

Stereoselective Synthesis of (4*S*,5*S*)-5-Vinylloxazolidin-2-one-4-carboxylate as a β -Vinylserine Synthetic Equivalent by Vinyl Grignard Addition to an *N*-Tosyl Version of Garner's Aldehyde

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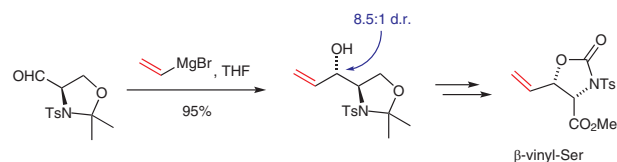
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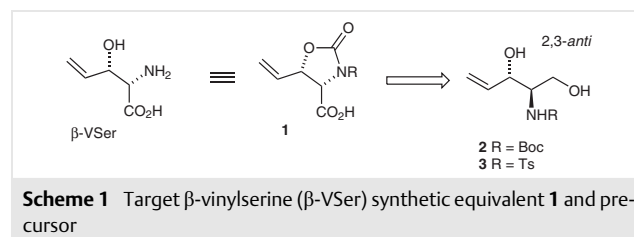
Abstract A highly efficient synthesis of a β -vinylserine synthetic equivalent is reported that exploits the stereodirecting effect of the *N*-toluenesulfonamide in an *anti*-diastereoselective (8.5:1) vinyl Grignard addition to an analogue of Garner's aldehyde. Both aryl and alkyl Grignards are shown to give increased *anti*-selectivity compared with *N*-Boc Garner's aldehyde.

Key words vinylserine, alkenyl amino acid, Garner aldehyde, oxazolidinones

As part of our synthetic work directed toward glycopeptide mimetics, we required a suitably protected (2*S*,3*S*)- β -vinylserine (β -VSer) for use as a synthetic building block. Many noncanonical amino acids have been incorporated into protein and peptide structures to interrogate various cellular functions.¹ In particular, alkenyl amino acids incorporated into peptides have proven to be useful for peptide stapling by a cross-metathesis reaction to afford conformationally restricted peptidomimetics.² In addition, Zhang and van der Donk have examined the effect of direct alkenyl amino acid incorporation.³ They incorporated a diastereomer of our desired β -VSer (referred to as a threonine analogue) into a peptide sequence of lactacin synthetase to examine substrate selectivity toward dehydration reactions. The pentenoic backbone of β -VSer itself is also a common scaffold for dipeptide isosteres,⁴ which have been investigated as enzyme inhibitors and as receptor antagonists.⁵

This platform has also been a versatile synthetic intermediate for preparing sphingomyelin analogues⁶ and glycosidase inhibitors such as the deoxynojirimycins.⁷ It has also served as a building block for antitumor agents such as 2-*epi*-pachastrissamine⁸ or for glycopeptide⁹ and β -lactam antibiotics.¹⁰ For our purposes, we sought to elaborate the β -VSer alkene through cross-metathesis and/or Trost-Tsujii π -allylic alkylation chemistry for the development of novel glycopeptides.

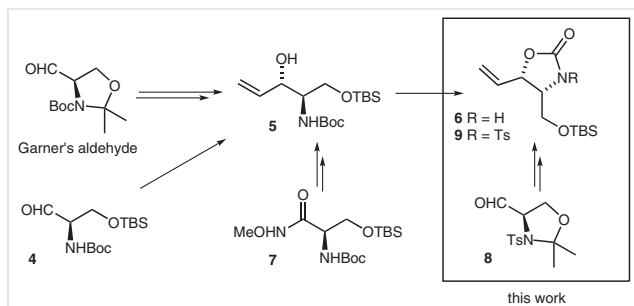
Given the versatility and interest in this simple building block, we elected to exploit an oxazolidinone scaffold **1** as a β -VSer synthetic equivalent in which both the amine and the hydroxy functions are simultaneously protected (Scheme 1). Although there are excellent reports on carbamate cyclizations¹¹ and an allylic C-H amination¹² that yield *trans*-4,5-disubstituted oxazolidinones stereospecifically, our studies required a *cis*-oxazolidinone. *cis*-4,5-Disubstituted oxazolidinones of this sort are known and are commonly derived from *anti*-2-aminopent-4-en-1,3-diols such as **2**.



Scheme 1 Target β -vinylserine (β -VSer) synthetic equivalent **1** and precursor

Both vinyl oxazolidinones and functionalized 2-aminopent-4-en-1,3-diols are valuable synthetic intermediates that have been used to prepare numerous natural products and medicinal targets, as discussed above. Although synthetic approaches from carbohydrates,¹³ azide epoxide

openings,^{6a} and chiral glycine enolate aldols¹⁴ are available, the more common synthetic approaches entailing nucleophilic additions to α -amino- β -hydroxy aldehydes or ketones provide varying degrees of control of stereochemistry (Scheme 2).



