.30 g, 0.97 mmol) in THF (10 mL) was added Bu_4NF (1 M solution in THF, 1.46 mL, 1.46 mmol). After 1.5 h, the solution was diluted with Et_2O (40 mL) and washed with water (50 mL), the aqueous layer was washed with Et_2O (3 × 50 mL); and the organic layers were combined, dried ($MgSO_4$), and concentrated. The dark oil was chromatographed (Et_2O) to produce **77d** as a colorless oil (0.17 g, 90%): TLC R_f 0.17 (Et_2O); IR (film) 3435, 1748 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 5.88−5.73 (m, 1H), 5.31−5.21 (m, 2H), 5.15 (dd, J = 1.0, 3.5 Hz, 1H), 4.28 (s, 4H), 4.00 (dd, J = 5.0, 15.5 Hz, 1H), 3.78 (dd, J = 3.5, 16.0 Hz, 1H), 3.31 (br s, 1H), 3.12 (dd, J = 4.0, 15.0 Hz, 1H), 2.92 (d, J = 15.0 Hz, 1H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 166.3, 131.8, 118.6, 85.9, 80.9, 80.2, 55.9, 50.6, 44.5, 43.5; MS (CI, NH_3) *m/z* 213 $(M + NH_4)^+$, 196 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{10}H_{14}NO_3$ $(M + H)^+$ 196.0973, found $(M + H)^+$ 196.0979. Anal. Calcd for $C_{10}H_{13}NO_3$: C, 61.53; H, 6.71; N, 7.17. Found: C, 61.30; H, 6.47; N, 7.41.

1-(*tert*-Butyldimethylsilyl)-4-(5-oxaoct-7-en-2-ynyl-1-oxy)azetidin-2-one (78a). To an ice-cooled solution of **76b** (76 mg, 0.39 mmol) and Et_3N (60 mg, 0.55 mmol) in CH_2Cl_2 (2 mL) was added *tert*-butyldimethylsilyl chloride (71 mg, 0.47 mmol). This mixture was stirred for 3 h, diluted with diethyl ether (10 mL), washed with water (3 × 10 mL), dried ($MgSO_4$), and concentrated, and the resulting oil was chromatographed

(1:1 Et_2O/hexanes) to give **78a** a colorless oil (84%): TLC R_f 0.36 (1:1 Et_2O/hexanes); IR (film) 1758 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 5.95−5.85 (m, 1H), 5.34−5.20 (m, 2H), 5.07 (dd, J = 1.5, 3.5 Hz, 1H), 4.25−4.03 (m, 6H), 3.16 (dd, J = 3.5, 15.5 Hz, 1H), 2.96 (dd, J = 1.5, 15.5 Hz, 1H), 0.97 (s, 9H), 0.26 (s, 3H), 0.25 (s, 3H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 170.5, 133.8, 117.9, 83.0, 81.4, 79.2, 70.7, 57.3, 54.6, 46.3, 26.0, 18.1, −5.8, −6.1; MS (CI, NH_3) m/z 327 $(M + NH_4)^+$, 310 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{16}H_{28}NO_3Si$ $(M + H)^+$ 310.1839, found $(M + H)^+$ 310.1848.

1-Benzyl-4-(5-oxa-oct-7-en-2-ynyl-1-oxy)azetidin-2-one (78b) from 76c. Lactam **78b** was synthesized in an analogous manner to the reaction producing **7** as a colorless oil (92%): TLC R_f 0.63 (Et_2O); IR (film) 1763 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 7.37−7.25 (m, 5H), 5.95−5.80 (m, 1H), 5.32−5.17 (m, 2H), 5.00 (dd, J = 1.5, 3.5 Hz, 1H), 4.58 (d, J = 15.5 Hz, 1H), 4.21 (d, J = 15.0 Hz, 1H), 4.13−4.11 (m, 4H), 4.01−3.98 (m, 2H), 3.07 (dd, J = 3.5, 15.0 Hz, 1H), 2.92 (dd, J = 1.0, 15.0 Hz, 1H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 165.7, 135.7, 133.8, 128.8, 128.2, 127.7, 117.8, 83.3, 81.3, 80.6, 70.7, 57.2, 55.5, 44.7; MS (CI, NH_3) m/z 303 $(M + NH_4)^+$, 286 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{17}H_{20}NO_3$ $(M + H)^+$ 286.1443, found $(M + H)^+$ 286.1432. Anal. Calcd for $C_{17}H_{19}NO_3$: C, 71.56; H, 6.71; N, 4.91. Found: C, 71.29; H, 6.45; N, 4.80.

