JOC Article

Radical Carbonylation/Reductive Cyclization for the Construction of Tetrahydrofuran-3-ones and Pyrrolidin-3-ones

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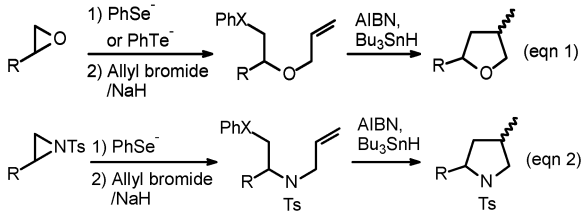
β-Hydroxyalkyl aryl chalcogenides obtained by regioselective ring-opening of epoxides with benzeneselenolate or -tellurolate were found to undergo efficient hetero-Michael addition when treated with ethyl propiolate. Subsequent carbonylation/reductive cyclization of the resulting vinylogous carbonates in the presence of AIBN/TTMSS and carbon monoxide (80 atm) afforded 2,5-disubstituted tetrahydrofuran-3-ones, predominantly as cis isomers (cis/trans = 4/1−9/1). Starting from a polymer-supported diaryl diselenide, the methodology was also successfully extended to solid-phase synthesis. Vinylogous carbamates prepared by hetero-Michael addition of aziridines to electron-deficient alkynes were regioselectively ring-opened with benzeneselenolate from the sterically least hindered side. Radical carbonylation/reductive cyclization of the resulting *N*-vinyl-β-amino-alkyl phenyl selenides afforded 2,5-disubstituted pyrrolidin-3-ones, predominantly as cis isomers (cis/trans = 3/1−12/1).

Introduction

Acyl radicals[1] take part in a large variety of inter- and intramolecular reactions and are therefore useful synthetic intermediates.[2] These species are commonly generated by homolysis of an acyl-X bond, where X could be hydrogen, halogen, chalcogen, or a metal. Since most of these derivatives suffer from certain shortcomings in radical processes (inefficient chain transfer, over-reduction, or poor reactivity toward stannyl or silyl radicals), selenol esters have become the most versatile precursors for acyl radicals.[3] As demonstrated by Ryu and coworkers, acyl radicals can also be readily formed by reaction of alkyl radicals with carbon monoxide.[1,4,5] These carbonylation reactions are commonly performed as one-pot reactions with 60−80 atm of CO, conditions that can easily be obtained by using an ordinary autoclave. Primary, secondary, and tertiary radicals can be efficiently carbonylated and further transformed in more or less elaborate ways into carbonyl derivatives such as aldehydes,[6] ketones,[7] esters,[8] lactones,[9] thiolactones,[10] amides,[11] lactams,[12] and acyl selenides.[13] Atom or group transfer, inter- or intramolecular radical addition, cascade reactions, radical translocation, one-electron oxidation, or ionic chemistry are involved in some of these transformations.



It occurred to us that radical carbonylation/reductive cyclization could provide easy access to tetrahydrofuran-3-one and pyrrolidin-3-one derivatives from readily avail-

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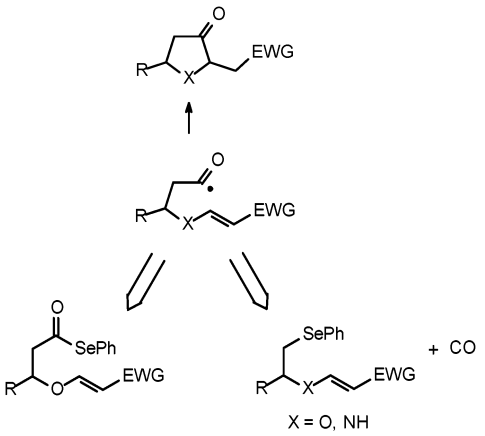
SCHEME 1

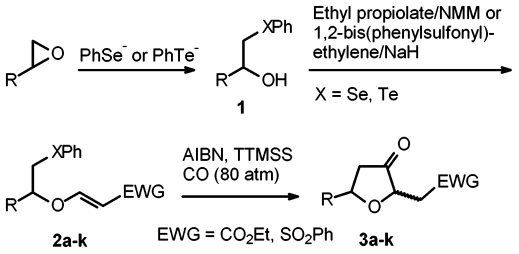


able organochalcogen precursors. Some time ago, we showed that epoxides can be regioselectively ring-opened by benzeneselenolate or benzenetellurolate. The resulting *β*-hydroxyalkyl phenyl chalcogenides were readily O-allylated by treatment with allyl bromide and base. Subsequent reductive cyclization with AIBN initiation then provided 2,4-disubstituted tetrahydrofurans in high yields as mixtures of diastereomers (eq 1).[14] In a similar fashion, *N*-tosyl aziridines were regioselectively ring-opened by benzeneselenolate and, following N-allylation, cyclized to give *N*-tosyl-2,4-disubstituted pyrrolidines (eq 2).[15] Evans and co-workers have already shown that 2,5-disubstituted tetrahydrofuran-3-ones can be efficiently prepared by intramolecular acyl radical cyclization (Scheme 1; X = O, lower left).[16,17] By generating acyl radicals directly from alkyl radicals and carbon monoxide (Scheme 1; lower right), we thought we could approach these systems, as well as the corresponding pyrrolidin-3-ones (X = NH) in a different way, using more readily available starting materials.[18]

Results and Discussion

Construction of Tetrahydrofuran-3-ones. For the preparation of 2,5-disubstituted tetrahydrofuran-3-ones by radical carbonylation/reductive cyclization, *β*-hydroxyalkyl phenyl selenides **1** were allowed to undergo hetero-Michael addition to ethyl propiolate[16a−c,17b,19] or to act as nucleophiles in the vinylogous substitution of (*E*)-1,2-bis(phenylsulfonyl)ethylene[16d,17a] (Scheme 2). The O-vinylation proceeded smoothly at room temperature to give radical precursors **2a**−**k** in 64−95% isolated yields as

SCHEME 2



pure (*E*)-isomers (Table 1). Attempts to vinylate ring-opened isobutylene oxide failed, and the starting material was recovered unchanged.

Carbonylation/radical cyclization was conducted in an autoclave. To elucidate the optimal reaction conditions, selenide **2a** was carbonylated/cyclized under various conditions (Table 2). The use of tri-*n*-butyltin hydride as a hydrogen atom donor was found to cause formation of substantial amounts of reduced product **4**. Better results were obtained using tris(trimethylsilyl)silane (TTMSS). With carbon monoxide at 80 atm, an 86% yield of compound **3a** was obtained as a 9:1 mixture of cis and trans isomers. At lower pressures (60 atm), reduction of the starting material again started to become an unwanted side-reaction. Tri-*n*-butylgermane was also tried as a hydrogen atom donor but found to be inferior to TTMSS. Thus, subsequent carbonylation/reductive cyclization of the remaining compounds **2** (Scheme 2) was carried out using TTMSS as a hydrogen atom donor and CO at 80 atm.

As shown in Table 1 (entries 1−4), organotellurium radical precursors **2b** and **2d** performed almost as well as their organoselenium counterparts in the chemistry developed. The unexpectedly low yield of compound **3c** (40%) was due to competing 1,5 hydrogen atom shift/6-*exo*-cyclization (Scheme 3). This gave rise to dioxane **5**, which was isolated in 32% yield starting from compound **2c** and 30% yield starting from **2d**. Only one diastereomer of the compound was isolated with all three substituents occupying equatorial positions. In a separate experiment carried out in the absence of CO, the dioxane derivative **5** was isolated in 82% yield along with 3% reduced starting material.

It is also noteworthy that the benzylic radical formed from compound **2e** (Table 1, entry 5) failed to undergo carbonylation/5-*exo*-cyclization. Only reduced starting material was isolated in 56% yield. However, this is in accord with previous observations that carbonylated stabilized radicals (benzylic, allylic, tertiary) decarbonylate more rapidly than other acyl radicals.[1] Carbonylation/cyclization of radical precursors **2f**−**i** afforded 2,5-disubstituted tetrahydrofuran-3-ones in moderate to good yields (59−82%). Small amounts (6−14%) of reduced starting material were formed in the reactions. The *β*-hydroxyalkyl phenyl selenides originating from 1,2-disubstituted epoxides (Table 1, entries 10−11) furnished 2,4,5-trisubstituted tetrahydrofuran-3-ones as inseparable mixtures of diastereomers. It seems that carbonylation of the secondary alkyl radical produces both of the possible diastereomeric acyl radicals (cis/trans), which then undergo 5-*exo*-cyclization.

The stereochemistry of the 2,5-disubstituted tetrahydrofuran-3-ones **3** prepared was assigned by using two-

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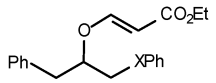
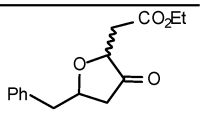
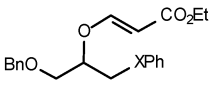
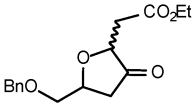
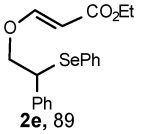
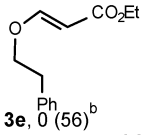
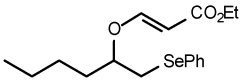
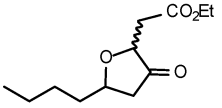
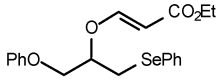
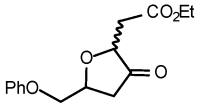
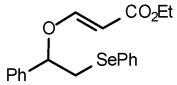
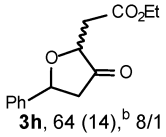
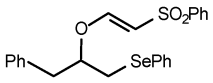
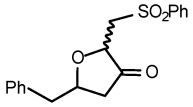
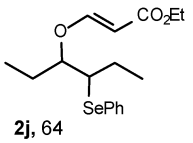
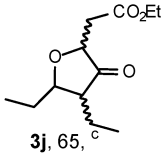
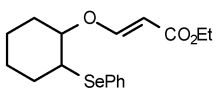
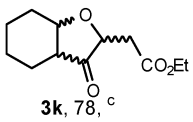
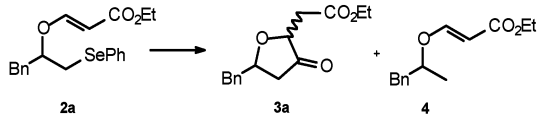
TABLE 1. O-Vinylated β-Hydroxyalkyl Phenyl Chalcogenides and Their Conversion to Tetrahydrofuran-3-ones via Radical Carbonylation/ Reductive Cyclization

Entry	*O*-Vinylated β-hydroxyalkyl phenyl chalcogenide, compound no., yield[a]	Tetrahydrofuran-3-one, compound no., yield,[a] *cis*/*trans*-ratio
1	 **2a,** X = Se, 95	 **3a**, 86, 9/1
2	**2b**, X = Te, 64	**3a**, 81, 9/1
3	 **2c**, X = Se, 83	 **3c**, 42, 7/1
4	**2d**, X = Te, 80	**3c**, 40, 7/1
5	 **2e,** 89	 **3e**, 0 (56)[b]
6	 **2f,** 76	 **3f**, 77 (10),[b] 7/1
7	 **2g,** 85	 **3g**, 82 (6),[b] 4/1
8	 **2h,** 83	 **3h**, 64 (14),[b] 8/1
9	 **2i,** 79	 **3i**, 59, 9/1
10	 **2j,** 64	 **3j**, 65,[c]
11	 **2k,** 86	 **3k**, 78,[c]

[a] Isolated yields (%). [b] Isolated yield of reduced uncyclized material (%). [c] Inseparable mixture of diastereomers.

dimensional NOESY experiments. A 1,3 NOE was always seen for the cis isomer. The selective formation of cis-2,5-disubstituted tetrahydrofuran-3-ones follows from the Beckwith−Houk rules for ring-closure, assuming that the 2,5-substituents are both pseudoequatorial in the chair-like transition state.[20] Some attempts were made to epimerize the diastereomeric mixture of cis-/trans-2,5-

TABLE 2. Optimization of Carbonylation Conditions

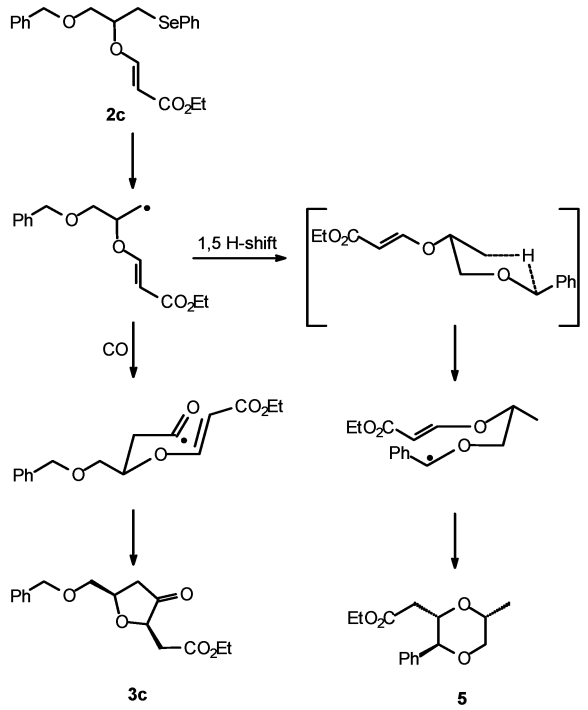


2a 3a 4

entry	H-atom donor	P_{CO} (atm)	**3a**[a]	**4**[a]
1	*n*-Bu_3SnH	60	20	66
2	TTMSS	60	81	6
3	TTMSS	80	86	0
4	*n*-Bu_3GeH	80	86	6

[a] Isolated yield in percent.