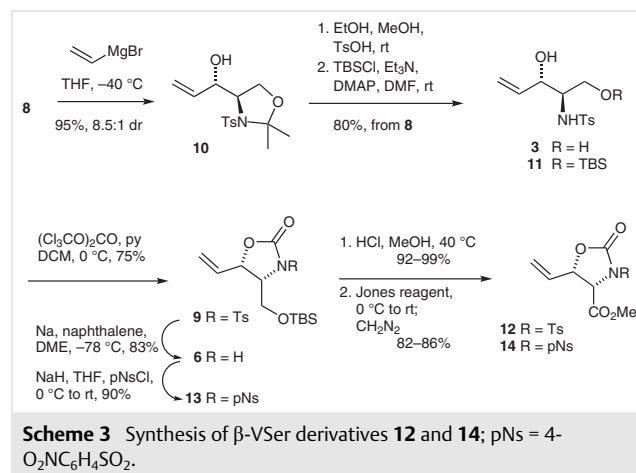
Scheme 2 Approaches to *cis*-oxazolidinones

A survey of the literature indicated one could proceed by a vinyl Grignard addition onto the well-known D-serine-derived Boc-protected Garner's aldehyde¹⁵ or the OTBS-Boc-serinal **4**,^{7a,10} followed by an intramolecular cyclization onto the Boc group to form an oxazolidinone. The Grignard approach has been widely used,^{7b,9,16} but is limited due to the selectivity of the Grignard addition; this led Herold to develop a three-step approach employing trimethylsilyl acetylide additions for improved *anti*-stereoselectivity.¹⁷ Although the *tert*-butyl(dimethyl)silyl ether substrate **4** gives **5** directly, it results in an undesirable 1:2 *anti/syn* diastereomeric ratio.^{7a} The typical *anti*-selectivity for vinyl addition to Garner's aldehyde is reported to range from 3:1^{16a} to 6:1 *anti/syn*, and experimental details indicate that additional purification by chromatography is necessary. From the Grignard product of Garner's aldehyde, hydrolysis of the *N,O*-acetal and selective protection of the primary hydroxy groups is needed, followed by formation of the oxazolidinone by a base-induced intramolecular cyclization onto the *tert*-butyl carbamate to afford **6**.¹⁸ In an improvement to these early approaches, the Weinreb amide **7** of a protected D-serine, available in four steps, has been employed to form an enone upon addition of vinylmagnesium bromide; this enone can be stereoselectively reduced with Li(*t*-BuO)₃AlH in ethanol giving **5** with a 10:1 preference toward the *anti*-diastereomer.¹⁹

Here, we report a highly selective alternative approach in which the *N*-tosylamide **8** is used as a stereodirecting orthogonal protecting group; this approach is complementary to the approaches discussed above.

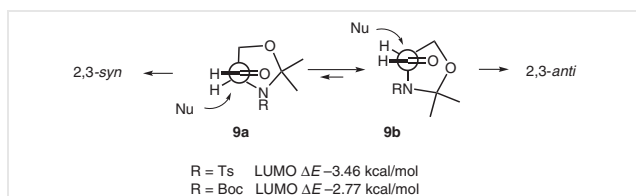
For our purpose, we had concerns about the *N*-Boc protecting group due to its potential for neighboring-group participation in our planned synthetic manipulations; we therefore initially desired an *N*-tosyl protected nitrogen on the oxazolidinone **9**. Although one could simply tosylate the known oxazolidinone **6** to give **9**, we considered initiating our synthesis with the acyclic silyl-protected *N*-tosyl-D-

Ser²⁰ or the *N*-tosyl equivalent of Garner's aldehyde.²¹ Vinyl Grignard additions to *N*-sulfonyl-protected acyclic amino acids are not usually selective. Literature reports suggest that additions to the aldehydes of TsNH-Ala²² and TsNH-Phe²³ give poor diastereoselectivities (2:3 *anti/syn* and 2:1 with the major isomer not identified, respectively). Given the poor selectivity of additions to acyclic amino aldehydes, we opted to pursue the use of a toluenesulfonamide derivative of Garner's aldehyde **8**. Surprisingly, no Grignard chemistry has been reported on this aldehyde. We found that vinylmagnesium bromide added cleanly to give a >95% yield²⁴ (Scheme 3) and was more selective than the *N*-Boc-protected Garner's aldehyde, giving the *anti*-allylic alcohol **10** with an 8.5:1 dr before chromatography. The use of LiCl as an additive in the vinylmagnesium bromide reaction did not alter the results. Although some trial runs using vinylmagnesium chloride directly did show >10:1 diastereoselectivity, these seemed highly dependent on the commercial source and age of the reagent. Conveniently, no rotamers are observed in the NMR spectra of the tosylamides, unlike the Boc-derivatives, making their interpretation more straightforward; moreover, TLC visualization and chromatographic detection is aided by the UV activity of the aromatic sulfonamide.