(3*S*,4*R*)-(−)-3-[(1*R*)-(*tert*-Butyldimethylsilyl)oxyethyl]-1-(2-butynyl)-4-(2-propenyl)azetidin-2-one (79): from **33** following the general procedure for *N*-alkylation employing 1-bromo-2-butyne (method C); pale yellow liquid (79%); TLC R_f 0.46 (1:1 Et_2O/hexanes); $[\alpha]^{25}_D$ = −1.8 (c 1.01, $CHCl_3$); IR (film) 1760 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 5.78−5.71 (m, 1H), 5.13−5.03 (m, 2H), 4.11−3.64 (m, 4H), 2.70 (m, 1H), 2.49−2.44 (m, 1H), 2.42−2.28 (m, 1H), 1.73 (s, 3H), 1.11 (d, J = 6.0 Hz, 3H), 0.81 (s, 9H), 0.00 (s, 6H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 167.1, 135.5, 118.1, 79.7, 72.4, 65.1, 62.2, 53.1, 37.1, 30.1, 25.7, 22.8, 17.8, 3.3, −4.4, −5.0; MS (CI, NH_3) m/z 339 $(M + NH_4)^+$, 322 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{18}H_{32}NO_2Si$ $(M + H)^+$ 322.2202, found $(M + H)^+$ 322.2199. Anal. Calcd for $C_{18}H_{31}NO_2Si$: C, 67.24; H, 9.72; N, 4.36. Found: C, 67.26; H, 9.78; N, 4.29.

(3*S*,4*R*)-(+)-1-[4-(*tert*-Butyldimethylsilyl)oxy-2-butynyl]-3-[(1*R*)-(*tert*-butyldimethylsilyl)oxyethyl]-4-(2-propenyl)-azetidin-2-one (80): from **33** following the general procedure for *N*-alkylation employing 4-bromo-1-(*tert*-butyldimethylsilyloxy)-2-butyne (method C); pale yellow liquid (54%); TLC R_f 0.57 (1:1 Et_2O/hexanes); $[\alpha]^{26}_D$ = +1.1 (c 0.97, $CHCl_3$); IR (film) 1761 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 5.82−5.76 (m, 1H), 5.19−5.08 (m, 2H), 4.34−3.89 (m, 5H), 3.83−3.78 (m, 1H), 2.77 (dd, J = 2.0, 4.5 Hz, 1H), 2.53−2.52 (m, 1H), 2.40−2.37 (m, 1H), 1.16 (d, J = 6.0 Hz, 3H), 0.90 (s, 9H), 0.86 (s, 9H), 0.10 (s, 6H), 0.05 (s, 6H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 167.3, 133.5, 118.4, 82.6, 78.1, 65.3, 62.6, 53.8, 51.8, 37.2, 30.2, 25.8, 22.9, 18.4, 18.0, −4.3, −4.8, −5.1; MS (CI, NH_3) m/z 469 $(M + NH_4)^+$, 452 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{24}H_{46}NO_3Si_2$ $(M + H)^+$ 452.3016, found $(M + H)^+$ 452.3014.

5-Oxa-oct-7-en-2-yn-1-yl Bromide (82). To a solution of 4-(2-propenoxy)-2-butyn-1-ol (0.25 g, 1.98 mmol) in Et_2O (5 mL) were added CBr_4 (1.32 g, 3.96 mmol) and Ph_3P (1.04 g, 3.96 mmol). After 4 h, the mixture was filtered through Celite and chromatographed (1:9 Et_2O/hexanes) to produce **82** as a colorless oil (0.37 g, 98%): TLC R_f 0.63 (1:3 Et_2O/hexanes); IR (film) 2853, 1082 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 5.97−5.83 (m, 1H), 5.35−5.19 (m, 2H), 4.21−4.18 (m, 2H), 4.06−4.02 (m, 2H), 3.96 (t, J = 2.0 Hz, 2H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 133.9, 117.9, 82.9, 81.4, 70.7, 57.3, 14.3; MS (CI, NH_3) m/z 208 $(M + NH_4)^+$, 206 $(M + NH_4)^+$; HRMS (CI, NH_3) calcd for $C_7H_{13}NOBr$ $(M + NH_4)^+$ 208.0160, found $(M + NH_4)^+$ 208.0151.

