Solid-Phase Tandem Radical Addition-Cyclization Reaction: Triethylborane-Induced Reaction of Oxime Ethers Anchored to Polymer Support

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Tandem radical addition-cyclization of oxime ethers anchored to polymer support was studied. The reaction of oxime ethers with stannyl radical proceeded effectively by the use of triethylborane as a radical initiator. The alkyl radical addition-cyclization reactions of oxime ether connected with α,β-unsaturated carbonyl group proceeded under iodine atom-transfer reaction conditions to give the functionalized azacycles *via* two carbon–carbon bonds-forming process.

Key words radical reaction; solid-phase; oxime ether; cyclization; triethylborane

Solid-phase radical reactions have been developed for an important carbon–carbon bond-forming method on solid support under mild reaction conditions.[2—11] We have recently demonstrated that triethylborane has the potential to induce the intermolecular radical reactions on solid support.[12,13] Moreover, the employment of triethylborane and its related radical initiator such as diethylzinc at low reaction temperature would facilitate the control of stereochemistry in solid-phase reactions.[14,15] As a part of our program directed toward the solid-phase radical reactions, the development of solid-phase multi bonds-forming reactions has been the new focus of our efforts. We report here in detail the triethylborane-induced tandem radical addition-cyclization reaction of oxime ethers anchored to polymer support.[16,17]

Free radical-mediated cyclization has been developed as a powerful method for preparing various types of cyclic compounds *via* carbon–carbon bond-forming processes.[18—29] Our laboratory is interested in developing the effective and convenient methods for the synthesis of highly functionalized cyclic compounds. For this purpose, we have studied the radical reaction using oxime ether group as a radical acceptor.[30—36] Particularly, strategies involving the radical addition-cyclization reactions offer the advantage of the formation of multiple carbon–carbon and carbon–heteroatom bonds in a single operation.

We recently reported the carbon radical addition–cyclization of oxime ethers connected with the α,β-unsaturated carbonyl group and the heteroatom radical addition-cyclization reaction of oxime ethers having an olefin moiety (Fig. 1).[37—40] Based on these results, we have studied the tandem radical addition-cyclization of oxime ethers (TYPE 1 and TYPE 2) anchored to polymer support.[16,17] In this paper, we report the radical addition-cyclization reaction of oxime ether (TYPE 1). To enhance the reactivity of resin-bound substrates, we introduced a temporary spacer generated from glutaric anhydride.[41]

Results and Discussion

Preparation of oxime ether **4** anchored to a polymer support is shown in Chart 1. The reaction of α-chloroacetaldoxime ether **1**, which was prepared from chloroacetaldehyde and *O*-benzylhydroxyamine hydrochloride,[42] with allylamine gave the secondary amine **2** in 76% yield. The amine **2** was treated with glutaric anhydride to give the oxime ether **3** as an *E*/*Z* mixture concerning oxime ether moiety in a 3 : 2 ratio. In our recent studies on the radical reaction of oxime ethers, we have observed no remarkable effect of the geometry of the starting oxime ether group on either the chemical yield or stereoselectivity by employing geometrically pure *E* and *Z*-isomers.[32] Thus, oxime ether **3** was subjected to the following reactions without the separation of *E*/*Z*-isomers. Wang resin purchased from Novabiochem was used. The oxime ether **3** having the spacer moiety was attached to Wang resin by treatment with DCC in the presence of DMAP in CH_2Cl_2 at 20 °C for 12 h to give the Wang resin-bound oxime ether **4** in *ca.* 95% loading level. The loading level of the Wang resin-bound oxime ether **4** was determined to be 0.81 mmol/g by quantification of nitrogen by elemental

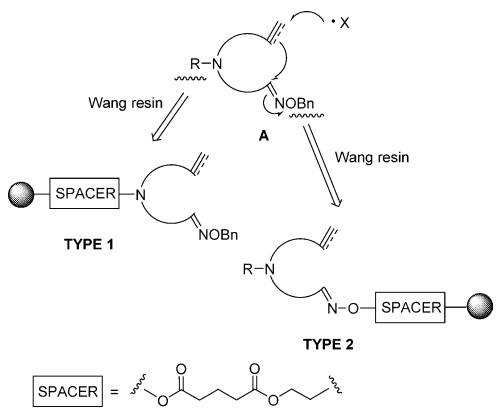

Fig. 1

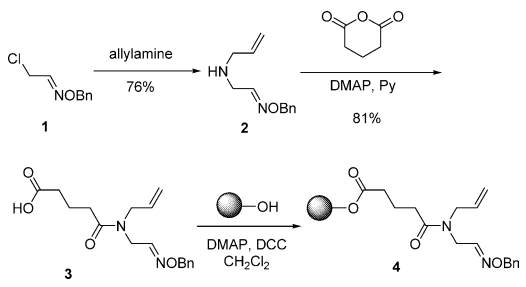

Chart 1

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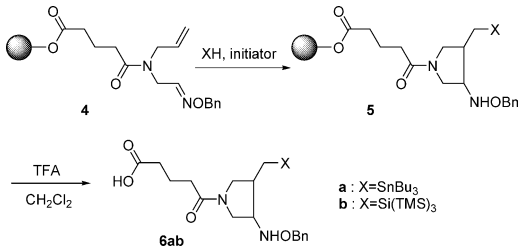

Chart 2

Table 1. Solid-Phase Radical Reaction of Oxime Ether **4**

Entry	XH	Solvent	T (°C)	Initiator	Product	Yield (%)[a]
1	Bu_3SnH	Toluene	80	AIBN	**6a**	47
2	Bu_3SnH	Toluene	80	Et_3B	**6a**	64
3	Bu_3SnH	$(CH_2Cl)_2$	80	Et_3B	**6a**	13
4	Bu_3SnH	CH_2Cl_2	35	Et_3B	**6a**	16
5	Bu_3SnH	Toluene	80	9-BBN	**6a**	Trace
6	Bu_3SnH	Toluene	80	Et_2Zn	**6a**	26
7	Et_3SiH	Toluene	80	Et_3B		ND
8	$(TMS)_3SiH$	Toluene	80	Et_3B	**6b**	50

a) Yields are for the isolated products after purification by preparative TLC.

analysis.