Scheme 3 Synthesis of β -Vser derivatives **12** and **14**; pNs = 4-O₂NC₆H₄SO₂.

The improved diastereoselectivity can be partially explained by examining the LUMO energies of the reactive Felkin-Anh conformations (Scheme 4). With the *N*-sulfonyl amide there is a strong preference for the C-NTs bond of **9b** to lie perpendicular to the plane defined by the aldehyde carbonyl as opposed to the C-CH₂O bond in **9a**. The LUMO of **9a** is 3.46 kcal mol⁻¹ higher in energy than that of **9b**, as determined by ground-state gas-phase DFT calculations using an ω -B97XD hybrid GGA functional. This predicts that nucleophilic approach should favor attack on **9b**, leading to the 2,3-*anti*-product. In contrast, the *N*-Boc derivative has a smaller LUMO energy difference (2.77 kcal mol⁻¹) between the two Felkin-Anh conformations, so it would not be expected to be as stereoselectively based on this analysis.



Scheme 4 Felkin-Anh depiction of nucleophilic attacks

The trend favoring the 2,3-*anti*-diastereomer is also observed for aryl and methyl Grignards, with >7:1 ratios being observed (Table 1). Interestingly, ethyl Grignard also afforded an 8:1 selectivity toward the *anti*-product, which is a near reversal of the *syn*-preference observed by Joullié and others.²⁵ The 2,3-*syn*-selectivity has been suggested to arise from chelation to the Boc carbonyl oxygen,²⁶ which might contribute to our observed *anti*-preference with the less chelation-prone tosylamide. Finally, the allyl Grignard gave poor selectivity in this reaction.

Table 1 Comparison of Grignard Additions to **8** and to Garner's Aldehyde

Entry	R	Pg = Ts <i>anti</i> / <i>syn</i> ^a	Yield ^b (%)	Pg = Boc <i>anti</i> / <i>syn</i>	Ref.
1	vinyl	8.5:1	95	3–6:1	7b,9,16
2	Ph	12:1	70	1.5–5:1	27,25b
3	4-MeOC ₆ H ₄	14:1	n.d. ^c	5:1 ^d	27
4	Me	7:1	93	2:1	25a
5	Et	8:1	87 ^e	1:9	25a
6	All	1:1.6	94	1.5:1	28

^a Determined by ¹H NMR integration on the crude sample or after hydrolysis to the diol.

^b The crude product contained 1–4% of starting aldehyde.

^c Not determined due to contamination by anisole. Hydrolysis gave the diol in 59% yield over two steps.

^d Aryllithium rather than Grignard.

^e *anti*-Configuration confirmed by comparison with hydrogenated **10**.

For most of the *N*-tosyl Grignard products, we observed significant decomposition to the diol or rearrangement to dioxolanes on silica gel chromatography, so for **10**, the crude product was always carried forward. Acidic hydrolysis of the *N,O*-acetal by using 4-toluenesulfonic acid in an ethanol/methanol mixture gave chromatographically pure diol **3**,²⁴ which could be selectively protected at the primary hydroxy group with *tert*-butyl(dimethyl)silyl chloride to supply **11** in 80% over three steps from **8**. Note that this silylation is much more easily achieved than that of the similar Boc-amine diol **2** derived from Garner's aldehyde, which tends to give disilylation products if great care is not taken.

To confirm our stereochemical assignment of the vinyl addition, the known oxazolidinone²⁹ **9** was formed in 75% yield from **11** by using triphosgene and pyridine. Unfortunately, the ¹H NMR spectrum reported in the literature was not sufficiently resolved to permit comparison of coupling constants, but, in general, the H-4 to H-5 coupling (oxazolidinone numbering) can be easily used to distinguish between the *cis*- and *trans*-diastereomers, with *cis* $J_{4,5} \approx 7$ Hz and the *trans* $J_{4,5} \approx 4$ Hz.³⁰ Oxazolidinone **9** has $J_{4,5}$ of 7.6 Hz, indicative of a *cis*-relationship. In addition, removal of the toluenesulfonyl protecting group could be accomplished in good yield (83%) by using Na/naphthalene in 1,2-dimethoxyethane, and the *cis*-coupling constant between H5 at $\delta = 5.04$ ppm and H4 at $\delta = 3.83$ ppm of oxazolidinone **6** was revealed to be 8.1 Hz, matching that reported by Ibuka,¹⁸ and thereby confirming our assignment of the *anti*-diastereomer **10** from the Grignard chemistry. Note that this synthetic route to **6** via *N*-tosyl serinal **8** is a significant improvement compared with previously reported Grignard chemistry.