(3*S*,4*R*)-(−)-3-[(1*R*)-(*tert*-Butyldimethylsilyl)oxyethyl]-1-(5-oxa-oct-7-en-2-yn-1-yl)-4-(2-propenyl)azetidin-2-one (83): from **33** following the general procedure for *N*-alkylation employing **82** (method C); yellow oil (68%); TLC R_f 0.41 (1:1 Et_2O/hexanes); $[\alpha]^{29}_D$ = −1.2 (c 1.01, $CHCl_3$); IR (film) 1758 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 5.91−5.78 (m, 2H), 5.34−5.10 (m, 4H), 4.23−3.81 (m, 8H), 2.79 (dd, J = 2.0, 14.0 Hz, 1H), 2.57−2.52 (m, 1H), 2.42−2.40 (m, 1H), 1.18 (d, J = 6.0 Hz, 3H), 0.87 (s, 9H), 0.06 (s, 6H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 167.2, 133.9, 133.4, 118.4, 117.8, 79.9, 79.7, 70.6, 65.1, 62.5, 57.3, 53.6, 37.2, 30.1, 25.7, 22.8, 17.9, −4.3, −4.9; MS (CI, NH_3) m/z 395 $(M + NH_4)^+$, 378 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{21}H_{36}NO_3Si$ $(M + H)^+$ 378.2465, found $(M + H)^+$ 378.2481.

General Procedure for the Metathesis of 70−75, 77a−d, 78a−b, 79, 80, and 83 (Table 3). To the appropriate enyne (50 mg) was added a solution of ruthenium catalyst **3** (21.0 mg, 0.03 mmol, 10 mol %) in CH_2Cl_2 (3 mL). This solution was heated to reflux for 3 h, allowed to cool, concentrated, and chromatographed.

3-(2-Propenyl)-1-azabicyclo[4.2.0]oct-3-en-8-one (85): colorless oil (44 mg, 88%); TLC R_f 0.46 (Et_2O); IR (film) 1752 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 5.95 (t, J = 2.5 Hz, 1H), 4.90 (s, 1H), 4.88 (s, 1H), 4.28 (dd, J = 2.0, 17.0 Hz, 1H), 3.66 (dd, J = 1.5, 17.0 Hz, 1H), 3.50−3.43 (m, 1H), 3.21 (ddd, J = 2.0, 4.5, 14.5 Hz, 1H), 2.65−2.55 (m, 2H), 2.27−2.17 (m, 1H), 1.91 (s, 3H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 166.8, 140.6, 132.8, 121.0, 111.1, 45.0, 42.7, 38.6, 29.3, 20.7; MS (CI, NH_3) m/z 181 $(M + NH_4)^+$, 164 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{10}H_{14}NO$ $(M + H)^+$ 164.1075, found $(M + H)^+$ 164.1083.

3-(2-Propenyl)-1-azabicyclo[5.2.0]non-3-en-9-one (86): colorless oil (82%); TLC R_f 0.35 (Et_2O); IR (film) 1746 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 5.99−5.94 (m, 1H), 4.95 (s, 1H), 4.92 (s, 1H), 4.69 (d, J = 17.0 Hz, 1H), 3.87−3.82 (m, 1H), 3.64 (dd, J = 0.5, 17.0 Hz, 1H), 3.09 (ddd, J = 1.0, 5.0, 14.5 Hz, 1H), 2.61 (dd, J = 2.0, 14.5 Hz, 1H), 2.46−2.40 (m, 1H), 2.25−2.04 (m, 2H), 1.87 (s, 3H), 1.81−1.70 (m, 1H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 166.2, 142.3, 138.1, 128.2, 111.7, 52.4, 42.7, 41.6, 31.5, 22.8, 21.4; MS (CI, NH_3) m/z 195 $(M + NH_4)^+$, 178 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{11}H_{16}NO$ $(M + H)^+$ 178.1231, found $(M + H)^+$ 178.1233.