At first, we examined the stannyl radical addition-cyclization reaction of oxime ether **4** (Chart 2). The reaction of **4** with Bu_3SnH was carried out in toluene at 80 °C by using AIBN (1.0 eq×3) (Table 1, entry 1). After the reaction mixture was stirred at 80 °C for 20 h, the resin was then filtered and washed successively with CH_2Cl_2, AcOEt followed by MeOH, and the subsequent cleavage of the resin by treatment with TFA/CH_2Cl_2 (1 : 5, v/v) gave the crude product **6a**. Purification of **6a** was accomplished by preparative TLC (MeOH/$CHCl_3$ 1 : 20, v/v) to afford the product **6a** in 47% isolated yield. In the solid-phase reactions, the often-tedious workup to remove excess reagents from reaction mixture was eliminated simply by washing of the resin with solvents. We next examined the reaction by using triethylborane as a radical initiator. Triethylborane worked well at high temperature without interference of the polystyrene skeleton of the resin (entry 2). In contrast to the reaction using AIBN, the reaction using triethylborane proceeded smoothly. To a flask containing oxime ether **4** and Bu_3SnH in toluene was added three times a commercially available 1.0 M solution of triethylborane (13 eq) in hexane at 80 °C, and the reaction mixture was stirred at 80 °C for 8 h. The cyclic product **6a** was obtained in 64% isolated yield after cleavage of the resin.

The rationale of the triethylborane-induced reaction pathway is that the stannyl radical added to the olefin moiety of **4** to form intermediate alkyl radical A, which attacked intramolecularly the triethylborane-activated oxime ether group as in 5-*exo*-*trig* radical cyclization to form the benzyl–oxyaminyl radical B (Chart 3). The benzyloxyaminyl radical B was trapped by triethylborane as a radical terminator to give the product C and an ethyl radical, although another possible route to the product **6a** from B *via* the reaction with Bu_3SnH would not be rigorously excluded. In regard to the solvent effect, the reaction in $(CH_2Cl)_2$ or CH_2Cl_2 gave poor yields of the product **6a** (entries 3, 4). The use of 9-BBN[43] as a radical initiator was less effective for the reaction of **4**

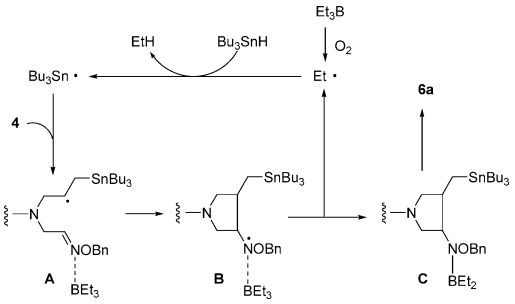

Chart 3

(entry 5). Recently, Ryu and Komatsu reported that diethylzinc-air system can serve as an initiator of tin hydride-mediated radical reaction as well as triethylborane.[44—46] In order to test the viability of diethylzinc on a solid support, we also investigated the reaction of oxime ether **4** using a commercially available 1.0 M solution of diethylzinc. Diethylzinc also worked as a radical initiator at high temperature to give a 26% yield of the product **6a** (entry 6). We also investigated the reaction using different radical precursors (entries 7, 8). The use of the less reactive triethylsilane did not give the desired product, probably due to the predominant formation of the ethylated product as a result of the competitive intermolecular addition of an ethyl radical, generated from triethylborane, to oxime ether group (entry 7).[47] Moderate chemical yield of **6b** was also observed in the addition-cyclization of oxime ether **4** using more reactive tris(trimethylsilyl)silane (entry 8). The cyclic products **6a** and **6b** were obtained as a diastereomeric mixture, although these stereostructures have not been determined.

To survey the scope and limitations of the triethylborane-induced radical addition-cyclization, we next investigated the reaction of different oxime ethers **9** and **13** anchored to polymer support (Chart 4). The reaction of alkyne-tethered oxime ethers such as **9** with a stannyl radical has been studied in solution phase.[48—50] Additionally, we recently developed the reaction between oxime ether group and alkoxycarbonyl-stabilized radicals in solution phase.[37—39] Thus, the solid-phase reaction of oxime ether **13** connected with the α,β-unsaturated carbonyl group is the new focus of our efforts.[17] The reaction of α-chloroacetaldoxime ether **1** with propargylamine gave the secondary amine **7** in 77% yield. The amine **7** was treated with glutaric anhydride to give the oxime ether **8**, which was attached to Wang resin by treatment with DCC in the presence of DMAP in CH_2Cl_2 at 20 °C for 12 h to give the Wang resin-bound oxime ether **9**. Wang resin-bound oxime ether **13** was easily prepared from α-chloroacetaldoxime ether **1** as shown in Chart 4.

At first, we examined the stannyl radical addition-cyclization of oxime ether **9** having propargyl group (Chart 5). Treatment of **9** with triethylborane in the presence of Bu_3SnH gave the cyclic product **15** in 77% isolated yield, after cleavage of the resin. The cyclic product **15** was stable under acidic conditions. Treatment of the cyclic product **15** with hydrogen chloride in ethanol did not give the protodestannylated product.[40] We next investigated of oxime ether **13** having an electron-deficient carbon–carbon double bond. As expected, the stannyl radical added to an electron-deficient carbon–carbon double bond as well as an isolated