SCHEME 3



disubstituted tetrahydrofuran-3-ones. However, by treatment of compound **3a** with 0.1 equiv of DBU in refluxing benzene, the cis/trans ratio could only be increased to 1:2.

Solid-Phase Synthesis of Tetrahydrofuran-3-ones. Polymer-supported selenium linkers were introduced by Nicolaou[21] in 1998. They have already found extensive use in combinatorial chemistry,[22] as well as in natural product synthesis.[23] Although an increasing number of radical reactions on solid support[24] have been

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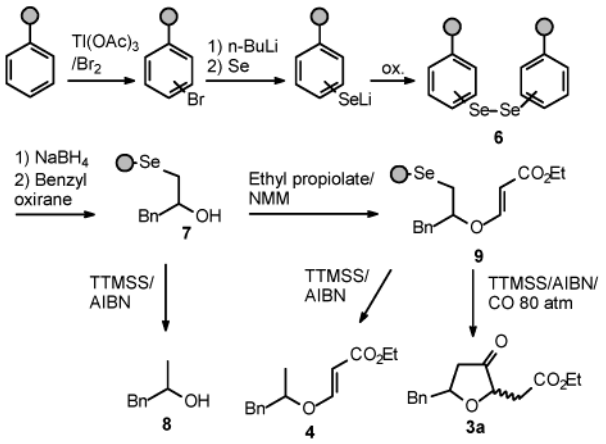
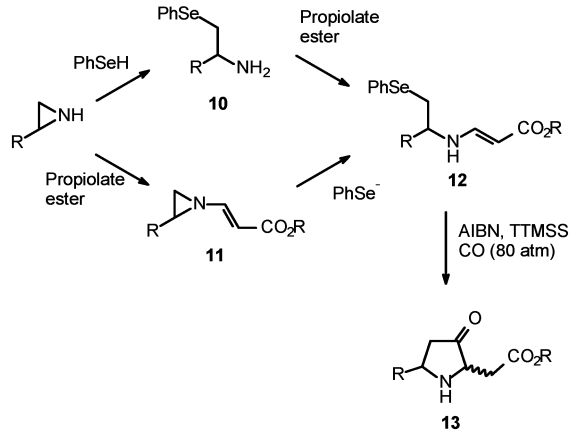
SCHEME 4



SCHEME 5



desribed, they have, to the best of our knowledge, never been applied to radical carbonylation/reductive cyclization reactions. We therefore thought it would be interesting to try to extend the methodology developed for the preparation of tetrahydrofuran-3-ones to the solid phase.

A diselenide resin **6** was prepared in a few steps from cross-linked (1%) polystyrene as described in the literature.[25] The loading of selenium, based on elemental analysis for nitrogen after treatment of the polymer with $NaBH_4$/*N*,*N*-dimethyl carbamoyl chloride, was 2.25 mmol/g resin.

Benzyloxirane was added to an ethanolic suspension of resin **6**, which had been treated with $NaBH_4$ (Scheme 4). The resulting β-hydroxyalkyl phenyl selenide resin **7** was then purified by washing and filtration. To show that the reaction had proceeded well, the resin was treated in refluxing benzene with a large excess of TTMSS/AIBN. On the basis of the loading of selenium on the resin, the alcohol **8** was isolated in near quantitative yield (97%). The Michael addition of alcohol resin **7** was carried out with ethyl propiolate in neat NMM at 60° C. Reductive cleavage of resin **9** as described above provided vinylogous carbonate **4** in excellent yield (91%). The resin bound radical precursor was finally subjected to radical carbonylation/reductive cyclization. Initial attempts with a low concentration (0.01 M) of hydrogen atom donor resulted in poor conversion. When a more concentrated solution (0.1 M) was used, tetrahydrofuran-3-one **3a** was isolated in 55% yield, along with 12% of vinylogous carbonate **4** (yields were calculated over three steps). Attempts to further increase the concentration of TTMSS (0.3 M) resulted in lower product yields (37%) and formation of more reduced material (42%).

Construction of Pyrrolidin-3-ones. For extending the methodology developed to the preparation of 2,5-disubstituted pyrrolidin-3-ones, we envisioned a reaction sequence involving benzenselenol ring-opening of an aziridine,[26] Michael addition[27] of the resulting β-aminoalkyl phenyl selenide to a propiolate ester, and carbonylation/reductive[28] cyclization (Scheme 5, upper pathway). However, it turned out that the conjugate addition products from primary amines were labile and more difficult to isolate than those from the corresponding alcohols. Thus, ring-opened 2-benzylaziridine (**10**, R = Bn) reacted only sluggishly with methyl propiolate/NMM when heated in DMF at 80 °C to give a complex mixture of products. The corresponding N-tosylated compound was found to be even less reactive. Spurred by the early finding by Vessiere and co-workers[29] that electron-deficient aziridines undergo efficient Michael addition to electron-deficient alkynes by gentle heating in polar solvents, we tried to add aziridines to methyl propiolate. The idea was to regiospecifically ring-open the resulting N-vinylated aziridine **11** from the sterically least hindered side to obtain the desired radical precursor **12** for carbonylation/radical cyclization (Scheme 5, lower part). Simple stirring of neat 2-benzylaziridine with a slight excess of methyl propiolate provided a quantitative yield of adduct **11a** as a 6/1 mixture of (*E*)- and (*Z*)-isomers (Table 3). Other mono- and disubstituted aziridines reacted similarly. Dimethyl acetylene dicarboxylate (entry 5) and ethynyl *p*-tolyl sulfone (entry 6) could also be

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(28) Radical cyclization onto derivatives of vinylogous carbamates has been frequently used in heterocycle construction.[28a] However, additions of acyl radicals are scarce.[28b] (a) Navarro-Vázquez, A.; Garcia, A.; Dominguez, D. *J. Org. Chem.* **2002**, *67*, 3213 and references therein. (b) Evans, P. A.; Manangan, T.; Rheingold, A. L. *J. Am. Chem. Soc.* **2000**, *122*, 11009.

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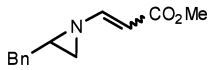
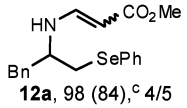
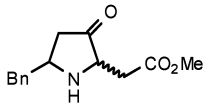
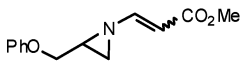
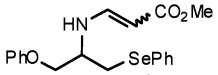
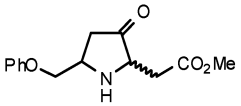
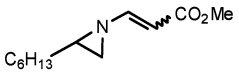
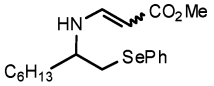
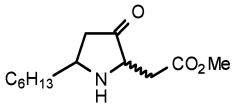
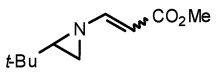
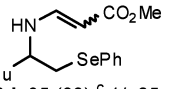
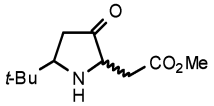
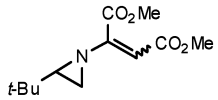
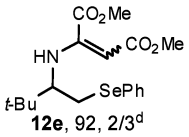
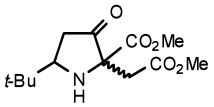
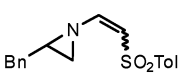
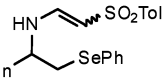
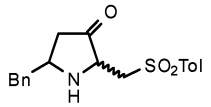
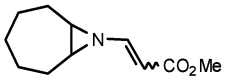
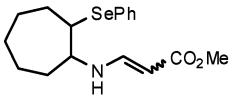
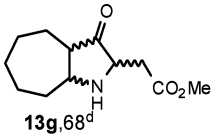
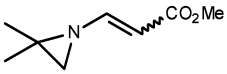
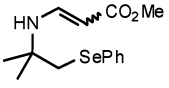
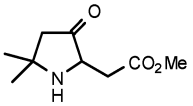
TABLE 3. Vinyl Aziridines: Their Ring-Opening with Benzeneselenolate and Conversion to Pyrrolidin-3-ones

Entry	*N*-Vinyl aziridin, compound no., yield,[a] *E*/*Z*-ratio	*N*-Vinylated β-aminoalkyl phenyl selenide, compound no., yield,[b] *E*/*Z*-ratio	Pyrrolidin-3-one, compound no., yield,[a] *cis*/*trans*-ratio
1	 **11a**, quant., 6/1	 **12a**, 98 (84),[c] 4/5	 **13a**, 79, 4/1
2	 **11b**, quant., 6/1	 **12b**, 96 (73),[c] 2/1	 **13b**, 85, 3/1
3	 **11c**, quant., 7/3	 **12c**, 96 (85),[c] 1/4	 **13c**, 65, 3/1
4	 **11d**, quant., 9/1	 **12d**, 95 (83),[c] 1/>25	 **13d**, 71, 12/1
5	 **11e**, quant., 10/1	 **12e**, 92, 2/3[d]	 **13e**, 68, 1/>25
6	 **11f**, quant., 1/>25	 **12f**, 92, 9/1	 **13f**, 0
7	 **11g**, quant., 5/1	 **12g**, 92, 2/5	 **13g**,68[d]
8	 **11h**, quant., 5/1	 **12h**, 95, 2/3	 **13h**,65

[a] Isolated yield (%). [b] Crude yield (%) after $NaBH_4/BrCH_2CO_2H$ treatment. [c] Isolated yield (%) after flash chromatography on Al_2O_3 (neutral). [d] Stereochemistry not determined.

used as Michael acceptors. Except for sulfone **11f**, the addition products were always enriched in the (*E*)-isomer.

The *N*-vinyl aziridines **11** were then subjected to ring-opening (Scheme 5). Whereas N-unsubstituted aziridines have to be protonated before they can be ring-opened by chalcogen nucleophiles, aziridines carrying electron-withdrawing N-substituents react in the unprotonated form. After treatment of the aziridine with benzeneselenolate at ambient temperature in ethanol, complete and regiospecific ring-opening was usually seen after 2−3 h (Table 3). The only exception was the 8-azabicyclo[5.1.0]-octane-derived *N*-vinylaziridine **11g**, which required heating at reflux in methanol for 3 h to undergo ring-opening. Judging from the ^1H NMR spectra of the crude product, the vinylogous carbamates **12** were only contaminated by some diphenyl diselenide. However, attempted purification on silica caused extensive decomposition of the materials. Purification on neutral alumina worked better, producing pure enamines. Treatment of the crude product with sodium borohydride in ethanol until colorlessness was achieved and addition of excess bromoacetic acid as a trapping agent for benzeneselenolate turned out to be a more practical way of removing the diphenyl diselenide.[30] After final extraction with base, crude vinylogous carbamates **12** were obtained in 92−98% yields (Table 3). Ring-opening was accompanied by double-bond isomerization of the enamine moiety. Thus, compound **11a** as a 6/1 *E*/*Z* mixture afforded enamine **12a** as a 4/5 *E*/*Z* mixture. Ring-opening of both the pure (*E*)- and (*Z*)-isomers of compound **11a** produced the same mixture (*E*/*Z* = 4/5) of compound **12a**, suggesting that the product ratio is thermodynamically controlled. As

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seen in Table 3, the *E*/*Z* ratios of vinylaziridines varied dramatically for the compounds prepared (e.g., *E*/*Z* = 9/1 for vinylic sulfone **12f** and *E*/*Z* = 1/>25 for acrylate **12d**).

Radical carbonylation/reductive cyclization was conducted at 80 °C with carbon monoxide at 80 atm. In terms of avoiding formation of reduced starting material, TTMSS again proved to be superior to *n*-Bu_3SnH as a hydrogen atom donor. Pyrrolidin-3-ones **13** were isolated in 65−85% yields, often as a mixture of cis and trans isomers (Table 3). For some reason, radical precursor **12f**, carrying a vinyl sulfone acceptor, failed to give a pyrrolidin-3-one on attempted radical carbonylation/reductive cyclization. Except for compound **13e** (cis/trans = 12/1), cis/trans ratios were in many cases close to 3/1 for 2,5-disubstituted compounds. Thus, the selectivity is lower than that seen above in the preparation of 2,5-disubstituted tetrahydrofuran-3-ones **2** (Table 1, entries 1, 6, 7). Evans and co-workers[28b] have recently reported the formation of a trans-2,5-disubstituted pyrrolidin-3-one as the major diastereomer (cis/trans = 1/2) in the intramolecular addition of an acyl radical to a vinylogous carbamate. However, this change in diastereoselectivity can probably be ascribed to an additional N-substituent (benzyl), which could change the diastereoselectivity predicted assuming a chairlike Beckwith−Houk transition state.[31] Some of the pyrrolidin-3-ones prepared were surprisingly unstable (**13a**−**c**). Significant decomposition was observed already when the compounds were kept for a few days in the refrigerator. N-Tosylation was found to significantly improve their stability and allow for proper characterization.