3-(2-Propenyl)-6-(4-toluenesulfonyl)-1,6-diazabicyclo-[5.2.0]non-3-en-9-one (87): white solid (70%); mp 105−106 °C; TLC R_f 0.45 (3:1 Et_2O:hexane); IR (KBr) 1764 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 7.68 (d, J = 8.5 Hz, 2H), 7.25 (d, J = 8.5 Hz, 2H), 5.62−5.55 (m, 2H), 4.87 (s, 1H), 4.61 (s, 1H), 4.50 (d, J = 18.0 Hz, 1H), 4.31 (dd, J = 8.0, 18.0 Hz, 1H), 3.91 (d, J = 18.0 Hz, 1H), 3.29 (dd, J = 4.0, 15.5 Hz, 1H), 3.11 (d, J = 18.0 Hz, 1H), 2.83 (dd, J = 1.5, 15.5 Hz, 1H), 2.38 (s, 3H), 1.72 (s, 3H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 165.2, 144.1, 140.7, 138.3, 136.9, 129.7, 127.3, 122.2, 113.0, 84.2, 64.9, 44.0, 42.9, 39.1, 21.2; MS (CI, NH_3) m/z 350 $(M + NH_4)^+$, 333 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{17}H_{24}N_3O_3S$ $(M + NH_4)^+$ 350.1538, found $(M + NH_4)^+$ 350.1536. Anal. Calcd for $C_{17}H_{20}N_2O_3S$: C, 61.42; H, 6.06; N, 8.43. Found: C, 61.21; H, 5.95; N, 8.18.

3-(2-Propenyl)-1-aza-6-oxabicyclo[5.2.0]non-3-en-9-one (88): colorless oil (88%); TLC R_f 0.50 (Et_2O); IR (film) 1762 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 5.86−5.82 (m, 1H), 5.36 (d, J = 3.0 Hz, 1H), 4.99 (s, 1H), 4.97 (s, 1H), 4.75 (d, J = 18.0 Hz, 1H), 4.50 (dd, J = 2.0, 16.0 Hz, 1H), 4.16 (dd, J = 6.5, 16.0 Hz, 1H), 3.81 (d, J = 18.0 Hz, 1H), 3.13 (ddd, J = 1.5, 3.5, 15.5 Hz, 1H), 2.94 (d, J = 15.5 Hz, 1H), 1.88 (s, 3H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 165.5, 141.2, 139.0, 125.5, 113.3, 81.7, 60.1, 43.5, 42.8, 21.4; MS (CI, NH_3) m/z 197 $(M + NH_4)^+$, 180 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{10}H_{14}NO_2$ $(M + H)^+$ 180.1025, found $(M + H)^+$ 180.1030. Anal. Calcd for $C_{10}H_{13}NO_2$: C, 67.02; H, 7.31; N, 7.82. Found: C, 67.08; H, 7.03; N, 7.63.

3-(2-Propenyl)-1-aza-7-oxabicyclo[6.2.0]dec-3-en-10-one (89): pale yellow oil (12%); TLC R_f 0.42 (Et_2O); IR (film) 1756 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 6.01 (t, J = 8.5 Hz, 1H), 5.21 (s, 1H), 5.00 (s, 1H), 4.80 (dd, J = 1.5, 4.0 Hz, 1H), 4.23 (d, J = 8.5 Hz, 1H), 4.10−4.01 (m, 1H), 3.91 (d, J = 14.5 Hz, 1H), 3.61−3.53 (m, 1H), 3.02 (dd, J = 4.0, 14.5 Hz, 1H), 2.74 (dd, J = 1.5, 14.5 Hz, 1H), 2.58−2.51 (m, 1H), 2.46−2.40 (m, 1H), 1.92 (s, 3H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 164.9, 141.3, 137.2, 128.2, 112.9, 81.8, 69.9, 43.1, 36.7, 30.6, 20.7; MS (CI, NH_3) m/z 211 $(M + NH_4)^+$, 194 $(M + H^+)$; HRMS (CI, NH_3) calcd for $C_{11}H_{16}NO_2$ $(M + H)^+$ 194.1181, found $(M + H)^+$ 194.1190.