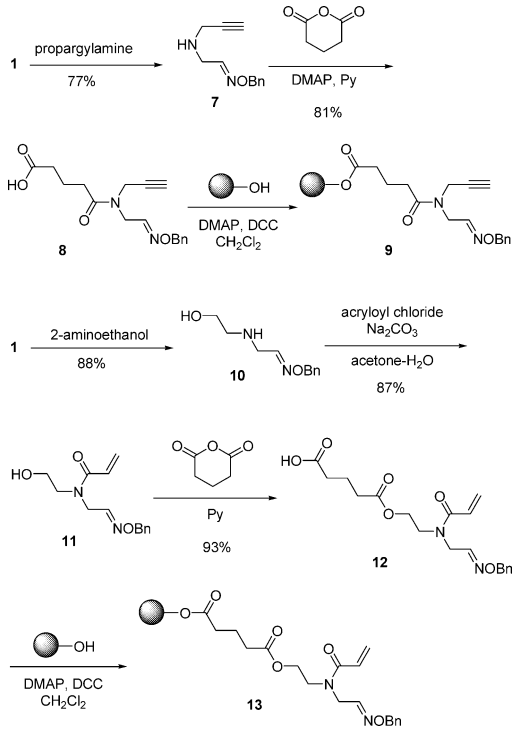

Chart 4

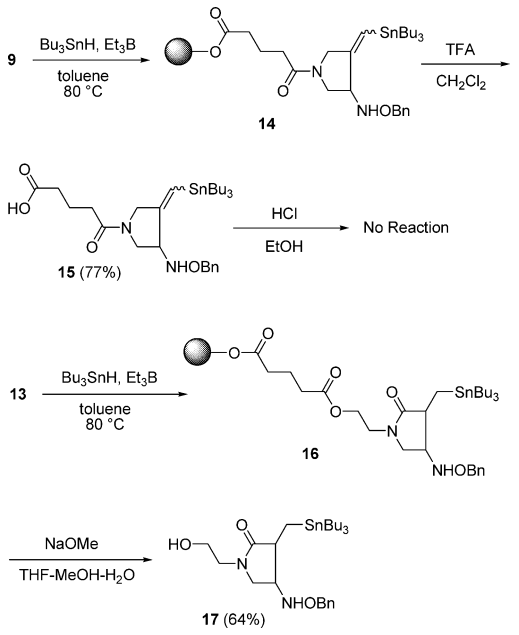

Chart 5

carbon–carbon double bond by using triethylborane as a radical initiator. Treatment of **13** with triethylborane in the presence of Bu_3SnH gave the cyclic product **17** in 64% yield, after cleavage of the resin.

The development of tandem carbon–carbon bonds-forming radical reactions is a new subject of considerable interest; thus, a number of extensive investigations were reported in recent years.[21] We also reported a new Mannich-type reaction based on tandem carbon–carbon bonds-forming radical reaction, which has been successfully applied to the asymmetric synthesis of γ-butyrolactones and β-amino acids.[37—39] We newly investigated the construction of two carbon–carbon

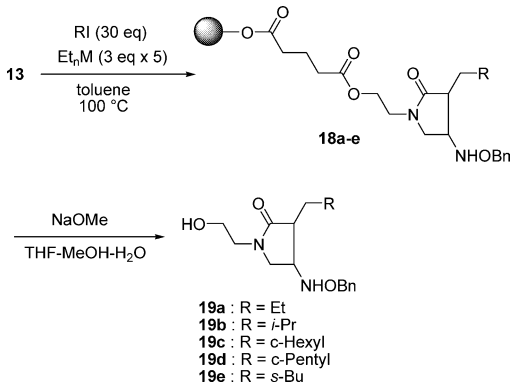

Chart 6

Table 2. Tandem Reaction of Oxime Ether **13**

Entry	RI	Et_nM	Product	Yield (%)[a]
1	None	Et_3B	**19a**	72
2	None	Et_2Zn	**19a**	12
3[b]	*i*-PrI	Et_3B	**19b**	69
4[b]	c-Hexyl I	Et_3B	**19c**	54
5[b]	c-Pentyl I	Et_3B	**19d**	59
6[b]	*s*-Bu I	Et_3B	**19e**	55
7[b]	*t*-Bu I	Et_3B		ND

a) Yields are for the isolated products after purification by preparative TLC. *b*) Reactions were carried out with RI (30 eq) and Et_3B (3 eq×3) in toluene at 100 °C.

bonds on a solid support by using Wang resin-bound oxime ether **13** (Chart 6).

The reaction of **13** with an ethyl radical proceeded effectively by the treatment of simple triethylborane (Table 2, entry 1). To a flask containing aldoxime ether **13** in toluene was added three times a commercially available 1.0 M solution of triethylborane in hexane (3 eq) at 100 °C. After the reaction mixture was stirred at 100 °C totally for 2 h, the resin was then filtered and successively washed with CH_2Cl_2, AcOEt, and MeOH. The cleavage of the resin by treatment with NaOMe gave the desired azacyclic product **19a** in 72% isolated yield. However, the tandem radical reaction of oxime ether **13** did not proceed at 25 °C. The reaction using diethylzinc as a radical initiator gave the desired product **19a** in 12% isolated yield, after cleavage of the resin (entry 2). The treatment of **13** with *i*-PrI (30 eq) and triethylborane (3 eq× 3) in toluene at 100 °C gave the desired azacyclic product **19b** in 69% isolated yield. A favorable experimental feature of this method is that the reaction proceeds smoothly even in the absence of toxic tin hydride or heavy metals *via* a route involving an iodine atom-transfer process (Chart 7).[51] Moreover, it is noteworthy that this reaction provided the new method for the construction of two carbon–carbon bonds on solid support under mild reaction conditions without strictly anhydrous solvents and reagents. Good chemical yields were also observed in the radical reaction using different radical precursors such as cyclohexyl, cyclopentyl, and *sec*-butyl iodides under the iodine atom-transfer reaction conditions, except for a bulky *tert*-butyl radical (entries 3—7). In this reaction, triethylborane acted as an effective reagent for trapping the intermediate aminyl radicals E to regenerate an ethyl radical; therefore, more than a stoichiometric amount of tri-

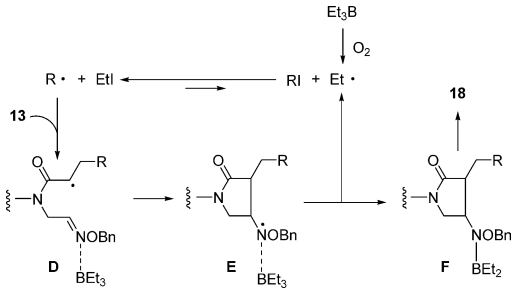

Chart 7

ethylborane would be required.

In conclusion, we have demonstrated the solid-phase tandem radical reaction of oxime ethers connected with olefin moieties. The reaction of oxime ethers with stannyl radical proceeded smoothly by the use of triethylborane as a radical initiator to provide the functionalized pyrrolidines. The tandem carbon–carbon bonds-forming reaction of oxime ether with alkyl radicals proceeded smoothly *via* a route involving an iodine atom-transfer process without strictly anhydrous solvents and reagents.