In conclusion, novel methodology for the preparation of tetrahydrofuran-3-ones and pyrrolidin-3-ones from readily available starting materials was developed, using radical carbonylation/reductive cyclization in the key step. The required radical precursors were obtained either from epoxides via regioselective benzenselenolate ring-opening and O-vinylation (for tetrahydrofuran-3-ones) or from aziridines via N-vinylation and regioselective benzeneselenolate ring-opening (for pyrrolidin-3-ones).

Experimental Section

Coupling constants given below are in hertz (Hz).

Typical Procedure for Conjugate Addition of β-Hydroxylalkyl Phenyl Chalcogenides to Ethyl Propiolate: Preparation of (*E*)-3-(1-Benzyl-2-phenylselenenyl-ethoxy)-acrylic Acid Ethyl Ester (2a). To a stirred solution of 2-hydroxy-3-phenylpropyl phenyl selenide (0.965 g, 3.3 mmol) in CH_2Cl_2 (10 mL) was added *N*-methyl morpholine (NMM) (0.546 mL, 5.0 mmol). Ethyl propiolate (0.503 mL, 5.0 mmol) was then added, and the resulting mixture was stirred for 6 h at room temperature. The solvent was removed in vacuo, and the residue was purified by flash chromatography (5−10% ethyl acetate/pentane) to afford 1.23 g (95%) of the title compound as a yellowish oil: ^{1}H NMR ($CDCl_3$) δ 1.25 (t, J = 7.1, 3H), 2.97−3.10 (several peaks, 4H), 4.12 (q, J = 7.1, 2H), 4.22 (m, 1H), 5.12 (d, J = 12.4, 1H), 7.14 (m, 2H), 7.21−7.31 (several peaks, 6H), 7.34 (d, J = 12.4, 1H), 7.48 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 14.3, 30.9, 40.2, 59.7, 83.5, 97.7, 126.9, 127.5, 128.6, 129.1, 129.3, 129.5, 133.2, 136.3, 161.6, 167.8; IR (neat) 1132, 1629, 1705 cm^{-1}. Anal. Calcd for $C_{20}H_{22}O_3Se$: C, 61.70; H, 5.70. Found: C, 61.60; H, 5.63.

Spectral data for compounds **2** prepared are shown below. For yields, see Table 1.

(*E*)-3-(1-Benzyl-2-phenyltellurenyl-ethoxy)-acrylic Acid Ethyl Ester (2b): ^{1}H NMR ($CDCl_3$) δ 1.27 (t, J = 7.1, 3H), 3.00−3.12 (several peaks, 4H), 4.14 (q, J = 7.1, 2H), 4.32 (m, 1H) 5.14 (d, J = 12.4, 1H), 7.13−7.33 (several peaks, 8H), 7.36 (d, J = 12.4, 1H), 7.75 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 12.3, 14.4, 41.3, 59.7, 84.6, 97.9, 111.1, 126.8, 128.1, 128.6, 129.4, 129.5, 136.4, 138.8, 161.3, 167.7; IR (neat) 1129, 1637, 1701 cm^{-1}. Anal. Calcd for $C_{20}H_{22}O_3Te$: C, 54.85; H, 5.06. Found: C, 54.66; H, 5.25.

(*E*)-3-(1-Benzyloxymethyl-2-phenylselenenyl-ethoxy)-acrylic Acid Ethyl Ester (2c): ^{1}H NMR ($CDCl_3$) δ 1.26 (t, J = 7.1, 3H), 3.08 (dd, J = 13.1, 6.5, 1H), 3.15 (dd, J = 13.1, 6.3, 1H), 3.64 (dd, J = 10.5, 6.5, 1H), 3.69 (dd, J = 10.5, 4.0, 1H), 4.15 (q, J = 7.1, 2H), 4.19 (m, 1H), 4.49 (s, 2H), 5.18 (d, J = 12.3, 1H), 7.26−7.36 (several peaks, 8H), 7.48 (d, J = 12.3, 1H), 7.51 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 14.4, 28.1, 59.7, 70.5, 73.4, 81.8, 98.0, 127.6, 127.7, 128.4, 129.1, 129.3, 133.3, 137.5, 161.8, 167.7; IR (neat) 1132, 1369, 1641, 1703 cm^{-1}. Anal. Calcd for $C_{21}H_{24}O_4Se$: C, 60.14; H, 5.77. Found: C, 60.06; H, 5.65.

(*E*)-3-(1-Benzyloxymethyl-2-phenyltellurenyl-ethoxy)-acrylic Acid Ethyl Ester (2d): ^{1}H NMR ($CDCl_3$) δ 1.25 (t, J = 7.1, 3H), 3.07 (dd, J = 12.6, 6.1, 1H), 3.13 (dd, J = 12.6, 3.9, 1H), 3.65 (dd, J = 10.4, 5.4, 1H), 3.86 (dd, J = 10.4, 4.4, 1H), 4.14 (q, J = 7.1, 2H), 4.27 (m, 1H), 4.48 (s, 2H), 5.17 (d, J = 12.4, 1H), 7.17−7.37 (several peaks, 8H), 7.48 (d, J = 12.4, 1H), 7.74 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 8.7, 14.3, 59.7, 71.6, 73.4, 82.7, 98.0, 127.7, 127.8, 128.1, 128.3, 128.4, 129.4, 137.5, 138.8, 161.6, 167.7; IR (neat) 1133, 1640, 1705 cm^{-1}. Anal. Calcd for $C_{21}H_{24}O_4Te$: C, 53.89; H, 5.17. Found: C, 53.71; H, 5.18.

(*E*)-3-(2-Phenyl-2-phenylselenenyl-ethoxy)-acrylic Acid Ethyl Ester (2e): ^{1}H NMR ($CDCl_3$) δ 1.27 (t, J = 7.1, 3H), 4.16 (q, J = 7.1, 2H), 4.25 (m, 1H), 4.39 (dd, J = 10.4, 9.2, 1H), 4.56 (dd, J = 9.2, 5.5, 1H), 5.16 (d, J = 12.7, 1H), 7.23−7.38 (several peaks, 8H), 7.49 (d, J = 12.7, 1H), 7.53 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 14.3, 45.3, 59.7, 73.0, 96.9, 127.6, 127.7, 128.1, 128.4, 128.6, 128.7, 129.1, 135.5, 161.5, 167.4; IR (neat) 1133, 1625, 1708 cm^{-1}. Anal. Calcd for $C_{19}H_{20}O_3Se$: C, 60.80; H, 5.37. Found: C, 60.71; H, 5.42.

(*E*)-3-(1-Phenylselenenylmethyl-pentyloxy)-acrylic Acid Ethyl Ester (2f): ^{1}H NMR ($CDCl_3$) δ 0.89 (m, 3H), 1.27 (t, J = 7.1, 3H), 1.20−1.40 (several peaks, 4H), 1.71 (m, 2H), 3.03 (dd, J = 12.9, 6.1, 1H), 3.09 (dd, J = 12.9, 6.3, 1H), 4.01 (m, 1H), 4.15 (q, J = 7.1, 2H), 5.14 (d, J = 12.3, 1H), 7.25−7.31 (several peaks, 3H), 7.45 (dd, J = 12.3, 0.5, 1H), 7.53 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 13.8, 14.4, 22.4, 27.2, 31.8, 33.9, 59.7, 83.2, 97.4, 127.5, 129.2, 129.4, 133.3, 162.0, 168.0; IR (neat) 1138, 1639, 1702 cm^{-1}. Anal. Calcd for $C_{17}H_{24}O_3Se$: C, 57.46; H, 6.81. Found: C, 57.25; H, 6.89.

(*E*)-3-(2-Phenoxy-1-phenylselenenylmethyl-ethoxy)-acrylic Acid Ethyl Ester (2g): ^{1}H NMR ($CDCl_3$) δ 1.30 (t, J = 7.2, 3H), 3.20 (dd, J = 13.3, 6.1, 1H), 3.26 (dd, J = 13.3, 6.6, 1H), 4.13−4.24 (several peaks, 4H), 4.40 (m, 1H), 5.23 (d, J = 12.3, 1H), 6.87 (m, 2H), 7.01 (m, 1H), 7.27−7.32 (several peaks, 5H), 7.55 (d, J = 12.3, 1H), 7.58 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 14.3, 27.8, 59.7, 68.3, 80.9, 98.2, 114.5, 121.3, 127.6, 129.3, 129.4, 133.3, 157.9, 161.5, 167.5; IR (neat) 1133, 1642, 1707 cm^{-1}; HRMS calcd for $C_{20}H_{22}O_4Se$ 406.0683, found 406.0695.

(*E*)-3-(1-Phenyl-2-phenylselenenyl-ethoxy)-acrylic Acid Ethyl Ester (2h): ^{1}H NMR ($CDCl_3$) δ 1.24 (t, J = 7.1, 3H), 3.22 (dd, J = 13.0, 5.2, 1H), 3.40 (dd, J = 13.0, 8.2, 1H), 4.10 (dq, J = 10.8, 7.1, 1H), 4.14 (dq, J = 10.8, 7.1, 1H), 5.02 (ddm, J = 8.2, 5.2, 1H), 5.19 (d, J = 12.5, 1H), 7.26−7.40 (several peaks, 8H), 7.48 (dd, J = 12.5, 0.4, 1H), 7.51 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 14.3, 34.4, 59.7, 83.7, 98.9, 126.2, 127.4, 128.7, 128.8, 129.2, 129.5, 133.3, 138.9, 160.9, 167.5; IR (neat) 1131, 1642,

(31) For other examples of diastereocontrol by the N-substitutent, see ref 15b.

1708 cm^{-1}. Anal. Calcd for $C_{19}H_{20}O_3Se$: C, 60.80; H, 5.37. Found: C, 60.66; H, 5.21.

(*E*)-3-(1-Ethyl-2-phenylselenenyl-butoxy)-acrylic Acid Ethyl Ester (2j): ^{1}H NMR ($CDCl_3$) δ 0.91 (t, J = 7.4, 3H), 1.12 (t, J = 7.3, 3H), 1.28 (t, J = 7.1, 3H), 1.58 (m, 1H), 1.78−1.89 (several peaks, 3H), 3.17 (m, 1H), 3.93 (m, 1H), 4.17 (q, J = 7.1, 2H), 5.22 (d, J = 12.2, 1H), 7.27−7.31 (several peaks, 3H), 7.42 (d, J = 12.2, 1H), 7.56 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 9.8, 12.7, 14.4, 23.9, 25.6, 51.9, 59.6, 88.3, 97.1, 129.1, 127.7, 129.1, 134.9, 163.0, 168.1; IR (neat) 1141, 1637, 1698 cm^{-1}. Anal. Calcd for $C_{17}H_{24}O_3Se$: C, 57.46; H, 6.81. Found: C, 57.30; H, 6.74.

(*E*)-3-(*trans*-2-Phenylselenenyl-cyclohexyloxy)-acrylic Acid Ethyl Ester (2k): ^{1}H NMR ($CDCl_3$) δ 1.27 (t, J = 7.1, 3H), 1.29−1.36 (several peaks, 3H), 1.45−1.78 (several peaks, 5H), 2.08−2.19 (several peaks, 2H), 3.25 (ddd, J = 10.0, 8.4, 4.3, 1H), 3.95 (ddd, J = 12.6, 8.6, 4.0, 1H), 4.15 (q, J = 7.1, 2H), 5.15 (d, J = 12.4, 1H), 7.25−7.31 (several peaks, 3H), 7.48 (d, J = 12.5, 1H), 7.56 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 14.4, 23.1, 25.1, 31.1, 31.7, 46.2, 59.7, 80.1, 97.3, 126.7, 128.4, 129.4, 137.0, 161.6, 168.0; IR (neat) 1133, 1639, 1707 cm^{-1}; HRMS calcd for $C_{17}H_{22}O_3Se$ 354.0734, found 354.0748.

Preparation of (*E*)-3-(1-Benzyl-2-phenylselenenyl-ethoxy)-phenylsulfonyl Ethylene (2i). To a stirred solution of 2-hydroxy-3-phenylpropyl phenyl selenide (412 mg, 1.4 mmol) in THF (13 mL) was added NaH (60% in mineral oil, 85 mg, 2.1 mmol) in small portions. The resulting suspension was stirred for 15 min. (*E*)-1,2-Bis-(phenylsulfonyl)ethylene (567 mg, 1.8 mmol) was then added, and the mixture was stirred for an additional 6 h at rt. The solvent was removed in vacuo, and the residue was purified using flash chromatography (5−10% ethyl acetate/pentane) to afford the title compound (512 mg, 79%) as a yellowish oil: ^{1}H NMR ($CDCl_3$) δ 2.96 (dd, J = 14.1, 7.5, 1H), 3.04 (d, J = 6.1, 2H), 3.08 (dd, J = 14.1, 5.0, 1H), 4.21 (ddt, J = 7.4, 6.1, 5.0, 1H), 5.48 (d, J = 12.0, 1H), 7.11 (m, 2H), 7.19−7.31 (several peaks, 7H), 7.42−7.60 (several peaks, 5H), 7.74 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 30.7, 40.2, 84.8, 107.1, 126.4, 126.4, 127.1, 128.1, 128.4, 128.2, 128.8, 128.9, 131.9, 132.9, 135.6, 142.7, 159.9; IR (neat) 1084, 1142, 1217, 1305, 1625, 2928, 3065 cm^{-1}; HRMS calcd for $C_{23}H_{22}O_3SSe$ 458.0455, found 458.0494.