4-(2-Propenyl)-1-aza-6-oxabicyclo[5.2.0]non-3-en-9-one (90): colorless oil (74%); TLC R_f 0.42 (Et_2O); IR (film) 1762 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ 5.70 (t, J = 5.0 Hz, 1H), 5.29 (dd, J = 1.0, 3.5 Hz, 1H), 4.93 (s, 2H), 4.62 (d, J = 15.5

Hz, 1H), 4.45 (dd, J = 5.5, 19.0 Hz, 1H), 4.35 (d, J = 15.0 Hz, 1H), 3.77 (dd, J = 0.5, 19.0 Hz, 1H), 3.13 (ddd, J = 1.5, 3.5, 15.0 Hz, 1H), 2.90 (dd, J = 1.5, 15.0 Hz, 1H), 1.87 (s, 3H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 165.5, 142.9, 141.1, 123.4, 112.6, 82.3, 64.0, 44.1, 41.2, 21.8; MS (CI, NH_3) m/z 197 $(M + NH_4)^+$, 180 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{10}H_{14}NO_2$ $(M + H)^+$ 180.1025, found $(M + H)^+$ 180.1024.

4-[1-(*tert*-Butyldimethylsilyl)oxy-2-propenyl]-1-aza-6-oxabicyclo[5.2.0]non-3-en-9-one (91): colorless oil (82%); TLC R_f 0.40 (3:1 Et_2O/hexanes); IR (film) 1768 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 5.61−5.58 (m, 1H), 5.28−5.21 (m, 2H), 5.03 (s, 1H), 4.45 (dd, J = 5.0, 19.0 Hz, 1H), 4.29 (dd, J = 1.0, 15.5 Hz, 1H), 4.23 (d, J = 15.0 Hz, 1H), 4.20 (s, 2H), 3.74 (d, J = 19.0 Hz, 1H), 3.13 (ddd, J = 1.5, 3.5, 15.5 Hz, 1H), 2.89 (d, J = 15.0 Hz, 1H), 0.89 (s, 9H), 0.07 (s, 6H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 165.4, 146.9, 139.4, 123.3, 111.5, 82.3, 65.1, 64.5, 44.1, 41.3, 25.9, 18.4, −5.3; MS (CI, NH_3) m/z 327 $(M + NH_4)^+$, 310 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{16}H_{28}NO_3Si$ $(M + H)^+$ 310.1838, found $(M + H)^+$ 310.1838. Anal. Calcd for $C_{16}H_{27}NO_3Si$: C, 62.50; H, 8.20; N, 4.56. Found: C, 62.70; H, 8.30; N, 4.55.

4-[1-(Hydroxy-2-propenyl]-1-aza-6-oxabicyclo[5.2.0]non-3-en-9-one (92): colorless oil (32%); TLC R_f 0.27 (EtOAc); IR (film) 3435, 1747 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.75−5.73 (m, 1H), 5.31 (dd, J = 0.5, 3.5 Hz, 1H), 5.20 (s, 1H), 5.09 (s, 1H), 4.60 (d, J = 15.5 Hz, 1H), 4.46 (dd, J = 5.0, 19.0 Hz, 1H), 4.32 (d, J = 15.5 Hz, 1H), 4.25 (s, 2H), 3.78 (d, J = 19.0 Hz, 1H), 3.15 (ddd, J = 1.5, 3.5, 15.0 Hz, 1H), 2.91 (d, J = 15.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.4, 147.3, 139.1, 124.4, 112.8, 82.3, 64.7, 64.6, 44.1, 41.3; MS (CI, NH_3) m/z 213 $(M + NH_4)^+$, 196 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{10}H_{14}NO_3$ $(M + H)^+$ 196.0973, found $(M + H)^+$ 196.0971.