Experimental

General ^{1}H- and ^{13}C-NMR spectra were recorded at 200, 300, or 500 MHz and at 50 or 125 MHz, respectively. IR spectra were recorded using FTIR apparatus. Mass spectra were obtained by EI or CI methods. Preparative TLC separations were carried out on precoated silica gel plates (E. Merck $60F_{254}$). Flash column chromatography was performed using E. Merck Kieselgel 60 (230—400 mesh).

***N*-(2-Propenyl)aminoethanal *O*-Benzyloxime (2)** Preparation of **2** was reported in our previous article.[40]

4-[*N*-(2-(Benzyloxyimino)ethyl)-*N*-(2-propenyl)carbamoyl]butanoic Acid (3) To a solution of **2** (100 mg, 0.526 mmol) in pyridine (2.0 ml) were added glutaric anhydride (72.1 mg, 0.632 mmol) and DMAP (32.16 mg, 0.263 mmol) at room temperature. After being stirred at room temperature for 4 h, the reaction mixture was diluted with AcOEt, washed with 5% HCl, water, and brine, dried over Na_2SO_4 and concentrated at reduced pressure. Purification of the residue by flash columun chromatography (hexane/AcOEt 1 : 2) afforded **3** (136 mg, 81%) as a colorless oil and a 3 : 2 mixture of *E*/*Z*-oxime. The presence of rotamers and *E*/*Z*-isomers precluded a comprehensive assignment of all proton resonances. IR ($CHCl_3$) cm^{-1}: 3682, 2938, 1711, 1649, 1455. ^{1}H-NMR ($CDCl_3$) δ: 9.13 (1H, br s), 7.37 (3/5H, t, J=5.7 Hz), 7.40—7.26 (5H, m), 6.69 (2/5H, t, J=4.4 Hz), 5.82—5.60 (1H, m), 5.24—5.02 (4H, m), 4.27—3.80 (4H, m), 2.48—2.24 (4H, m), 2.04—1.84 (2H, m). HR-MS *m*/*z*: 318.1559 (Calcd for $C_{17}H_{22}N_2O_4$ (M^+): 318.1578).

Attachment of Oxime Ether 3 to Wang Resin To a suspension of Wang resin (0.83 mmol/g, 1.0 g, 0.83 mmol) in CH_2Cl_2 (50 ml) were added the acid derivative **3** (792 mg, 2.49 mmol), DCC (856 mg, 4.15 mmol) and DMAP (50.7 mg, 0.415 mmol) under a nitrogen atmosphere at room temperature. After the reaction mixture was stirred at the same temperature for 1 h and then staid for 11 h, the resin was filtered, washed well with CH_2Cl_2, AcOEt followed by MeOH and then dried *in vacuo* to give the Wang resin-bound oxime ether **4**.

Radical Reaction of Oxime Ether 4 Using AIBN (Table 1, Entry 1) To a suspension of **4** (250 mg, 0.208 mmol) and Bu_3SnH (0.182 ml, 0.675 mmol) in toluene (10 ml) was added portionwise a solution of AIBN (34 mg, 0.208 mmol) in toluene (1.5 ml) under a nitrogen atmosphere at 80 °C. After being stirred at 80 °C for 5 h, Bu_3SnH (0.182 ml, 0.675 mmol) and a solution of AIBN (34 mg, 0.208 mmol) in toluene (1.5 ml) were added to the reaction mixture. After being stirred at 80 °C for 5 h, Bu_3SnH (0.182 ml, 0.675 mmol) and a solution of AIBN (34 mg, 0.208 mmol) in toluene (1.5 ml) were added to the reaction mixture. After being stirred at 80 °C for 10 h, the resin was filtered, washed well with CH_2Cl_2, AcOEt, and MeOH, and then dried *in vacuo*. To a flask with the resulting resin was added TFA/CH_2Cl_2 (1 : 5, v/v, 4.0 ml) under a nitrogen atmosphere at room temperature. After the reaction mixture was stirred at the same temperature for 30 min, the reaction mixture was filtered and washed with CH_2Cl_2 (50 ml), and the filtrate was concentrated at reduced pressure. After the resulting residue was dissolved in CH_2Cl_2, the organic phase was washed with diluted aqueous $NaHCO_3$, and water, dried over $MgSO_4$, and concentrated at reduced pressure. Purification of the residue by preparative TLC ($CHCl_3$/MeOH 20 : 1) afforded product **6a** (57.4 mg, 45%).

Radical Reaction of Oxime Ether 4 Using Et_3B (Table 1, Entries 2 and 8) To a suspension of **4** (250 mg, 0.208 mmol) and Bu_3SnH or $(TMS)_3SiH$ (0.675 mmol) in toluene (10 ml) was added portionwise a 1.0 M solution of Et_3B in hexane (2.7 ml, 2.70 mmol) under a nitrogen atmosphere at 80 °C. After being stirred at 80 °C for 3 h, Bu_3SnH or $(TMS)_3SiH$ (0.675 mmol) and Et_3B in hexane (2.7 ml, 2.70 mmol) were added to the reaction mixture. After being stirred at 80 °C for 3 h, Bu_3SnH or $(TMS)_3SiH$ (0.675 mmol) and Et_3B in hexane (2.7 ml, 2.70 mmol) were added to the reaction mixture. After being stirred at 80 °C for 2 h, the resin was filtered, washed well with CH_2Cl_2, AcOEt, and MeOH, and then dried *in vacuo*. To a flask with the resulting resin was added TFA/CH_2Cl_2 (1 : 5, v/v, 4.0 ml) under a nitrogen atmosphere at 20 °C. After the reaction mixture was stirred at the same temperature for 30 min, the reaction mixture was filtered and washed with CH_2Cl_2 (50 ml), and the filtrate was concentrated at reduced pressure. After the resulting residue was dissolved in CH_2Cl_2, the organic phase was washed with diluted aqueous $NaHCO_3$, and water, dried over $MgSO_4$, and concentrated at reduced pressure. Purification of the residue by preparative TLC ($CHCl_3$/MeOH 20 : 1) afforded products **6a** and **6b**.