Typical Procedure for Carbonylation/Reductive Radical Cyclization. Preparation (5-Benzyl-3-oxo-tetrahydro-furan-2-yl)-acetic Acid Ethyl Ester (3a). To a solution of compound **2a** (0.143 g, 0.36 mmol) in dry benzene (50 mL) were added AIBN (0.018 g, 0.11 mmol) and tris(trimethylsilyl)silane (TTMSS) (0.192 mL, 0.62 mmol). The solution was then purged once with 20 atm of carbon monoxide and then pressurized to 80 atm and heated to 80 °C. After 12 h, the reaction mixture was cooled and the solvent removed in vacuo. The residue, on purification by flash chromatography (20−25% ethyl acetate/pentane), afforded the title compound **3a** (0.083 g, 86%) as a 9:1 mixture of cis and trans isomers. **[(2*R**,5*S**)-5-Benzyl-3-oxo-tetrahydro-furan-2-yl]-acetic Acid Ethyl Ester (3a, *cis*):** ^{1}H NMR ($CDCl_3$) δ 1.28 (t, J = 7.1, 3H), 2.44 (dd, J = 17.9, 9.5, 1H), 2.49 (dd, J = 17.9, 6.9, 1H), 2.74 (dd, J = 16.8, 5.3, 1H), 2.84 (dd, J = 16.8, 4.3, 1H), 2.97 (dd, J = 13.7, 6.6, 1H), 3.14 (dd, J = 13.7, 6.0, 1H), 4.05 (dd, J = 5.3, 4.4, 1H), 4.16 (m, 2H), 4.44 (m, 1H), 7.23−7.29 (several peaks, 3H), 7.30−7.35 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 14.1, 36.0, 41.5, 41.8, 60.9, 76.7, 77.6, 126.6, 128.4, 129.3, 136.9, 169.8, 214.4. **[(2*S**,5*S**)-5-Benzyl-3-oxo-tetrahydro-furan-2-yl]-acetic Acid Ethyl Ester (3a, *trans*):** ^{1}H NMR ($CDCl_3$) δ 1.25 (t, J = 7.2, 3H), 2.34 (ddd, J = 18.1, 6.6, 1.0, 1H), 2.71 (ddd, J = 18.1, 7.3, 0.7, 1H), 2.72 (dd, J = 16.7, 5.2, 1H), 2.77 (dd, J = 16.7, 4.7, 1H), 2.88 (dd, J = 13.8, 6.9, 1H), 3.05 (dd, J = 13.8, 6.0, 1H), 4.14 (q, J = 7.2, 2H), 4.21 (m, 1H), 4.73 (m, 1H), 7.20−7.26 (several peaks, 3H), 7.30 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 14.1, 36.8, 41.3, 42.0, 61.0, 75.7, 76.9, 126.7, 128.6, 129.4, 137.0, 170.2, 214.6. **3a** (mixture of diastereomers): IR (neat) 1095, 1156, 1189, 1375, 1735, 1758, 2917, 2981 cm^{-1}; HRMS calcd for $C_{15}H_{18}O_4$ 262.1205, found 262.1173.

Spectral data for compounds **3** prepared are shown below. For yields and isomeric ratios, see Table 1.

[(2*R,5*R**)-5-Benzyloxymethyl-3-oxo-tetrahydro-furan-2-yl]-acetic Acid Ethyl Ester (3c, *cis*):** ^{1}H NMR ($CDCl_3$) δ 1.23 (t, J = 7.1, 3H), 2.52 (dd, J = 17.9, 6.9, 1H), 2.58 (dd, J = 18.0, 9.3, 1H), 2.75 (dd, J = 16.9, 5.5, 1H), 2.83 (dd, J = 16.9, 4.2, 1H), 3.65 (dd, J = 10.5, 5.3, 1H), 3.69 (dd, J = 10.5, 3.9, 1H), 4.09 (dd, J = 4.2, 5.5, 1H), 4.12 (q, J = 7.1, 2H), 4.43 (m, 1H), 4.59 (s, 2H), 7.27−7.36 (several peaks, 5H); ^{13}C NMR ($CDCl_3$) δ 14.1, 36.1, 38.8, 60.9, 71.6, 73.5, 75.2, 77.4, 127.7, 127.7, 128.4, 137.7, 169.9, 213.9. **[(2S*,5R*)-5-Benzyloxymethyl-3-oxo-tetrahydro-furan-2-yl]-acetic Acid Ethyl Ester (3c, *trans*):** ^{1}H NMR ($CDCl_3$) δ 1.25 (t, J = 7.1, 3H), 2.51 (ddd, J = 17.9, 4.8, 0.8, 1H), 2.73−2.84 (several peaks, 3H), 3.54 (dd, J = 10.3, 4.0, 1H), 3.72 (dd, J =10.3, 3.2, 1H), 4.14 (q, J = 7.1, 2H), 4.29 (m, 1H), 4.53 (d, J = 12.1, 1H), 4.60 (d, J = 12.1, 1H), 4.63 (m, 1H), 7.26−7.36 (several peaks, 5H); ^{13}C NMR ($CDCl_3$) δ 15.2, 37.0, 38.1, 65.7, 73.0, 73.5, 75.2, 76.1, 127.5, 128.5, 129.1, 137.6, 170.1, 214.5. **3c** (mixture of diastereomers): IR (neat) 1028, 1096, 1378, 1735, 1758, 2982 cm^{-1}; HRMS calcd for $C_{16}H_{20}O_5$ 292.1311, found 292.1274.

In the preparation of tetrahydrofuran-3-one **3c** (Table 1, entries 3 and 4), compound **5** was isolated as a byproduct in 32 and 30% yields. A control experiment conducted under the same conditions as in the typical procedure for carbonylation/reductive radical cyclization, but in the absence of CO, afforded compound **5** as a colorless oil in 82% yield: **[(2*R**,3*R**,6*S**)-6-Methyl-3-phenyl-[1,4]dioxan-2-yl]-acetic Acid Ethyl Ester (5):** ^{1}H NMR ($CDCl_3$) δ 1.13 (d, J = 6.2, 3H), 1.18 (t, J = 7.1, 3H), 2.19 (dd, J = 15.3, 3.9, 1H), 2.35 (dd, J = 15.3, 8.6, 1H), 3.42 (dd, J = 10.2, 11.4, 1H), 3.88 (dd, J = 11.3, 2.7, 1H), 3.93 (m, 1H), 4.02 (q, J = 7.1, 2H), 4.04 (m, 1H), 4.19 (d, J = 9.1, 1H), 7.28−7.38 (several peaks, 5H); ^{13}C NMR ($CDCl_3$) δ 14.1, 16.7, 37.2, 60.5, 71.8, 72.4, 77.2, 81.9, 127.7, 128.5, 128.6, 137.6, 170.6; IR (neat) 1094, 1157, 1740, 2978 cm^{-1}; HRMS calcd for $C_{15}H_{20}O_4$ 264.1362, found 264.1346.

[(2*R,5*S**)-5-Butyl-3-oxo-tetrahydro-furan-2-yl]-acetic Acid Ethyl Ester (3f, *cis*):** ^{1}H NMR ($CDCl_3$) δ 0.89−0.95 (m, 3H), 1.26 (t, J = 7.1, 3H), 1.28−1.48 (several peaks, 4H), 1.61 (m, 1H), 1.79 (m, 1H), 2.33 (dd, J = 17.9, 10.7, 1H), 2.53 (dd, J = 17.9, 5.7, 1H), 2.72 (dd, J = 16.8, 5.4, 1H), 2.81 (dd, J = 16.8, 4.3, 1H), 4.01 (dd, J = 5.4, 4.3, 1H), 4.14 (m, 1H), 4.15 (q, J = 7.1, 2H); ^{13}C NMR ($CDCl_3$) δ 14.0, 14.1, 22.6, 27.4, 35.1, 36.0, 42.5, 60.9, 76.3, 77.6, 170.0, 215.5. **[(2S*,5S*)-5-Butyl-3-oxo-tetrahydro-furan-2-yl]-acetic Acid Ethyl Ester (3f, *trans*):** ^{1}H NMR ($CDCl_3$) δ 0.86−0.97 (m, 3H), 1.26 (t, J = 7.1, 3H), 1.26−1.47 (several peaks, 4H), 1.54 (m, 1H), 1.72 (m, 1H), 2.24 (ddd, J = 18.0, 6.6, 1.0, 1H), 2.75 (m, 2H), 2.76 (ddd, J = 18.0, 7.2, 0.5, 1H), 4.15 (q, J = 7.1, 2H), 4.20 (m, 1H), 4.43 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 14.0, 14.1, 22.5, 27.6, 35.6, 36.7, 42.2, 61.0, 75.3, 43.1, 170.3, 215.5. **3f** (mixture of diastereomers): IR (neat) 1027, 1099, 1159, 1192, 1377, 1741, 1763, 2863 cm^{-1}; HRMS calcd for $C_{12}H_{20}O_4$ 228.1362, found 228.1342.

[(2*R,5*R**)-3-Oxo-5-phenoxymethyl-tetrahydro-furan-2-yl]-acetic Acid Ethyl Ester (3g, *cis*):** ^{1}H NMR ($CDCl_3$) δ 1.24 (t, J = 7.2, 3H), 2.65 (dd, J = 18.1, 7.2, 1H), 2.69 (dd, J = 18.0, 9.2, 1H), 2.79 (dd, J = 17.0, 5.4, 1H), 2.88 (dd, J = 17.0, 4.3, 1H), 4.13 (q, J = 7.2, 2H), 4.14−4.18 (several peaks, 3H), 4.64 (m, 1H), 6.90−6.99 (several peaks, 3H), 7.26−7.31 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 14.0, 36.1, 38.6, 60.8, 69.5, 74.3, 77.3, 114.4, 121.1, 129.4, 158.3, 169.8, 213.3. **[(2S*,5R*)-3-Oxo-5-phenoxymethyl-tetrahydro-furan-2-yl]-acetic Acid Ethyl Ester (3g, *trans*):** ^{1}H NMR ($CDCl_3$) δ 1.26 (t, J = 7.2, 3H), 2.64 (ddd, J = 18.0, 4.7, 0.8, 1H), 2.80 (dd, J = 17.0, 4.5, 1H), 2.85 (dd, J = 17.0, 4.5, 1H), 2.94 (dd, J = 18.0, 8.6, 1H), 4.05 (dd, J = 10.1, 3.6, 1H), 4.14 (q, J = 7.2, 2H), 4.16 (m, 1H), 4.24 (dd, J = 10.1,3.1, 1H), 4.83 (m, 1H), 6.89−6.97 (several peaks, 3H), 7.26−7.31 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 14.1, 37.0, 38.0, 61.0, 74.5, 76.1, 114.5, 121.3, 129.5, 158.4, 170.2, 214.4. **3g** (mixture of diastereomers): IR (neat) 1033,

1156, 1195, 1234, 1735, 1763, 2929 cm^{-1}; HRMS calcd for $C_{15}H_{18}O_5$ 278.1154, found 278.1168.

[(2*R,5*R**)-3-Oxo-5-phenyl-tetrahydro-furan-2-yl]-acetic Acid Ethyl Ester (3h, *cis*):** ^{1}H NMR ($CDCl_3$) δ 1.26 (t, J = 7.2, 3H), 2.76 (dd, J = 18.0, 10.6, 1H), 2.84 (dd, J = 18.0, 6.3, 1H), 2.87 (dd, J = 17.0, 4.7, 1H), 2.94 (dd, J = 17.0, 4.3, 1H), 4.18 (dd, J = 4.7, 4.3, 1H) 4.18 (dq, J = 10.8, 7.2, 1H), 4.19 (dq, J = 10.8, 7.2, 1H), 5.16 (dd, J = 10.6, 6.3, 1H), 7.29−7.45 (several peaks, 5H); ^{13}C NMR ($CDCl_3$) δ 14.1, 35.8, 44.6, 61.0, 77.8, 77.9, 126.3, 128.4, 128.6, 139.9, 169.9, 214.2. **[(2*S**,5*R**)-3-Oxo-5-phenyl-tetrahydro-furan-2-yl]-acetic Acid Ethyl Ester (3h, *trans*):** ^{1}HNMR ($CDCl_3$) δ 1.30 (t, J = 7.2, 3H), 2.69 (ddd, J = 18.1, 7.0, 1.1, 1H), 2.90 (d, J = 4.7, 2H), 3.15 (ddd, J = 18.1, 7.7, 0.6, 1H), 4.20 (q, J = 7.2, 2H), 4.39 (tdd, J = 4.7, 1.1, 0.6, 1H), 5.56 (dd, J = 7.7, 7.0, 1H), 7.24−7.55 (several peaks, 5H); ^{13}C NMR ($CDCl_3$) δ 14.1, 36.8, 44.0, 61.1, 75.9, 77.6, 125.7, 128.0, 128.7, 141.2, 170.4, 214.5. **3h** (mixture of diastereomers): IR (neat) 1026, 1094, 1151, 1192, 1375, 1735, 1758, 2929 cm^{-1}; HRMS calcd for $C_{14}H_{16}O_4$ 248.1049, found 248.1026.