(6*R*,7*S*)-(+)-7-[(1*R*)-(*tert*-Butyldimethylsilyl)oxyethyl]-3-(2-propenyl)-1-azabicyclo[4.2.0]oct-3-en-8-one (93): colorless oil (100%); TLC R_f 0.46 (1:1 Et_2O/hexanes); $[\alpha]^{25}_D$ = +107.0 (c 0.58, $CHCl_3$); IR (film) 1756 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 5.94−5.91 (m, 1H), 4.87 (s, 1H), 4.85 (s, 1H), 4.25 (d, J = 16.5 Hz, 1H), 4.22−4.15 (m, 1H), 3.62 (d, J = 17.0 Hz, 1H), 3.46−3.40 (m, 1H), 2.75 (dd, J = 1.5, 5.5 Hz, 1H), 2.58−2.49 (m, 1H), 2.29−2.25 (m, 1H), 1.88 (s, 3H), 1.21 (d, J = 6.0 Hz, 3H), 0.85 (s, 9H), 0.05 (s, 6H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 167.0, 140.6, 132.7, 121.4, 111.0, 66.7, 65.8, 45.5, 38.3, 28.7, 25.8, 22.9, 20.7, 18.0, −4.1, −5.0; MS (CI, NH_3) m/z 339 $(M + NH_4)^+$, 322 $(M + H)^+$; HRMS (CI) calcd for $C_{18}H_{32}NO_2Si$ $(M + H)^+$ 322.2202, found $(M + H)^+$ 322.2207.

(6*R*,7*S*)-(+)-7-[(1*R*)-(*tert*-Butyldimethylsilyl)oxyethyl]-3-[1-(*tert*-butyldimethylsilyl)oxy-2-propenyl]-1-azabicyclo-[4.2.0]oct-3-en-8-one (94): colorless oil (72%); TLC R_f 0.57 (1:1 Et_2O/hexanes); $[\alpha]^{25}_D$ = +72.0 (c 1.15, $CHCl_3$); IR (film) 1757 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 5.90−5.87 (m, 1H), 5.24 (s, 1H), 5.00 (s, 1H), 4.34−4.15 (m, 4H), 3.61, (br d, J = 17.0, 1H), 3.47, 3.42 (m, 1H), 2.75 (dd, J = 1.5, 5.5 Hz, 1H), 2.59−2.47 (m, 1H), 2.34−2.13 (m, 1H), 1.23 (d, J = 6.0 Hz, 3H), 0.91 (s, 9H), 0.86 (s, 9H), 0.07 (s, 6H), −0.01 (s, 6H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 167.0, 143.8, 130.6, 120.5, 110.1, 66.9, 65.8, 63.8, 45.6, 38.6, 28.6, 26.0, 25.8, 22.9, 18.4, 18.0, −4.1, −4.9, −5.3; MS (CI, NH_3) m/z 339 $(M + NH_4)^+$, 322 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{24}H_{46}NO_3Si_2$ $(M + H)^+$ 452.3016, found $(M + H)^+$ 452.2997.

1-(*tert*-Butyldimethylsilyl)-4-[2-(2,5-dihydro-3-furanyl)-2-propenyl-1-oxy]azetidin-2-one (95): colorless oil (100%); TLC R_f 0.48 (3:1 Et_2O/hexanes); IR (film) 1757 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 5.89 (s, 1H), 5.26 (s, 1H), 4.96 (dd, J = 1.0, 3.5 Hz, 1H), 4.90 (s, 1H), 4.74 (s, 4H), 4.17 (s, 2H), 3.09 (dd, J = 3.5, 15.0 Hz, 1H), 2.85 (dd, J = 1.0, 15.0 Hz, 1H), 0.93 (s, 9H), 0.22 (s, 3H), 0.20 (s, 3H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 170.7, 136.9, 136.3, 122.7, 115.0, 79.3, 76.7, 75.1, 67.7, 45.8, 26.1, 18.2, −5.8, −6.1; MS (CI, NH_3) m/z 327 $(M + NH_4)^+$, 310 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{16}H_{28}NO_3Si$ $(M + H)^+$ 310.1839, found $(M + H)^+$ 310.1831.