5-[3-(Benzyloxyamino)-4-((tributylstannyl)methyl)pyrrolidin-1-yl]-5-oxopentanoic Acid (6a) The presence of rotamers and isomers precluded a comprehensive assignment of all proton resonances. As a colorless oil and a 1 : 1 *trans*/*cis* mixture: IR ($CHCl_3$) cm^{-1}: 2927, 1713, 1626. ^{1}H-NMR ($CDCl_3$) δ: 7.40—7.30 (5H, m), 6.21 (2H, br s), 4.68 (1H, s), 4.67 (1H, br s), 3.90—2.10 (11H, m), 1.97 (2H, m), 1.62—0.67 (28H, m). HR-MS *m*/*z*: 610.2794 (Calcd for $C_{29}H_{50}N_2O_4Sn^{120}$ (M^+): 610.2790) and 608.2778 (Calcd for $C_{29}H_{50}N_2O_4Sn^{118}$ (M^+): 608.2784).

5-[3-(Benzyloxyamino)-4-((tristrimethylsilyl)methyl)pyrrolidin-1-yl]-5-oxopentanoic Acid (6b) The presence of rotamers and isomers precluded a comprehensive assignment of all proton resonances. As a a colorless oil and a 1 : 1 *trans*/*cis* mixture: IR ($CHCl_3$) cm^{-1}: 3691, 1730, 1602. ^{1}H-NMR ($CDCl_3$) δ: 7.39—7.30 (5H, m), 7.15 (2H, br s), 4.67 (2H, br s), 4.00—2.90 (5H, m), 2.50—2.20 (4H, m), 1.95 (2H, m) 1.10—0.60 (3H, m), 0.19 (27H, s). HR-MS *m*/*z*: 566.2850 (Calcd for $C_{26}H_{50}N_2O_4Si_4$ (M^+): 566.2845).

***N*-(2-Propynyl)aminoethanal *O*-Benzyloxime (7)** Preparation of **7** was reported in our previous article.[40]

4-[*N*-(2-(Benzyloxyimino)ethyl)-*N*-(2-propynyl)carbamoyl]butanoic Acid (8) To a solution of **7** (5.85 g, 31.1 mmol) in pyridine (15.0 ml) were added glutaric anhydride (4.26 g, 37.4 mmol) and DMAP (761 mg, 6.23 mmol) at room temperature. After being stirred at room temperature for 3 h, the reaction mixture was diluted with AcOEt, washed with 5% HCl, water, and brine, dried over Na_2SO_4 and concentrated at reduced pressure. Purification of the residue by flash columun chromatography (hexane/AcOEt 1 : 2) afforded **8** (7.31 g, 74%) as a colorless oil and a 3 : 2 mixture of *E*/*Z*-oxime. The presence of rotamers and *E*/*Z*-isomers precluded a comprehensive assignment of all proton resonances. IR ($CHCl_3$) cm^{-1}: 3694, 1711, 1655. ^{1}H-NMR ($CDCl_3$) δ: 7.80 (1H, br s), 7.42 (3/10H, t, J=4.9 Hz), 7.36 (3/10H, t, J=5.8 Hz), 7.34—7.27 (5H, m), 6.80 (2/10H, t, J=4.1 Hz), 6.74 (2/10H, t, J=4.3 Hz), 5.13 (2/5H, s), 5.11 (2/5H, s), 5.06 (6/5H, br s), 4.34—3.94 (4H, m), 2.56—2.20 (5H, m), 2.06—1.84 (2H, m). HR-MS *m*/*z*: 316.1444 (Calcd for $C_{17}H_{20}N_2O_4$ (M^+): 316.1421).

Attachment of Oxime Ether 8 to Wang Resin To a suspension of Wang resin (0.83 mmol/g, 12.2 g, 10.2 mmol) in CH_2Cl_2 (200 ml) were added the acid derivative **8** (6.42 g, 20.3 mmol), DCC (10.5 g, 50.8 mmol) and DMAP (621 mg, 5.08 mmol) under a nitrogen atmosphere at room temperature. After the reaction mixture was stirred at the same temperature for 1 h and then staid for 11 h, the resin was filtered, washed well with CH_2Cl_2, AcOEt followed by MeOH and then dried *in vacuo* to give the Wang resin-bound oxime ether **9**.

***N*-[2-(Benzyloxyimino)ethyl]-*N*-(2-hydroxyethyl)-2-propenamide (11)** Preparation of **11** was reported in our previous article.[40]

4-[(2-(*N*-(2-(Benzyloxyimino)ethyl)-*N*-(2-propenoyl)amino)ethoxy)carbonyl]butanoic Acid (12) To a solution of **11** (100 mg, 0.382 mmol) in pyridine (1.0 ml) was added glutaric anhydride (65.3 mg, 0.573 mmol) at room temperature. After being stirred at 100 °C for 2 h, glutaric anhydride (43.6 mg, 0.382 mmol) was added to the reaction mixture. After being stirred at 100 °C for 2 h, the reaction mixture was diluted with AcOEt, washed with 5% HCl, water, and brine, dried over Na_2SO_4 and concentrated at reduced pressure. Purification of the residue by flash columun chromatography ($CHCl_3$/MeOH 30 : 1) afforded **12** (134 mg, 93%) as a colorless oil

and a 1 : 1 mixture of *E*/*Z*-oxime. The presence of rotamers and *E*/*Z*-isomers precluded a comprehensive assignment of all proton resonances. IR ($CHCl_3$) cm^{-1}: 3619, 1965, 1735, 1651. ^{1}H-NMR ($CDCl_3$) δ: 8.25 (1H, br s), 7.45—7.25 (11/2H, m), 6.34 (1/2H, t, J=4.5 Hz), 6.64—6.28 (2H, m), 5.78—5.66 (1H, m), 5.14 (1/2H, s), 5.13 (1/2H, s), 5.06 (1H, br s), 4.33—4.14 (4H, m), 3.69—3.55 (2H, m), 2.43—2.28 (4H, m), 1.99—1.82 (2H, m). HR-MS *m*/*z*: 376.1649 (Calcd for $C_{19}H_{24}N_2O_6$ (M^+): 376.1632).