(2*S,5*S**)-2-Benzenesulfonylmethyl-5-benzyl-dihydrofuran-3-one (3i, *cis*):** ^{1}H NMR ($CDCl_3$) δ 2.24 (dd, J = 18.3, 10.3, 1H), 2.50 (dd, J = 18.3, 5.7, 1H), 2.90 (dd, J = 14.1, 6.0, 1H), 2.98 (dd, J = 14.1, 6.0, 1H), 3.33 (dd, J = 14.8, 8.2, 1H), 3.57 (dd, J = 14.8, 2.6, 1H), 4.23 (dd, J = 8.2, 2.6, 1H), 4.40 (m, 1H), 7.01−7.08 (m, 2H), 7.19−7.35 (m, 3H), 7.53−7.60 (m, 2H), 7.64−7.70 (m, 1H), 7.93−7.98 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 40.8, 40.8, 57.4, 76.3, 76.9, 126.7, 128.2, 128.4, 129.1, 129.4, 133.8, 136.3, 140.0, 211.7. **(2*R**,5*S**)-2-Benzenesulfonylmethyl-5-benzyl-dihydro-furan-3-one (3i, *trans*):** ^{1}H NMR ($CDCl_3$) δ 2.36 (ddd, J = 18.3, 6.6, 0.9, 1H), 2.59 (dd, J = 18.3, 7.2, 1H), 2.85 (dd, J = 14.0, 6.5, 1H), 2.97 (dd, J = 14.0, 6.1, 1H), 3.37 (dd, J = 14.7, 8.1, 1H), 3.49 (dd, J = 14.7, 3.0, 1H), 4.13 (m, 1H), 4.61 (m, 1H), 7.14−7.34 (several peaks, 5H), 7.47−7.53 (m, 2H), 7.63 (m, 1H), 7.86−7.93 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 40.5, 41.2, 57.3, 74.2, 76.6, 126.8, 128.1, 128.6, 129.2, 129.4, 134.2, 136.4, 139.8, 212.1. **3i** (mixture of diastereomers): IR (neat) 1084, 1144, 1308, 1448, 1762, 2918 cm^{-1}; HRMS calcd for $C_{18}H_{18}O_4S$ 330.0926, found 330.0930.

(4,5-Diethyl-3-oxo-tetrahydro-furan-2-yl)-acetic Acid Ethyl Ester (3j): Characteristic peaks in ^{1}H NMR ($CDCl_3$) δ 1.26 (t, J = 7.1, 3H), 2.19 (dt, J = 9.7, 6.0, 1H), 2.70 (dd, J = 16.5, 5.6, 1H), 2.81 (dd, J = 16.5, 4.4, 1H), 3.79 (ddd, J = 9.7, 7.1, 3.9, 1H), 3.96 (dd, J = 5.5, 4.3, 1H), 4.15 (q, J = 7.1, 2H); MS *m*/*e* 229 (M + 1), 213, 201, 149, 139; IR (neat) 1033, 1161, 1188, 1376, 1466, 1740, 1758, 2937 cm^{-1}; HRMS calcd for $C_{12}H_{20}O_4$ 228.1362, found 228.1345.

(3-Oxo-octahydro-benzofuran-2-yl)-acetic Acid Ethyl Ester (3k): Characteristic peaks in ^{1}H NMR ($CDCl_3$) δ 1.23 (t, J = 7.1, 3H), 2.73 (dd, J = 16.8, 5.8, 1H), 2.81 (dd, J = 16.8, 4.1, 1H), 3.45 (dt, J = 11.3, 3.9, 1H), 3.97 (dd, J = 5.8, 4.1, 1H), 4.13 (q, J = 7.1, 2H); MS *m*/*e* 227 (M + 1), 213, 185, 149, 139; IR (neat) 1132, 1187, 1264, 1376, 1448, 1740, 1762, 2863 cm^{-1}; HRMS calcd for $C_{12}H_{18}O_4$ 226.1205, found 226.1182.

Below are spectroscopic data for reduced, uncyclized products isolated in the preparation of compounds **3a** and **3e**−**h**, respectively.

(*E*)-3-(1-Methyl-2-phenyl-ethoxy)-acrylic Acid Ethyl Ester (4): ^{1}H NMR ($CDCl_3$) δ 1.26 (t, J = 7.1, 3H), 1.28 (d, J = 6.2, 3H), 2.80 (dd, J = 13.8, 6.3, 1H), 2.98 (dd, J = 13.8, 6.6, 1H), 4.14 (q, J = 7.1, 2H), 4.22−4.30 (m, 1H), 5.24 (d, J = 12.5, 1H), 7.16−7.32 (m, 5H), 7.47 (d, J = 12.5, 1H); ^{13}C NMR ($CDCl_3$) δ 14.4, 19.6, 42.5, 59.7, 80.1, 97.3, 126.7, 128.4, 129.4, 137.0, 161.6, 168.0; IR (neat) 1130, 1622, 1642, 1708, 2980, 3029 cm^{-1}; HRMS calcd for $C_{14}H_{18}O_3 + H^+$ 235.1334, found 235.1346.

(*E*)-3-Phenethyloxy-acrylic Acid Ethyl Ester: ^{1}H NMR ($CDCl_3$) δ 1.28 (t, J = 7.1, 3H), 3.04 (t, J = 6.9, 2H), 4.07 (t, J = 6.9, 2H), 4.18 (q, J = 7.1, 2H), 5.23 (d, J = 12.5, 1H), 7.21−7.39 (several peaks, 5H), 7.60 (d, J = 12.5, 1H); ^{13}C NMR ($CDCl_3$) δ 14.3, 35.3, 59.8, 71.3, 96.7, 126.7, 128.6, 128.8, 137.2, 162.1, 167.7.

(*E*)-3-(1-Methyl-pentyloxy)-acrylic Acid Ethyl Ester: ^{1}H NMR ($CDCl_3$) δ 0.90 (m, 3H), 1.26 (d, J = 6.2, 3H), 1.26 (t, J = 7.2, 3H), 1.25−1.40 (several peaks, 2H), 1.45−1.72 (several peaks, 4H), 4.03 (m, 1H), 4.15 (q, J = 7.2, 2H), 5.22 (d, J = 12.4, 1H), 7.54 (dd, J = 12.4, 0.5, 1H); ^{13}C NMR ($CDCl_3$) δ 13.9, 14.4, 20.0, 22.5, 27.4, 36.0, 59.6, 79.8, 97.0, 162.0, 168.3.

(*E*)-3-(1-Methyl-2-phenoxy-ethoxy)-acrylic Acid Ethyl Ester: ^{1}H NMR ($CDCl_3$) δ 1.27 (t, J = 7.2, 3H), 1.40 (d, J = 6.4, 3H), 3.99 (dd, J = 10.1, 4.2, 1H), 4.05 (dd, J = 10.1, 6.3, 1H), 4.17 (q, J = 7.2, 2H), 4.43 (ddd, J = 6.4, 6.3, 4.2, 1H), 5.30 (d, J = 12.4, 1H), 6.90 (m, 2H), 6.97 (m, 1H), 7.28 (m, 2H), 7.61 (d, J = 12.4, 1H); ^{13}C NMR ($CDCl_3$) δ 14.3, 16.9, 59.7, 70.8, 77.5, 98.0, 114.8, 121.4, 129.5, 158.4, 161.7, 167.8.

(*E*)-3-(1-Phenyl-ethoxy)-acrylic Acid Ethyl Ester: ^{1}H NMR ($CDCl_3$) δ 1.25 (t, J = 7.2, 3H), 1.61 (d, J = 6.5, 3H), 4.12 (dq, J = 10.9, 7.2, 1H), 4.13 (dq, J = 10.9, 7.2, 1H), 5.06 (dq, J = 6.5, 0.5, 1H), 5.25 (d, J = 12.5, 1H), 7.29−7.41 (several peaks, 5H), 7.54 (dd, J = 12.5, 0.5, 1H); ^{13}C NMR ($CDCl_3$) δ 14.3, 23.3, 59.6, 80.4, 98.5, 125.7, 128.1, 128.7, 141.3, 161.2, 167.7.

Preparation of Resin-Bound 2-Hydroxy-3-phenylpropyl Phenyl Selenide (7). To a suspension of polymer-supported diaryl diselenide **6** (300 mg, 0.675 mmol) in ethanol (5 mL) was added $NaBH_4$ (51 mg, 1.35 mmol). The suspension was gently stirred for 6 h after which benzyloxirane (450 mg, 3.38 mmol) was added. The mixture was stirred overnight. HCl (2 mL, 2 M) was added, and the reaction was stirred for another 2 h. The filtered resin was washed successively with water, ethanol, water, ethanol, ether, and ethanol to afford title resin **7**.

Preparation of Resin-Bound (*E*)-3-(1-Benzyl-2-phenylselenenyl-ethoxy)-acrylic Acid Ethyl Ester (9). To a preheated (60 °C) suspension of resin **7** from the previous experiment in NMM (10 mL) was added ethyl propiolate (342 μl, 3.38 mmol). The suspension was gently stirred for 6 h. The resin was then filtered and washed successively with water, CH_2Cl_2, ethanol, water, ethanol, ether, and ethanol to afford the title resin **9**.

Typical Procedure for Radical Carbonylation/Reductive Cyclization of Polymer-Bound Resin: Preparation of (5-Benzyl-3-oxo-tetrahydro-furan-2-yl)-acetic Acid Ethyl Ester (3a). To resin **9** from the previous experiment swelled in benzene (114 mL) were added AIBN (33 mg, 0.2 mmol) and TTMSS (354 μL, 1.14 mmol, 0.01 M). The suspension was pressurized with CO at 80 atm and heated to 80 °C for 12 h. After filtration and washing with diethyl ether, the combined filtrate was concentrated and purified by column chromatography to afford the title compound (97 mg, 55% over three steps) as a 9:1 mixture of cis and trans isomers.

Typical Procedure for Reductive Cleavage of Resin-Bound Selenides. To a preheated (80 °C) suspension of resin **9** from the above experiment in benzene (10 mL) were added AIBN (11 mg, 0.06 mmol) and TTMSS (1.04 mL, 3.38 mmol). The suspension was heated at reflux for 8 h, after which the reaction was filtered and the solid washed with diethyl ether. The combined filtrate was concentrated and purified by flash chromatography to afford vinyl ether **4** (144 mg, 91% over three steps).

Reductive cleavage of resin **7** similarly afforded 1-phenyl-2-propanol **8** (89 mg, 97% over two steps) with spectral data in accordance with literature.[32]

2-Amino-3-phenylpropyl Phenyl Selenide (10, R = Bn): ^{1}H NMR ($CDCl_3$) δ 1.46 (br s, 2H), 2.67 (dd, J = 7.9, 13.3, 1H), 2.86 (dd, J = 8.0, 12.4, 1H), 2.88 (dd, J = 5.4, 13.3, 1H), 3.14 (dd, J = 4.3, 12.4, 1H), 3.22 (m, 1H) 7.18 (m, 2H), 7.21−7.34 (several peaks, 6H), 7.47−7.52 (several peaks, 2H); ^{13}C NMR ($CDCl_3$) δ 36.6, 44.0, 52.5, 126.4, 126.9, 128.5, 129.1, 129.2, 130.1, 132.6, 138.8; IR (neat) 1437, 1478, 1578, 2920,

(32) Hayashi, T.; Matsumoto, Y.; Ito, Y. *Tetrahedron: Asymmetry* **1991**, *2*, 601.

3025, 3058, 3366 cm^{-1}; HRMS calcd for $C_{15}H_{17}NSe + H^+$ 292.0605, found 292.0610.

Typical Procedure for N-Vinylation of Aziridines. Synthesis of 3-(2-Benzyl-aziridin-1-yl)-acrylic Acid Methyl Ester (11a). Methyl propiolate (0.775 mL, 8.7 mmol) was slowly added to 2-benzyl-aziridine (1.10 g, 8.3 mmol), and the reaction mixture was stirred at room temperature for 2 h. After in vacuo removal of excess methyl propiolate, the pure title compound was obtained in quantitative yield as a colorless oil. For the synthesis of *N*-vinyl aziridines **11e** and **11f**, stoichiometric amounts of ethynyl *p*-tolyl sulfone and dimethyl acetylenedicarboxylate, respectively, were used instead of methyl propiolate. **(*E*)-11a:** ^{1}H NMR ($CDCl_3$) δ 2.02 (dt, J = 5.9, 0.4, 1H), 2.10 (dm, J = 3.4, 1H), 2.27 (m, 1H), 2.78 (dd, J = 14.3, 6.8, 1H), 2.78 (dd, J = 14.3, 5.5, 1H), 3.67 (s, 3H), 5.24 (d, J = 13.4, 1H), 7.22−7.37 (several peaks, 5H), 7.50 (d, J = 13.4, 1H); ^{13}C NMR ($CDCl_3$) δ 33.8, 38.5, 40.3, 51.1, 105.2, 126.6, 128.6, 128.6, 138.2, 157.2, 168.0. **(*Z*)-11a:** ^{1}H NMR ($CDCl_3$) δ 2.12 (dt, J = 5.7, 0.4, 1H), 2.10 (dm, J = 3.6, 1H), 2.42 (m, 1H), 2.75 (dd, J = 14.3, 6.5, 1H), 3.15 (dd, J = 14.3, 5.7, 1H), 3.72 (s, 3H), 5.15 (d, J = 8.9, 1H), 6.61 (d, J = 8.9, 1H), 7.22−7.34 (several peaks, 5H); ^{13}C NMR ($CDCl_3$) δ 37.2, 38.3, 42.5, 50.8, 102.3, 126.4, 128.5, 128.6, 138.4, 155.0, 165.8. **11a** (mixture of diastereomers): IR (neat) 1154, 1266, 1621, 1709, 2949 cm^{-1}. Anal. Calcd for $C_{13}H_{15}NO_2$: C, 71.87; H, 6.96. Found: C, 71.85; H, 6.97.