1-Benzyl-4-[2-(2,5-dihydro-3-furanyl)-2-propenyl-1-oxy]-azetidin-2-one (96): colorless oil (98%); TLC R_f 0.39 (Et_2O); IR (film) 1758 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 7.38−7.25 (m, 5H), 5.77 (s, 1H), 5.12 (s, 1H), 4.92 (dd, J = 1.5, 3.5 Hz, 1H), 4.86 (s, 1H), 4.72 (s, 4H), 4.57 (d, J = 15.0 Hz, 1H), 4.22 (d, J = 15.5 Hz, 1H), 4.12 (d, J = 12.5 Hz, 1H), 4.07 (d, J = 12.5 Hz, 1H), 3.06 (dd, J = 3.5, 15.0 Hz, 1H), 2.89 (d, J = 15.0 Hz, 1H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 166.0, 136.7, 136.2, 135.8, 128.9, 128.3, 127.9, 122.9, 115.4, 80.8, 76.7, 75.1, 68.8, 44.8, 44.4; MS (CI, NH_3) m/z 303 $(M + NH_4)^+$, 286 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{17}H_{20}NO_3$ $(M + H)^+$ 286.1443, found $(M + H)^+$ 286.1451.

4-(2,5-Dihydro-3-furanyl)-6-oxa-1-azabicyclo[5.2.0]non-3-en-9-one (97): white solid (64%); mp 73−74 °C; TLC R_f 0.16 (Et_2O); IR (KBr) 1766 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 5.82 (s, 1H), 5.36−5.27 (m, 2H), 4.74−4.67 (m, 4H), 4.48 (dd, J = 5.0, 19.5 Hz, 1H), 4.44−4.39 (m, 2H), 3.79 (d, J = 19.0 Hz, 1H), 3.17 (ddd, J = 1.5, 2.0, 15.0 Hz, 1H), 2.93 (d, J = 15.0 Hz, 1H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 165.4, 138.0, 133.4, 124.8, 121.4, 82.3, 76.6, 75.2, 63.9, 44.0, 41.2; MS (CI, NH_3) m/z 225 $(M + NH_4)^+$, 208 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{11}H_{14}NO_3$ $(M + H)^+$ 208.0974, found $(M + H)^+$ 208.0976.

(6*R*,7*S*)-(+)-7-[(1*R*)-(*tert*-Butyldimethylsilyl)oxyethyl]-3-[(2,5-dihydro)-3-furanyl]-1-azabicyclo[4.2.0]oct-3-en-8-one (98): colorless oil (67%); TLC R_f 0.10 (1:1 Et_2O/hexanes); $[\alpha]^{24}_D$ = +98.0 (c 2.00, $CHCl_3$); IR (film) 1754 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ 5.71 (s, 1H), 5.61−5.59 (m, 1H), 4.78−4.73 (m, 4H), 4.29 (d, J = 17.0 Hz, 1H), 4.24−4.18 (m, 1H), 3.67 (d, J = 17.0 Hz, 1H), 3.52−3.47 (m, 1H), 2.78 (dd, J = 1.5, 5.5 Hz, 1H), 2.62−2.44 (m, 1H), 2.39−2.21 (m, 1H), 1.24 (d, J = 6.0 Hz, 3H), 0.88 (s, 9H), 0.08 (s, 6H); ^{13}C NMR (67.5 MHz, $CDCl_3$) δ 166.9, 137.0, 126.5, 122.5, 120.0, 76.6, 74.8, 66.8, 65.6, 45.6, 38.4, 28.4, 25.7, 22.8, 17.9, −4.2, −5.0; MS (CI, NH_3) m/z 395 $(M + NH_4)^+$, 378 $(M + H)^+$; HRMS (CI, NH_3) calcd for $C_{19}H_{32}NO_3Si$ $(M + H)^+$ 350.2465, found $(M + H)^+$ 350.2151.

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Supporting Information Available: Copies of ^{1}H and ^{13}C NMR spectra of **6**, **8**−**11**, **16**−**18**, **21** (^{1}H NMR only), **23**−**24, 25**−**26** (^{1}H NMR only), **27a**, **28**, **29a**−**b**, **30a**−**b**, **32a**−**b**, **38b**−**c**, **39**, **40**, **42**, **44**, **45**, **47**−**50**, **52**−**55**, **58**, **59**, **61**, **66**, **67**, **70**−**75**, **76c**, **77c**, **78a**, **80**, **82**−**86**, **89**, **90**, and **92**−**98** and X-ray crystallographic data and ORTEP diagrams for **42**, **51**, and **69** (143 pages). This material is contained in libraries on microfiche, immediately follows this article in the microfilm version of the journal, and can be ordered from the ACS; see any current masthead page for ordering information.

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