Attachment of Oxime Ether 12 to Wang Resin To a suspension of Wang resin (0.83 mmol/g, 4.27 g, 3.55 mmol) in CH_2Cl_2 (100 ml) were added the acid derivative **12** (2.00 g, 5.32 mmol), DCC (2.74 g, 13.3 mmol) and DMAP (162 mg, 1.33 mmol) under a nitrogen atmosphere at room temperature. After the reaction mixture was stirred at the same temperature for 1 h and then staid for 11 h, the resin was filtered, washed well with CH_2Cl_2, AcOEt followed by MeOH and then dried *in vacuo* to give the Wang resin-bound oxime ether **13**.

5-[3-(Benzyloxyamino)-4-((tributylstannyl)methylene)pyrrolidin-1-yl]-5-oxopentanoic Acid (15) To a suspension of **9** (250 mg, 0.208 mmol) and Bu_3SnH (0.208 ml, 0.675 mmol) in toluene (10 ml) was added portionwise a 1.0 M solution of Et_3B in hexane (2.08 ml, 2.08 mmol) under a nitrogen atmosphere at 80 °C. After being stirred at 80 °C for 2 h, Bu_3SnH (0.208 ml, 0.675 mmol) and Et_3B in hexane (2.08 ml, 2.08 mmol) were added to the reaction mixture. After being stirred at 80 °C for 2 h, Bu_3SnH (0.208 ml, 0.675 mmol) and Et_3B in hexane (2.08 ml, 2.08 mmol) were added to the reaction mixture. After being stirred at 80 °C for 2 h, the resin was filtered, washed well with CH_2Cl_2, AcOEt, and MeOH, and then dried *in vacuo*. To a flask with the resulting resin was added TFA/CH_2Cl_2 (1 : 5, v/v, 4.0 ml) under a nitrogen atmosphere at room temperature. After the reaction mixture was stirred at the same temperature for 30 min, the reaction mixture was filtered and washed with CH_2Cl_2 (50 ml), and the filtrate was concentrated at reduced pressure. After the resulting residue was dissolved in CH_2Cl_2, the organic phase was washed with diluted aqueous $NaHCO_3$, and water, dried over $MgSO_4$, and concentrated at reduced pressure. Purification of the residue by preparative TLC ($CHCl_3$/MeOH 20 : 1) afforded product **15** (91 mg, 77%). The presence of rotamers and *E*/*Z*-isomers precluded a comprehensive assignment of all proton resonances. As a colorless oil and a 1 : 1 *E*/*Z* mixture: IR ($CHCl_3$) cm^{-1}: 2960, 1636. ^{1}H-NMR ($CDCl_3$) δ: 7.40—7.23 (5H, m), 5.28—5.06 (2H, m), 4.74—4.64 (2H, m), 4.24—3.96 (3H, m), 3.80—3.50 (2H, m), 2.42—2.22 (4H, m), 1.94 (2H, m), 1.59 (6H, m), 1.34 (6H, m), 1.25 (6H, m), 0.91 (9H, t, J=7.4 Hz). HR-MS *m*/*z*: 608.2646 (Calcd for $C_{29}H_{48}N_2O_4Sn^{120}$ (M^+): 608.2633) and 606.2617 (Calcd for $C_{29}H_{48}N_2O_4Sn^{118}$ (M^+): 606.2627).

4-(Benzyloxyamino)-3-[(tributylstannyl)methyl]-1-(2-hydroxyethyl)-pyrrolidin-2-one (17)[40] To a suspension of **13** (200 mg, 0.166 mmol) and Bu_3SnH (0.179 ml, 0.664 mmol) in toluene (5 ml) was added portionwise a 1.0 M solution of Et_3B in hexane (0.664 ml, 0.664 mmol) under a nitrogen atmosphere at 80 °C. After being stirred at 80 °C for 30 min, Bu_3SnH (0.179 ml, 0.664 mmol) and Et_3B in hexane (0.664 ml, 0.664 mmol) were added to the reaction mixture. After being stirred at 80 °C for 30 min, Bu_3SnH (0.179 ml, 0.664 mmol) and Et_3B in hexane (0.664 ml, 0.664 mmol) were added to the reaction mixture. After being stirred at 80 °C for 1 h, the resin was filtered, washed well with CH_2Cl_2, AcOEt, and MeOH, and then dried *in vacuo*. To a flask with the resulting resin were added THF (6 ml), MeOH (3 ml), H_2O (0.1 ml), and NaOMe (a 5.2 M solution of in MeOH, 0.08 ml, 0.415 mmol) under a nitrogen atmosphere at room temperature. After the reaction mixture was stirred at the same temperature for 1 h, the reaction mixture was filtered and washed with CH_2Cl_2 (50 ml), and the filtrate was concentrated at reduced pressure. After the resulting residue was dissolved in CH_2Cl_2, the organic phase was washed with diluted aqueous $NaHCO_3$, and water, dried over $MgSO_4$, and concentrated at reduced pressure. Purification of the residue by preparative TLC ($CHCl_3$/MeOH 20 : 1) afforded product **17** (58.4 mg, 64%). Major isomer: as a colorless oil: IR ($CHCl_3$) cm^{-1}: 3368, 2925, 1674, 1488. ^{1}H-NMR ($CDCl_3$) δ: 7.40—7.25 (5H, m), 4.70 (2H, s), 3.72 (2H, t, J=5.1 Hz), 3.55 (1H, m), 3.46—3.26 (4H, m), 2.43 (1H, dt, J=5.7, 8.1 Hz), 1.52—1.41 (4H, m), 1.36—1.22 (6H, m), 0.92—0.81 (21H, m). ^{13}C-NMR ($CDCl_3$) δ: 177.4, 137.2, 128.4, 128.3, 128.0, 76.5, 63.5, 60.6, 50.9, 46.1, 43.9, 29.0, 27.3, 13.6, 9.7. One carbon peak was missing due to overlapping. HR-MS *m*/*z*: 554.2548 (Calcd for $C_{26}H_{46}N_2O_3Sn^{120}$ (M^+): 554.2528). Minor isomer: as a colorless oil: IR ($CHCl_3$) cm^{-1}: 3368, 2925, 1675, 1486. ^{1}H-NMR ($CDCl_3$) δ: 7.40—7.25 (5H, m), 4.70 (2H, s), 3.72 (2H, t, J=5.4 Hz), 3.64 (1H, m), 3.51—3.26 (4H, m), 2.69 (1H, dt, J=7.5, 9.3 Hz), 1.55—1.40 (4H, m), 1.38—1.21 (6H, m), 0.92—0.81 (21H, m). ^{13}C-NMR ($CDCl_3$) δ: 177.1, 137.1, 128.5, 128.4, 128.0, 76.4, 60.6, 58.0, 51.0, 46.2, 43.2, 29.1, 27.3, 13.6, 9.9. One carbon peak was missing due to overlapping. HR-MS *m*/*z*: 554.2540 (Calcd for $C_{26}H_{46}N_2O_3Sn^{120}$ (M^+): 554.2528).