Spectral data for compounds **11** prepared are shown below. For yields and *E*/*Z* ratios, see Table 3.

3-(2-Phenoxymethyl-aziridin-1-yl)-acrylic Acid Methyl Ester (11b). (*E*)-11b: ^{1}H NMR ($CDCl_3$) δ 2.11 (dt, J = 6.1, 0.5, 1H), 2.20 (dd, J = 3.4, 0.5, 1H), 2.50 (m, 1H), 3.70 (s, 3H), 3.94 (dd, J = 10.5, 6.7, 1H), 4.13 (dd, J = 10.5, 4.2, 1H), 5.14 (d, J = 13.4, 1H), 6.89−7.00 (several peaks, 3H), 7.26−7.32 (several peaks, 2H), 7.54 (dm, J = 13.4, 1H); ^{13}C NMR ($CDCl_3$) δ 31.4, 37.6, 51.2, 68.8, 106.3, 114.6, 121.1, 129.5, 156.4, 158.3, 167.8. **(*Z*)-11b:** ^{1}H NMR ($CDCl_3$) δ 2.20 (d, J = 5.8, 1H), 2.40 (d, J = 3.5, 1H), 2.64 (m, 1H), 3.70 (s, 3H), 4.06 (dd, J = 10.5, 5.2, 1H), 4.32 (dd, J = 10.5, 5.3, 1H), 5.19 (d, J = 9.0, 1H), 6.67 (d, J = 9.0, 1H), 6.91−6.98 (several peaks, 3H), 7.24−7.31 (several peaks, 2H); ^{13}C NMR ($CDCl_3$) δ 34.7, 40.0, 50.8, 68.6, 103.0, 114.5, 120.9, 129.3, 154.3, 158.4, 165.7. **11b** (mixture of diastereomers): IR (neat) 1158, 1245, 1272, 1496, 1625, 1708, 2949, 2994 cm^{-1}. Anal. Calcd for $C_{13}H_{15}NO_3$: C, 66.94; H, 6.48. Found: C, 66.76; H, 6.47.

3-(2-Hexyl-aziridin-1-yl)-acrylic Acid Methyl Ester (11c). (*E*)-11c: ^{1}H NMR ($CDCl_3$) δ 0.88 (m, 3H), 1.24−1.54 (several peaks, 10H), 1.93−1.96 (several peaks, 2H), 2.01 (m, 1H), 3.69 (s, 3H), 5.31 (d, J = 13.3, 1H), 7.52 (d, J = 13.3, 1H); ^{13}C NMR ($CDCl_3$) δ 14.0, 22.5, 27.3, 29.0, 31.7, 32.2, 33.9, 39.8, 51.1, 105.0, 157.7, 168.1. **(*Z*)-11c:** ^{1}H NMR ($CDCl_3$) δ 0.88 (m, 3H), 1.24−1.54 (several peaks, 10H), 1.76 (m, 1H), 2.08 (m, 1H), 2.15 (m, 1H), 3.69 (s, 3H), 5.12 (d, J = 8.9, 1H), 6.64 (d, J = 13.3, 1H); ^{13}C NMR ($CDCl_3$) δ 14.0, 22.6, 26.8, 29.0, 31.8, 32.1, 32.7, 41.8, 50.8, 102.2, 155.3, 165.9. **11c** (mixture of diastereomers): IR (neat) 1152, 1266, 1623, 1714, 2929 cm^{-1}. Anal. Calcd for $C_{12}H_{21}NO_2$: C, 68.21; H, 10.02. Found: C, 68.24; H, 10.00.

3-(2-*tert*-Butyl-aziridin-1-yl)-acrylic Acid Methyl Ester (11d). (*E*)-11d: ^{1}H NMR ($CDCl_3$) δ 0.91 (s, 9H), 1.76 (dt, J = 6.2, 0.7, 1H), 1.81 (ddd, J = 6.2, 3.5, 0.5, 1H), 2.11 (dd, J = 3.6, 0.7, 1H), 3.68 (s, 3H), 5.29 (d, J = 13.4, 1H), 7.51 (dt, J = 13.4, 0.6, 1H); ^{13}C NMR ($CDCl_3$) δ 26.3, 30.4, 36.3, 49.3, 51.1, 104.5, 158.7, 168.2. **(*Z*)-11d:** ^{1}H NMR ($CDCl_3$) δ 0.92 (s, 9H), 1.86 (dd, J = 6.0, 3.7, 1H), 1.91 (dt, J = 6.0, 0.6, 1H), 2.53 (dt, J = 3.7, 0.5, 1H), 3.68 (s, 3H), 5.10 (d, J = 8.8, 1H), 6.64 (dm, J = 8.8, 1H); ^{13}C NMR ($CDCl_3$) δ 26.1, 30.4, 30.5, 49.6, 50.8, 101.7, 156.6, 165.9. **11d** (mixture of diastereomers): IR (neat) 1154, 1255, 1624, 1714, 2956 cm^{-1}. Anal. Calcd for $C_{10}H_{17}NO_2$: C, 65.54; H, 9.35. Found: C, 65.44; H, 9.42.

2-(2-*tert*-Butyl-aziridin-1-yl)-but-2-enedioic Acid Dimethyl Ester (11e).[33] **(*E*)-11e:** ^{1}H NMR ($CDCl_3$) δ 0.88 (s, 9H), 1.95 (d, J = 6.5, 1H), 2.07 (dd, J = 6.5, 3.9, 1H), 2.19 (d, J = 3.9, 1H), 3.66 (s, 3H), 3.87 (s, 3H), 5.27 (s, 1H); ^{13}C NMR ($CDCl_3$) δ 26.1, 30.4, 30.7, 49.2, 51.4, 52.6, 101.9, 160.3, 166.2, 166.4. **(*Z*)-11e:** ^{1}H NMR ($CDCl_3$) δ 0.97 (s, 9H), 1.73 (dd, J = 6.3, 1.0, 1H), 2.24 (dd, J = 6.4, 4.0, 1H), 2.60 (dd, J = 4.0, 1.0, 1H), 3.72 (s, 3H), 3.79 (s, 3H), 6.08 (s, 1H); ^{13}C NMR ($CDCl_3$) δ 26.7, 31.0, 35.4, 50.3, 51.3, 52.4, 106.8, 154.9, 164.4, 165.3. **11e** (mixture of diastereomers): IR (neat) 1171, 1261, 1437, 1608, 1714, 1744, 2953 cm^{-1}. Anal. Calcd for $C_{12}H_{19}NO_4$: C, 59.73; H, 7.94. Found: C, 59.51; H, 8.12.

2-Benzyl-1-[2-(toluene-4-sulfonyl)-vinyl]-aziridine (11f). (*Z*)-11f: ^{1}H NMR ($CDCl_3$) δ 2.33 (d, J = 3.7, 1H), 2.37 (d, J = 5.9, 1H), 2.43 (s, 3H), 2.58 (m, 1H), 2.70 (dd, J = 6.7, 14.3, 1H), 3.17 (dd, J = 5.4, 14.3, 1H), 5.57 (d, J = 8.9, 1H), 6.51 (d, J = 8.9, 1H), 7.20−7.33 (several peaks, 7H), 7.86−7.88 (several peaks, 2H); ^{13}C NMR ($CDCl_3$) δ 21.5, 37.7, 38.2, 42.6, 113.3, 126.5, 126.8, 128.4, 128.8, 129.5, 137.8, 140.0, 143.6, 151.0; IR (neat) 1142, 1300, 1596, 2922, 3062 cm^{-1}; HRMS calcd for $C_{18}H_{19}NO_2S + H^+$, 314.1215, found 314.1218.

3-(8-Aza-bicyclo[5.1.0]oct-8-yl)-acrylic Acid Methyl Ester (11g). (*E*)-11g: ^{1}H NMR ($CDCl_3$) δ 1.25−1.63 (several peaks, 6H), 1.73−1.85 (several peaks, 2H), 1.88−1.96 (several peaks, 2H), 2.13−2.19 (several peaks, 2H), 3.67 (s, 3H), 5.27 (d, J = 13.4, 1H), 7.53 (d, J = 13.4, 1H); ^{13}C NMR ($CDCl_3$) δ 25.6, 29.2, 31.5, 43.8, 51.0, 103.9, 158.5, 168.3. **(*Z*)-11g:** ^{1}H NMR ($CDCl_3$) δ 1.25−1.63 (several peaks, 6H), 1.73−1.85 (several peaks, 2H), 2.00−2.09 (several peaks, 2H), 2.24−2.30 (several peaks, 2H), 3.67 (s, 3H), 5.03 (d, J = 9.0, 1H), 6.60 (d, J = 9.0, 1H); ^{13}C NMR ($CDCl_3$) δ 25.7, 29.2, 31.7, 46.5, 50.7, 101.4, 155.9, 166.2. **11g** (mixture of diastereomers): IR (neat) 1147, 1620, 1714, 2925 cm^{-1}; HRMS calcd for $C_{11}H_{17}NO_2Se + H^+$, 196.1338, found 196.1366.

3-(2,2-Dimethyl-aziridin-1-yl)-acrylic Acid Methyl Ester (11h). (*E*)-11h: ^{1}H NMR ($CDCl_3$) δ 1.26 (s, 6H), 1.91 (s, 2H), 3.68 (s, 3H), 5.28 (d, J = 13.4, 1H), 7.50 (d, J = 13.4, 1H); ^{13}C NMR ($CDCl_3$) δ 21.9, 39.3, 40.5, 50.5, 104.1, 155.0, 167.6. **(*Z*)-11h:** ^{1}H NMR ($CDCl_3$) δ 1.30 (s, 6H), 2.06 (s, 2H), 3.68 (s, 3H), 5.16 (d, J = 8.9, 1H), 6.58 (d, J = 8.9, 1H); ^{13}C NMR ($CDCl_3$) δ 21.9, 41.6, 43.4, 50.1, 101.5, 152.7, 165.7. **11h** (mixture of diastereomers): IR (neat) 1158, 1312, 1618, 1711 cm^{-1}; HRMS calcd for $C_8H_{14}NO_2+H^+$, 156.1025, found 156.1032.

Typical Procedures (A and B) for Ring-Opening of Vinylated Aziridines with Benzeneselenolate. Synthesis of 3-(2-Phenyl-1-phenylselenenylmethyl-ethylamino)-acrylic Acid Methyl Ester (12a): Procedure A. $NaBH_4$ (0.149 g, 3.94 mmol) was added to a solution of diphenyl diselenide (1.12 g, 3.59 mmol) in ethanol (20 mL). To the colorless reaction mixture was added dropwise 3-(2-benzyl-aziridin-1-yl)-acrylic acid methyl ester **11a** (1.30 g, 5.98 mmol), and the solution was stirred for 6 h. α-Bromoacetic acid was added, and the reaction mixture was stirred for an additional hour. The white suspension was diluted with diethylether (50 mL) and then extracted with $NaHCO_3$ (saturated aqueous) and NaCl (saturated). After drying of the mixture over $MgSO_4$ and in vacuo removal of solvent, the title compound (2.19 g, 98%) was obtained as a 4/5 mixture of (*E*)- and (*Z*)-isomers. **Procedure B.** $NaBH_4$ (0.149 g, 3.94 mmol) was added to a solution of diphenyl diselenide (1.12 g, 3.59 mmol) in ethanol (20 mL). To the colorless reaction mixture was added dropwise 3-(2-benzyl-aziridin-1-yl)-acrylic acid methyl ester **11a** (1.30 g, 5.98 mmol), and the solution was stirred for 6 h. The solvent was evaporated and the residue diluted with diethyl ether (50 mL). The organic phase was extracted with H_2O and NaCl (saturated) and then dried over $MgSO_4$. After in vacuo removal of the solvent, the residue was purified by column chromatography on neutral alumina (3−10% EtOAc in pentane) to afford the title compound (1.87 g, 84%) as a 4/5 mixture of (*E*)- and (*Z*)-isomers. **(*Z*)-12a:** ^{1}H NMR ($CDCl_3$) δ 2.79−3.10 (several peaks, 4H), 3.43 (m, 1H), 3.65 (s, 3H), 4.40 (d, J = 8.1, 1H), 6.38 (dd, J = 12.9, 8.1, 1H), 7.07−7.18 (several peaks,

(33) Configuration was assigned in analogy with compounds prepared in ref 29.