In our case, we had no desire to remove the *N*-tosyl protection; instead, we sought to deprotect the primary hydroxy and to oxidize it to a carboxylic acid to form our β -Vser synthetic equivalent. Although there are reports of both steps being achieved in one pot with KF, Jones reagent, or similar compounds³¹ we found it better to do this in a stepwise manner by using HCl and MeOH to remove the silyl protection in 92% yield, and subsequent Jones oxidation to supply methyl ester **12** in 82% yield after diazomethane treatment. Unfortunately, attempts at oxidation with TEMPO-type reagents did not give a complete reaction, giving yields of around 50% in our hands.

Although we desired the *N*-tosyl protection, we recognize its versatility is limited for some cases, so we demonstrated that the final steps can also be carried out with a *p*-nosyl-protected nitrogen. From **6**, the *para*-nosyl group can be introduced using sodium hydride in THF to give **13** in 90% yield. Similar reactions have been reported to run in DMF and to give concomitant silyl ether cleavage,²⁹ but in our case a mixture was always observed. Therefore, we removed the silyl ether under acidic conditions and employed a Jones oxidation, as described earlier for **12**, to give **14** in similar yields.

In summary, an efficient synthesis of a β -vinyl serine (β -Vser) synthetic equivalent is reported that exploits the stereodirecting effect of the *N*-toluenesulfonamide group in a highly diastereoselective vinyl Grignard addition.

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Supporting Information

Supporting information for this article is available online at <https://doi.org/10.1055/a-1308-0370>.

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- (24) **N-[(1R,2S)-2-Hydroxy-1-(hydroxymethyl)but-3-en-1-yl]-4-methylbenzenesulfonamide (3)**
A 1.6 M solution of vinylmagnesium chloride in THF (18.9 mmol, 11.8 mL, 4 equiv) was added dropwise over 30 min to a solution of the aldehyde **8** (1.34 g, 4.74 mmol) in THF (44 mL) at -40 °C. The solution was then warmed to 0 °C and stirred overnight at rt. The solution was then poured into cold sat. aq NH₄Cl and extracted with EtOAc (×3). The combined organic extracts were washed with brine, dried (Na₂SO₄), filtered, and concentrated to give crude product **10** as a colorless viscous oil; yield: -1.5 g. ¹H NMR (400 MHz, CDCl₃): δ = 7.85–7.72 (m, 2 H), 7.39–7.29 (m, 2 H), 5.95–5.76 (m, 1 H), 5.49–5.23 (m, 2 H), 4.47 (td, J = 5.2, 2.4 Hz, 1 H), 4.01 (dd, J = 9.2, 4.2 Hz, 1 H), 3.91–3.65 (m, 2 H), 2.82 (d, J = 5.4 Hz, 1 H), 2.46 (s, 3 H), 1.72 (s, 3 H), 1.62–1.47 (m, 3 H)
To a solution of the crude vinyl alcohol **10** (~4.74 mmol) in MeOH (80 mL) and EtOH (80 mL) at r.t. was added TsOH·H₂O (180 mg 0.95 mmol, 2 equiv). The solution was stirred overnight then concentrated to half its original volume, diluted with EtOAc (400 mL), and washed with 2:1 sat. aq NaHCO₃-H₂O (100 mL). The aqueous phase was back-extracted with EtOAc, and the combined organic extracts were washed with brine (×2) and dried (Na₂SO₄). Flash column chromatography (silica gel, 50–75% EtOAc-hexanes) gave an off-white solid; yield: 1.00 g (80%); mp 75–76 °C; R_f = 0.20 (50% EtOAc-hexanes). ¹H NMR (400 MHz, CDCl₃): δ = 7.81 (d, J = 8.3 Hz, 2 H), 7.34 (d, J = 8.3 Hz, 2 H), 5.82 (ddd, J = 17.3, 10.6, 5.1 Hz, 1 H), 5.44 (d, J = 7.9 Hz, 1 H), 5.37 (bd, J = 17.2, Hz, 1 H), 5.37 (bd, J = 10.6 Hz, 1 H), 4.29 (m, 1 H), 3.86 (dt, J = 11.6, 3.6 Hz, 1 H), 3.51 (ddd, J = 11.5, 7.8, 3.8 Hz, 1 H), 3.28 (dt, J = 7.9, 3.7 Hz, 1 H), 2.54 (d, J = 10.6 Hz, 1 H), 2.46 (s, 3 H), 2.25 (dd, J = 7.8, 4.1 Hz, 1 H). ¹³C NMR (100 MHz, CDCl₃): δ = 143.8, 137.4, 136.4, 129.9, 127.1, 117.2, 74.8, 61.8, 57.3, 21.6. HRMS (ESI-TOF): m/z [M + Na]⁺ calcd for C₁₂H₁₇NNaO₄S: 294.0776; found: 294.0770.
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