General Procedure for Reaction of Oxime Ether 13 with Alkyl Radical To a suspension of **13** (200 mg, 0.166 mmol) and RI (4.98 mmol) in toluene (5 ml) was added portionwise a 1.0 M solution of Et_3B in hexane (0.498 ml, 0.498 mmol) under a nitrogen atmosphere at 100 °C. After being stirred at 100 °C for 30 min, Et_3B in hexane (0.498 ml, 0.498 mmol) was added to the reaction mixture. After being stirred at 100 °C for 30 min, Et_3B in hexane (0.498 ml, 0.498 mmol) was added to the reaction mixture. After being stirred at 100 °C for 1 h, the resin was filtered, washed well with CH_2Cl_2, AcOEt, and MeOH, and then dried *in vacuo*. To a flask with the resulting resin were added THF (6 ml), MeOH (3 ml), H_2O (0.1 ml), and NaOMe (5.2 M solution of in MeOH, 0.064 ml, 0.332 mmol) under a nitrogen atmosphere at room temperature. After the reaction mixture was stirred at the same temperature for 1 h, the reaction mixture was filtered and washed with CH_2Cl_2 (50 ml), and the filtrate was concentrated at reduced pressure. After the resulting residue was dissolved in CH_2Cl_2, the organic phase was washed with diluted aqueous $NaHCO_3$, and water, dried over $MgSO_4$, and concentrated at reduced pressure. Purification of the residue by preparative TLC (AcOEt) afforded product **19a**—**e**.

4-(Benzyloxyamino)-1-(2-hydroxyethyl)-3-propylpyrrolidin-2-one (19a)[38] *trans*-Isomer: As a colorless oil: IR ($CHCl_3$) cm^{-1}: 3401, 1670. ^{1}H-NMR ($CDCl_3$) δ: 7.38—7.30 (5H, m), 5.66—5.40 (1H, br m), 4.70 (2H, s), 3.72 (2H, t, J=5.0 Hz), 3.57—3.50 (2H, m), 3.43 (1H, m), 3.36—3.30 (2H, m), 2.30 (1H, dt, J=8.5, 4.5 Hz), 2.08—1.90 (1H, br m), 1.71 (1H, m), 1.51—1.36 (3H, m), 0.92 (3H, t, J=7.0 Hz). ^{13}C-NMR ($CDCl_3$) δ: 176.6, 137.3, 128.6, 128.5, 128.1, 76.7, 60.6, 59.2, 51.5, 46.1, 45.8, 32.0, 20.2, 14.0. HR-MS *m*/*z*: 292.1803 (Calcd for $C_{16}H_{24}N_2O_3$ (M^+): 292.1785). *cis*-Isomer: As a colorless oil: IR ($CHCl_3$) cm^{-1}: 3371, 1673, 1454. ^{1}H-NMR ($CDCl_3$) δ: 7.37—7.30 (5H, m), 5.69—5.43 (1H, br m), 4.69 (1H, d, J=11.5 Hz), 4.66 (1H, d, J=11.5 Hz), 3.71 (2H, t, J=5.0 Hz), 3.49—3.29 (4H, m), 2.51—2.47 (1H, m), 1.75—1.71 (1H, m), 1.47—1.33 (3H, m), 0.94 (3H, t, J=7.0 Hz). ^{13}C-NMR ($CDCl_3$) δ: 176.2, 137.2, 128.7, 128.5, 128.1, 76.5, 60.5, 56.1, 51.3, 46.1, 44.8, 26.1, 21.1, 14.1. HR-MS *m*/*z*: 292.1781 (Calcd for $C_{16}H_{24}N_2O_3$ (M^+): 292.1785).

4-(Benzyloxyamino)-1-(2-hydroxyethyl)-3-isobutylpyrrolidin-2-one (19b)[38] *trans*-Isomer: As a colorless oil: IR ($CHCl_3$) cm^{-1}: 2959, 1670, 1488, 1455. ^{1}H-NMR ($CDCl_3$) δ: 7.40—7.25 (5H, m), 5.69—5.43 (1H, br m), 4.70 (2H, s), 3.78—3.63 (2H, m), 3.60—3.23 (6H, m), 2.34 (1H, m), 1.82—1.50 (2H, m), 1.40—1.21 (1H, m), 0.928 (3H, d, J=6.2 Hz), 0.908 (3H, d, J=6.2 Hz). ^{13}C-NMR ($CDCl_3$) δ: 176.7, 137.3, 128.6, 128.4, 128.1, 76.7, 60.4, 59.6, 51.2, 45.9, 44.1, 39.1, 25.8, 23.1, 21.8. HR-MS *m*/*z*: 306.1955 (Calcd for $C_{17}H_{26}N_2O_3$ (M^+): 306.1941). *cis*-Isomer: As a colorless oil: IR ($CHCl_3$) cm^{-1}: 2959, 1674, 1486, 1467. ^{1}H-NMR ($CDCl_3$) δ: 7.40—7.25 (5H, m), 5.62—5.39 (1H, br m), 4.69 (1H, d, J=11.8 Hz), 4.66 (1H, d, J=11.8 Hz), 3.79—3.62 (3H, m), 3.58—3.23 (4H, m), 3.18—2.97 (1H, br m), 2.57 (1H, m), 1.80—1.21 (3H, m), 0.926 (3/3H, d, J=6.2 Hz), 0.911 (3/3H, d, J=6.2 Hz). ^{13}C-NMR ($CDCl_3$) δ: 176.3, 137.0, 128.6, 128.3, 128.0, 76.4, 60.5, 56.2, 51.2, 46.0, 42.8, 32.5, 26.0, 23.1, 21.7. HR-MS *m*/*z*: 306.1941 (Calcd for $C_{17}H_{26}N_2O_3$ (M^+): 306.1941).