2H), 7.21−7.31 (several peaks, 6H), 7.43−7.52 (several peaks, 2H), 7.92 (br m, 1H). Characteristic peaks in ^{13}C NMR ($CDCl_3$) δ 33.9, 42.2, 50.1, 60.8, 82.0, 126.6, 127.1, 128.5, 129.1, 129.3, 132.8, 137.2, 150.7, 170.8. **(*E*)-12a:** ^{1}H NMR ($CDCl_3$) δ 2.81−3.07 (several peaks, 4H), 3.66 (s, 3H), 3.67 (m, 1H), 4.66 (br m, 1H), 4.69 (d, J = 13.4, 1H), 7.05−7.20 (several peaks, 2H), 7.21−7.31 (several peaks, 6H), 7.36 (dd, J = 13.1, 9.1, 1H), 7.43−7.52 (several peaks, 2H). Characteristic peaks in ^{13}C NMR ($CDCl_3$) δ 34.8, 40.6, 50.6, 58.6, 86.6, 126.8, 127.5, 128.9, 129.2, 129.3, 133.2, 136.7, 169.7. **12a** (mixture of diastereomers): IR (neat) 1196, 1478, 1615, 1669, 2944, 3023, 3059, 3316 cm^{-1}; HRMS calcd for $C_{19}H_{21}NO_2Se + H^+$, 376.0817, found 376.0977.

Spectral data for compounds **12** prepared are shown below. For yields and *E*/*Z* ratios, see Table 3.

3-(2-Phenoxy-1-phenylselenenylmethyl-ethylamino)-acrylic Acid Methyl Ester (12b). (*Z*)-12b: ^{1}H NMR ($CDCl_3$) δ 3.16 (dd, J = 13.0, 6.7, 1H), 3.27 (dd, J = 13.0, 6.5, 1H), 3.60 (m, 1H), 3.65 (s, 3H), 4.02 (dd, J = 9.3, 4.9, 1H), 4.09 (dd, J = 9.3, 5.0, 1H), 4.54 (d, J = 8.1, 1H), 6.65 (dd, J = 12.9, 8.2, 1H), 6.68−6.88 (several peaks, 2H), 6.98 (m, 1H), 7.19−7.31 (several peaks, 5H), 7.48−7.58 (several peaks, 2H), 8.10 (dd, J = 11.8, 9.7, 1H); ^{13}C NMR ($CDCl_3$) δ 28.7, 50.3, 58.4, 67.3, 87.1, 114.6, 121.3, 127.4, 129.1, 129.2, 129.4, 133.0, 147.8, 158.1, 170.7. **(*E*)-12b:** ^{1}H NMR ($CDCl_3$) δ 3.18 (dd, J = 12.9, 7.6, 1H), 3.23 (dd, J = 12.9, 5.4, 1H), 3.66 (s, 3H), 3.67 (m, 1H), 3.99 (dd, J = 9.3, 4.5, 1H), 4.18 (dd, J = 9.3, 3.6, 1H), 4.65 (d, J = 13.4, 1H), 6.68−6.88 (several peaks, 2H), 6.96 (m, 1H), 7.19−7.31 (several peaks, 5H), 7.43 (dd, J = 13.4, 9.0, 1H), 7.48−7.58 (several peaks, 2H), 8.10 (dd, J = 11.8, 9.7, 1H); ^{13}C NMR ($CDCl_3$) δ 30.7, 50.6, 60.4, 69.5, 83.2, 114.4, 121.5, 127.7, 128.7, 129.4, 129.5, 133.3, 150.7, 158.0, 169.6. **12b** (mixture of diastereomers): IR (neat) 1201, 1242, 1478, 1495, 1618, 1671, 2946, 3401 cm^{-1}; HRMS calcd for $C_{19}H_{21}NO_3Se + H^+$, 388.0792, found 388.0789.

3-(1-Phenylselenenylmethyl-heptylamino)-acrylic Acid Methyl Ester (12c). (*Z*)-12c: ^{1}H NMR ($CDCl_3$) δ 0.86 (m, 3H), 1.18−1.72 (several peaks, 10H), 2.99 (dd, J = 7.3, 12.6, 1H), 3.04 (dd, J = 5.6, 12.7, 1H), 3.18 (m, 1H), 3.64 (s, 3H), 4.46 (d, J = 8.1, 1H), 6.58 (dd, J = 8.1, 13.0, 1H), 7.23−7.28 (several peaks, 3H), 7.48−7.53 (several peaks, 2H), 7.81 (br m, 1H); ^{13}C NMR ($CDCl_3$) δ 14.0, 22.5, 25.9, 28.9, 31.6, 35.2, 35.7, 50.1, 59.5, 81.6, 127.1, 129.1, 129.8, 132.9, 151.2, 171.1. **(*E*)-12c:** ^{1}H NMR ($CDCl_3$) δ 0.87 (m, 3H), 1.18−1.72 (several peaks, 10H), 3.03 (dd, J = 5.7, 11.5, 1H), 3.06 (dd, J = 5.2, 11.5, 1H), 3.36 (m, 1H), 3.65 (s, 3H), 4.47 (br m, 1H), 4.63 (d, J = 13.3, 1H), 7.23−7.28 (several peaks, 3H), 7.38 (dd, J = 9.4, 13.3, 1H), 7.48−7.53 (several peaks, 2H); ^{13}C NMR ($CDCl_3$) δ 14.0, 22.5, 25.8, 28.9, 31.7, 34.9, 35.6, 50.4, 57.0, 86.0, 127.4, 129.0, 129.2, 133.2, 148.7, 169.9. **12c** (mixture of diastereomers): IR (neat) 1194, 1478, 1615, 1670, 2856, 2929, 3324 cm^{-1}; HRMS calcd for $C_{18}H_{29}NO_2Se + H^+$, 372.1443, found 372.1450.

3-(2,2-Dimethyl-1-phenylselenenylmethyl-propylamino)-acrylic Acid Methyl Ester (12d). (*Z*)-12d: ^{1}H NMR ($CDCl_3$) δ 0.93 (s, 9H), 2.82−2.86 (several peaks, 2H), 3.27 (m, 1H), 3.64 (s, 3H), 4.45 (d, J = 7.9, 1H), 6.57 (dd, J = 7.9, 12.8, 1H), 7.23−7.27 (several peaks, 3H), 7.46−7.50 (several peaks, 2H), 7.92 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 26.5, 31.0, 35.9, 50.1, 69.8, 80.6, 127.1, 129.1, 130.0, 132.9, 152.9, 171.3; IR (neat) 1188, 1477, 1612, 1666, 2960, 3307 cm^{-1}; HRMS calcd for $C_{16}H_{23}NO_2Se + H^+$, 342.0973, found 342.0985.

2-(2,2-Dimethyl-1-phenylselenenylmethyl-propylamino)-but-2-enedioic Acid Dimethyl Ester (12e). Minor isomer: ^{1}H NMR ($CDCl_3$) δ 0.98 (s, 9H), 2.81 (dd, J = 10.9, 12.6, 1H), 3.32 (dd, J = 2.5, 12.6, 1H), 3.66 (s, 3H), 3.72 (s, 3H), 3.90 (m, 1H), 5.07 (s, 1H), 7.18−7.27 (several peaks, 3H), 7.40−7.45 (several peaks, 2H), 8.27 (d, J = 11.2, 1H); ^{13}C NMR ($CDCl_3$) δ 26.5, 32.0, 35.9, 50.7, 52.5, 61.7, 86.6, 126.7, 129.0, 130.7, 132.1, 147.1, 152.5, 170.9. **Major isomer:** ^{1}H NMR ($CDCl_3$) δ 0.99 (s, 9H), 2.80 (dd, J = 10.9, 12.6, 1H), 3.32 (dd, J = 2.5, 12.6, 1H), 3.66 (s, 3H), 3.72 (s, 3H), 3.90 (m, 1H), 5.07 (s, 1H), 7.18−7.27 (several peaks, 3H), 7.40−7.45 (several peaks, 2H), 8.27 (d, J = 11.2, 1H); ^{13}C NMR ($CDCl_3$) δ 26.5, 32.0, 35.9, 50.7, 52.5, 62.9, 86.8, 126.7, 129.0, 130.7, 132.1, 147.1, 152.5, 170.9; IR (neat) 1193, 1230, 1270, 1605, 1661, 1735, 1961, 3272 cm^{-1}; HRMS calcd for $C_{18}H_{25}NO_4Se + H^+$, 400.1028, found 400.1016.

(1-Benzyl-2-phenylselenenyl-ethyl)-[2-(toluene-4-sulfonyl)-vinyl]-amine (12f). (*Z*)-12f: ^{1}H NMR ($CDCl_3$) δ 2.40 (s, 3H), 2.69−3.12 (several peaks, 4H), 3.37 (m, 1H), 4.50 (d, J = 8.6, 1H), 6.22 (dd, J = 8.6, 13.4, 1H), 7.05 (m, 1H), 7.12 (m, 2H), 7.15−7.31 (several peaks, 8H), 7.48 (m, 2H), 7.68 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 31.4, 33.9, 42.0, 61.0, 89.8, 125.8, 127.4, 128.1, 128.3, 128.8, 129.0, 129.4, 132.1, 133.0, 136.9, 141.5, 142.8, 145.7. **(*E*)-12f:** ^{1}H NMR ($CDCl_3$) δ 2.40 (s, 3H), 2.82 (dd, J = 6.8, 14.0, 1H), 2.93 (dd, J = 6.3, 14.0, 1H), 2.94 (m, 2H), 3.49 (m, 1H), 4.86 (d, J = 12.7, 1H), 4.92 (t, J = 9.0, 1H), 7.06 (m, 2H), 7.20 (m, 1H), 7.15−7.31 (several peaks, 8H), 7.42 (m, 2H), 7.63 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 31.4, 31.8, 38.8, 61.0, 94.8, 126.2, 126.8, 127.6, 128.6, 129.1, 129.3, 129.3, 129.9, 133.3, 136.6, 141.6, 142.2, 146.2. **12f** (mixture of diastereomers): IR (neat) 1081, 1133, 1281, 1616, 2923, 3060, 3335 cm^{-1}; HRMS calcd for $C_{24}H_{25}NO_2SSe + Na^+$ 494.0670, found 494.0630.

3-(*trans*-2-Phenylselenenyl-cycloheptylamino)-acrylic Acid Methyl Ester (12g). (*Z*)-12g: ^{1}H NMR ($CDCl_3$) δ 1.37−2.17 (several peaks, 10H), 3.27−3.40 (several peaks, 2H), 3.64 (s, 3H), 4.45 (d, J = 7.9, 1H), 6.54 (dd, J = 7.9, 13.0, 1H), 7.23−7.32 (several peaks, 3H), 7.51−7.56 (several peaks, 2H), 8.15 (br m, 1H); ^{13}C NMR ($CDCl_3$) δ 23.0, 26.4, 28.3, 32.1, 33.6, 50.2, 52.5, 64.0, 81.7, 127.7, 129.1, 129.4, 134.9, 150.8, 171.1. **(*E*)-12g:** ^{1}H NMR ($CDCl_3$) δ 1.37−2.17 (several peaks, 10H), 3.19 (m, 1H), 3.33 (m, 1H), 3.66 (s, 3H), 4.59 (d, J = 13.4, 1H), 4.66 (br m, 1H), 7.23−7.32 (several peaks, 3H), 7.39 (dd, J = 9.0, 13.4, 1H), 7.51−7.56 (several peaks, 2H); ^{13}C NMR ($CDCl_3$) δ 23.5, 26.1, 28.2, 32.8, 33.6, 50.5, 52.5, 64.0, 86.6, 128.0, 129.2, 129.4, 135.3, 150.8, 169.8. **12g** (mixture of diastereomers): IR (neat) 1207, 1477, 1615, 1667, 2930, 3320 cm^{-1}; HRMS calcd for $C_{17}H_{23}NO_2Se + H$, 354.0972, found 354.0973.

3-(2,2-Dimethyl-1-phenylselenenylmethyl-propylamino)-acrylic Acid Methyl Ester (12h). (*Z*)-12h: ^{1}H NMR ($CDCl_3$) δ 1.33 (s, 6H), 3.07 (s, 2H), 3.62 (s, 3H), 4.47 (d, J = 8.2, 1H), 6.74 (dd, J = 8.2, 13.5, 1H), 7.16−7.25 (several peaks, 3H), 7.47−7.53 (several peaks, 2H), 8.17 (d, J = 13.5, 1H); ^{13}C NMR ($CDCl_3$) δ 27.6, 43.1, 50.0, 54.4, 82.1, 126.9, 128.9, 129.2, 132.9, 147.1, 170.7. **(*E*)-12h:** ^{1}H NMR ($CDCl_3$) δ 1.32 (s, 6H), 3.07 (s, 2H), 3.62 (s, 3H), 4.72 (d, J = 1.92, 1H), 4.94 (d, J = 13.6, 1H), 7.43 (dd, J = 12.9, 13.6, 1H), 7.16−7.25 (several peaks, 3H), 7.47−7.53 (several peaks, 2H); ^{13}C NMR ($CDCl_3$) δ 27.7, 42.8, 50.3, 54.7, 87.7, 127.2, 129.1, 128.7, 132.9, 146.6, 169.5. **12h** (mixture of diastereomers): IR (neat) 1081, 1133, 1281, 1616, 2923, 3060, 3335 cm^{-1}; HRMS calcd for $C_{14}H_{20}NO_2Se + H^+$, 314.0659, found 314.0662.