4-(Benzyloxyamino)-3-(cyclohexylmethyl)-1-(2-hydroxyethyl)pyrrolidin-2-one (19c)[38] *trans*-Isomer: As a colorless oil: IR ($CHCl_3$) cm^{-1}: 2926, 1671, 1488, 1450. ^{1}H-NMR ($CDCl_3$) δ: 7.40—7.25 (5H, m), 4.70 (2H, s), 3.70 (2H, t, J=5.2 Hz), 3.61—3.26 (6H, m), 2.37 (1H, m), 1.77—0.83 (14H, m). ^{13}C-NMR ($CDCl_3$) δ: 177.1, 137.3, 128.6, 128.5, 128.1, 76.7, 60.4, 59.7, 51.2, 45.9, 43.4, 37.6, 35.2, 33.9, 32.5, 26.5, 26.2, 26.1. HR-MS *m*/*z*: 346.2249 (Calcd for $C_{20}H_{30}N_2O_3$ (M^+): 346.2254). *cis*-Isomer: As a colorless oil: IR ($CHCl_3$) cm^{-1}: 2926, 1674, 1486, 1449. ^{1}H-NMR ($CDCl_3$) δ: 7.40—7.25 (5H, m), 5.61—5.33 (1H, br m), 4.71 (1H, d, J=11.6 Hz), 4.65 (1H, d, J=11.6 Hz), 3.72 (2H, t, J=5.0 Hz), 3.66 (1H, m), 3.52—3.24 (4H, m), 3.15—2.92 (1H, br m), 2.59 (1H, m), 1.80—0.45 (13H, m). ^{13}C-NMR ($CDCl_3$) δ: 176.5, 137.2, 128.8, 128.5, 128.2, 76.5, 60.7, 56.5, 51.3, 46.2, 42.3, 35.6, 33.9, 32.7, 31.2, 26.5, 26.3, 26.2. HR-MS *m*/*z*: 346.2239 (Calcd for $C_{20}H_{30}N_2O_3$ (M^+): 346.2254).

4-(Benzyloxyamino)-3-(cyclopentylmethyl)-1-(2-hydroxyethyl)pyrrolidin-2-one (19d)[38] *trans*-Isomer: As a colorless oil: IR ($CHCl_3$) cm^{-1}: 2952, 2468, 1670, 1489, 1454. ^{1}H-NMR ($CDCl_3$) δ: 7.40—7.25 (5H, m), 4.70 (2H, s), 3.71 (2H, t, J=5.6 Hz), 3.62—3.26 (6H, m), 2.30 (1H, m), 2.02—0.90 (11H, m). ^{13}C-NMR ($CDCl_3$) δ: 176.7, 137.2, 128.5, 128.3, 127.9, 76.5, 60.3, 59.2, 51.2, 45.9, 45.2, 37.6, 36.0, 32.9, 32.1, 25.1, 24.8. HR-MS *m*/*z*: 332.2112 (Calcd for $C_{19}H_{28}N_2O_3$ (M^+): 332.2099). *cis*-Isomer: As a colorless oil: IR ($CHCl_3$) cm^{-1}: 3401, 2951, 2464, 1678, 1485. ^{1}H-NMR ($CDCl_3$) δ: 7.40—7.25 (5H, m), 5.72—5.28 (1H, br m), 4.71 (1H, d, J=11.6 Hz), 4.65 (1H, d, J=11.6 Hz), 3.76—3.67 (3H, m), 3.52—3.24 (4H, m), 3.18—2.81 (1H, br m), 2.51 (1H, m), 2.00—1.00 (11H, m). ^{13}C-NMR

($CDCl_3$) δ: 176.2, 137.1, 128.6, 128.3, 128.0, 76.4, 60.5, 56.3, 51.2, 46.1, 44.1, 38.0, 33.0, 32.0, 29.7, 25.0, 24.9. HR-MS *m/z*: 332.2127 (Calcd for $C_{19}H_{28}N_2O_3$ (M^+): 332.2099).

4-(Benzyloxyamino)-1-(2-hydroxyethyl)-3-(2-methylbutyl)pyrrolidin-2-one (19e)[38] *trans*-Isomer: As a colorless oil and a 1 : 1 mixture of diastereomers concerning *sec*-butyl group: IR ($CHCl_3$) cm^{-1}: 3396, 2963, 1664, 1488, 1455. ^{1}H-NMR ($CDCl_3$) δ: 7.40—7.25 (5H, m), 4.70 (2H, s), 3.71 (2H, t, J=5.4 Hz), 3.61—3.24 (5H, m), 2.42—2.30 (1H, m), 1.78—0.80 (11H, m). ^{13}C-NMR ($CDCl_3$) δ: 176.4, 137.2, 128.5, 128.4, 128.3, 127.9, 76.5, 60.3, 59.7, 59.4, 51.2, 51.0, 45.8, 43.8, 43.7, 37.1, 36.7, 31.94, 31.85, 29.9, 28.4, 19.2, 18.3, 11.1, 10.9. HR-MS *m/z*: 320.2088 (Calcd for $C_{18}H_{28}N_2O_3$ (M^+) 320.2098). *cis*-Isomer: As a colorless oil and a 1 : 1 mixture of diastereomers concerning *sec*-butyl group: IR ($CHCl_3$) cm^{-1}: 3401, 2962, 1674, 1455. ^{1}H-NMR ($CDCl_3$) δ: 7.40—7.25 (5H, m), 4.71 (1H, d, J=11.6 Hz), 4.65 (1H, d, J=11.6 Hz), 3.75—3.65 (3H, m), 3.52—3.24 (4H, m), 2.64—2.50 (1H, m), 1.80—0.80 (11H, m). ^{13}C-NMR ($CDCl_3$) δ: 176.3, 137.0, 128.6, 128.4, 128.0, 76.4, 60.5, 56.6, 56.0, 51.22, 51.18, 46.1, 42.7, 42.6, 32.3, 32.2, 30.6, 30.14, 30.08, 28.5, 19.4, 18.5, 11.3, 11.0. HR-MS *m/z*: 320.2088 (Calcd for $C_{18}H_{28}N_2O_3$ (M^+) 320.2098).

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