Typical Procedure for Carbonylation/Reductive Radical Cyclization: Preparation of (5-Benzyl-3-oxo-pyrrolidin-2-yl)-acetic Acid Methyl Ester (13a). To a solution of compound **12a** (109 mg, 0.29 mmol) in dry benzene (49 mL) were added AIBN (14 mg, 0.09 mmol) and tris(trimethylsilyl)-silane (TTMSS) (0.153 mL, 0.5 mmol). The solution was then purged once with 20 atm of carbon monoxide and then pressurized to 80 atm and heated to 80 °C. After 12 h, the reaction mixture was cooled and the solvent removed in vacuo. The residue, on purification by flash chromatography (20−40% ethyl acetate/pentane), afforded the title compound (57 mg, 79%) as a 4:1 mixture of cis and trans isomers. Due to the poor stability of the material, it was characterized in the N-tosylated form. This was also true for compounds **13b** and **13c**. Thus, to a stirred solution of **13a** (57 mg, 0.23 mmol) in CH_2Cl_2 (10 mL) were added TsCl (89 mg, 0.46 mmol), DMAP (2 mg, 0.02 mmol), and Et_3N (0.032 mL, 0.23 mmol). After 12 h, the solvent was removed in vacuo and the residue purified by flash chromatography to afford (74 mg, 80%) of N-tosylated material. **[(2*R**,5*S**)-5-Benzyl-3-oxo-1-(toluene-4-sulfonyl)-**

pyrrolidin-2-yl]-acetic Acid Methyl Ester (13a, *cis*): ^{1}H NMR ($CDCl_3$) δ 2.23 (ddd, J = 18.8, 9.9, 1.4, 1H), 2.43 (s, 3H), 2.49 (ddm, J = 18.8, 3.8, 1H), 2.91 (dd, J = 13.3, 10.2, 1H), 2.92 (dd, J = 16.9, 4.2, 1H), 2.97 (dd, J = 16.9, 5.0, 1H), 3.38 (ddm, J = 13.3, 3.7, 1H), 3.72 (s, 3H), 3.85 (m, 1H), 4.21 (m, 1H), 7.17−7.42 (several peaks, 8H), 7.77 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 21.5, 38.0, 40.5, 43.5, 52.0, 58.3, 61.0, 126.8, 127.7, 128.7, 129.5, 130.1, 133.2, 137.1, 144.5, 170.8, 209.1. **[(2*S**,5*S**)-5-Benzyl-3-oxo-1-(toluene-4-sulfonyl)-pyrrolidin-2-yl]-acetic Acid Methyl Ester (13a, *trans*):** ^{1}H NMR ($CDCl_3$) δ 2.43 (s, 3H), 2.43 (dd, J = 17.7, 1.6, 1H), 2.66 (dd, J = 13.1, 10.0, 1H), 2.89 (dd, J = 18.0, 3.3, 1H), 2.95 (dd, J = 17.7, 9.5, 1H), 3.06 (dd, J = 18.0, 4.5, 1H), 3.36 (s, 3H), 3.42 (dd, J = 13.1, 3.2, 1H), 3.71 (m, 1H), 4.71 (m, 1H), 7.18−7.45 (several peaks, 8H), 7.85 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 21.5, 35.2, 41.5, 51.6, 59.1, 59.4, 60.4, 127.0, 127.1, 128.8, 129.7, 130.4, 131.5, 136.4, 143.7, 169.9, 210.6. **13a** (mixture of diastereomers): IR (neat) 1161, 1348, 1736, 1762, 2923, 3024 cm^{-1}; HRMS calcd for $C_{21}H_{23}NO_5S + Na^+$, 424.1195, found 424.1183.

Spectral data for compounds **13** (in the case of compounds **13b** and **13c**, the N-tosylated derivatives) prepared are shown below. For yields and isomeric ratios, see Table 3.

[(2*R,5*R**)-5-Phenoxymethyl-3-oxo-1-(toluene-4-sulfonyl)-pyrrolidin-2-yl]-acetic Acid Methyl Ester (13b, *cis*):** ^{1}H NMR ($CDCl_3$) δ 2.43 (ddd, J = 18.3, 10.0, 1.4, 1H), 2.45 (s, 3H), 2.61 (ddd, 18.3, 2.7, 0.9, 1H), 2.98 (dd, J = 16.7, 4.6, 1H), 3.11 (dd, J = 16.7, 5.4, 1H), 3.74 (s, 3H), 4.00 (m, 1H), 4.20 (dd, J = 9.3, 3.5, 1H), 4.24 (dd, J = 9.3, 5.6, 1H), 4.41 (m, 1H), 6.87 (m, 2H), 6.98 (m, 1H), 7.29 (m, 2H), 7.37 (m, 2H), 7.78 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 21.6, 38.2, 39.4, 52.1, 55.3, 60.7, 71.1, 114.4, 121.4, 127.6, 129.6, 130.2, 133.3, 144.7, 157.9, 171.2, 208.4. **[(2*S**,5*R**)-5-Phenoxymethyl-3-oxo-1-(toluene-4-sulfonyl)-pyrrolidin-2-yl]-acetic Acid Methyl Ester (13b, *trans*):** ^{1}H NMR ($CDCl_3$) δ 2.05 (s, 3H), 2.56 (dm, J = 17.5, 1H), 3.02 (dd, 17.6, 3.2, 1H), 3.23 (dd, J = 17.6, 4.7, 1H), 3.26 (dd, J = 17.5, 9.7, 1H), 3.56 (s, 3H), 3.89 (dd, J = 9.9, 1.5, 1H), 3.99 (m, 1H), 4.51 (dd, J = 9.9, 2.7, 1H), 4.76 (m, 1H), 6.90 (m, 2H), 7.06 (m, 1H), 7.16 (m, 2H), 7.43 (m, 2H), 7.85 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 21.6, 36.3, 40.4, 51.8, 56.7, 60.4, 69.7, 114.1, 121.3, 126.7, 129.2, 130.4, 133.0, 148.0, 158.0, 170.3, 209.8. **13b** (mixture of diastereomers): IR (neat) 1163, 1244, 1350, 1599, 1737, 1765, 2952 cm^{-1}; HRMS calcd for $C_{21}H_{23}NO_6S + Na^+$, 440.1144, found 440.1111.

[(2*R,5*S**)-5-Hexyl-3-oxo-1-(toluene-4-sulfonyl)-pyrrolidin-2-yl]-acetic Acid Methyl Ester (13c, *cis*):** ^{1}H NMR ($CDCl_3$) δ 0.88 (m, 3H), 1.13−1.36 (several peaks, 8H), 1.61 (m, 1H), 1.88 (m, 1H), 2.33−2.36 (several peaks, 2H), 2.44 (s, 3H), 2.91 (dd, J = 16.5, 4.5, 1H), 3.01 (dd, J = 16.5, 5.6, 1H), 3.71 (s, 3H), 3.90 (dd, J = 5.6, 4.5, 1H), 3.95 (m, 1H), 7.35 (m, 2H), 7.74 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 14.0, 21.6, 22.6, 25.8, 28.9, 31.7, 37.4, 38.4, 41.5, 52.1, 57.3, 60.7, 127.7, 130.0, 133.6, 144.4, 170.7, 209.9. **[(2*S**,5*S**)-5-Hexyl-3-oxo-1-(toluene-4-sulfonyl)-pyrrolidin-2-yl]-acetic Acid Methyl Ester (13c, *trans*):** ^{1}H NMR ($CDCl_3$) δ 0.85 (m, 3H), 1.13−1.36 (several peaks, 8H), 1.52 (m, 1H), 1.90 (m, 1H), 2.36 (m, 1H), 2.42 (s, 3H), 3.07 (m, 1H), 2.95 (dd, J = 17.8, 3.3, 1H), 3.10 (dd, J = 17.8, 4.5, 1H), 3.66 (s, 3H), 3.81 (m, 1H), 4.45 (m, 1H), 7.29 (m, 2H), 7.72 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 14.0, 21.5, 22.5, 25.2, 29.0, 31.6, 34.2, 35.8, 42.1, 51.7, 58.3, 59.3, 127.7, 129.6, 138.3, 143.5, 170.1, 211.1. **13c** (mixture of diastereomers): IR (neat) 1160, 1350, 1739, 1762, 2857, 2926 cm^{-1}; HRMS calcd for $C_{20}H_{29}NO_5Se + Na^+$ 418.1664, found 418.1658.

[(2*R,5*R**)-5-*tert*-Butyl-3-oxo-pyrrolidin-2-yl]-acetic Acid Methyl Ester (13d, *cis*):** ^{1}H NMR ($CDCl_3$) δ 0.90 (s, 9H), 2.09 (ddd, J = 18.3, 10.8, 0.4, 1H), 2.27 (ddd, 18.3, 6.4, 0.7, 1H), 2.42 (br s, 1H), 2.52 (dd, J = 17.1, 8.2, 1H), 2.81 (dd, J = 17.1, 3.3, 1H), 3.10 (dd, J = 10.8, 6.4, 1H), 3.54 (ddm, J = 8.1, 3.3, 1H), 3.68 (s, 3H); ^{13}C NMR ($CDCl_3$) δ 25.8, 32.7, 36.2, 38.4, 51.8, 61.6, 63.6, 172.4, 215.6. **[(2*S**,5*R**)-5-*tert*-Butyl-3-oxo-pyrrolidin-2-yl]-acetic Acid Methyl Ester (13d, *trans*):** ^{1}H NMR ($CDCl_3$) δ 0.90 (s, 9H), 2.24 (ddd, J = 18.4, 8.0, 1.2, 1H), 2.38 (ddd, J = 18.4, 7.2, 0.6, 1H), 2.43 (br s, 1H), 2.52 (dd, J = 16.2, 8.5, 1H), 2.65 (dd, J = 16.2, 3.8, 1H), 3.26 (dd, J = 8.0, 7.3, 1H), 3.69 (s, 3H), 3.74 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 25.9, 34.0, 36.7, 38.7, 51.9, 59.9, 62.9, 171.9, 216.9. **13d** (mixture of diastereomers): IR (neat) 1175, 1245, 1747, 1752, 2957, 3364 cm^{-1}; HRMS calcd for $C_{11}H_{20}NO_3$, 214.1443, found 214.1393.

(2*R,5*R**)-5-*tert*-Butyl-2-methoxycarbonylmethyl-3-oxo-pyrrolidine-2-carboxylic Acid Methyl Ester (13e, *trans*):** ^{1}H NMR ($CDCl_3$) δ 0.90 (s, 9H), 2.22 (dd, J = 18.7, 10.3, 1H), 2.36 (dd, 18.7, 7.2, 1H), 2.69 (d, J = 16.6, 1H), 2.88 (br s, 1H), 3.14 (d, J = 16.6, 1H), 3.43 (dd, J = 10.3, 7.1, 1H), 3.66 (s, 3H), 3.74 (s, 3H); ^{13}C NMR ($CDCl_3$) δ 25.6, 33.0, 37.5, 41.8, 51.9, 53.2, 62.1, 71.9, 170.4, 170.9, 209.1; IR (neat) 1205, 1736, 1766, 2956, 3368 cm^{-1}; HRMS calcd for $C_{13}H_{21}NO_5 + H^+$, 272.1498, found 272.1475.

(3-Oxo-decahydro-cyclohepta[*b*]pyrrol-2-yl)-acetic Acid Methyl Ester 13g: Characteristic peaks in ^{1}H NMR ($CDCl_3$) δ 2.40 (m, 1H), 2.49 (dd, J = 8.3, 17.2, 1H), 2.58 (dd, J = 8.3, 17.4, 1H), 2.85 (dd, J = 2.9, 17.2, 1H), 2.86 (dd, J = 3.5, 17.4, 1H), 3.08 (dt, J = 4.1, 10.2, 1H), 3.46 (dd, J = 3.2, 8.3, 1H), 3.57 (dd, J = 3.4, 8.3, 1H), 3.69 (s, 3H); IR (neat) 1162, 1739, 2927, 3344 cm^{-1}; HRMS calcd for $C_{12}H_{19}NO_3 + H^+$, 226.1443, found 226.1446.

(5,5-Dimethyl-3-oxo-pyrrolidin-2-yl)-acetic Acid Methyl Ester 13h: ^{1}H NMR ($CDCl_3$) δ 1.24 (s, 3H), 1.33 (s, 3H), 2.25 (s, 2H), 2.36 (br s, 1H), 2.55 (dd, J = 17.0, 7.9, 1H), 2.79 (dd, J = 17.0, 3,5, 1H), 3.66 (s, 3H), 3.68 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 27.7, 30.1, 36.2, 50.6, 51.8, 54.8, 59.4, 172.1, 217.0; IR (neat) 1174, 1203, 1739, 2960, 3339 cm^{-1}; HRMS calcd for $C_9H_{15}NO_3 + H^+$, 186.1130, found 186.1159.

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Supporting Information Available: General experimental methods. This material is available free of charge via the Internet at http://pubs.acs.org.

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