ChemPubSoc Europe

DOI: 10.1002/chem.201605955

CHEMISTRY A European Journal Communication

Asymmetric Catalysis

Catalyst-Controlled Multicomponent Aziridination of Chiral Aldehydes

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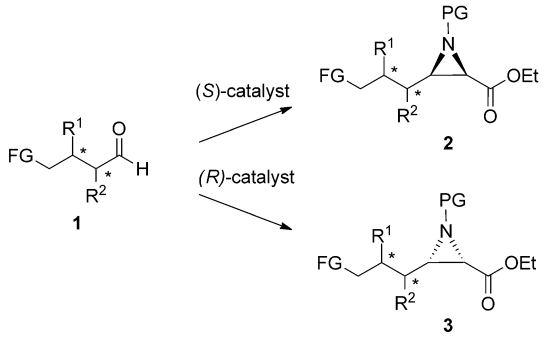
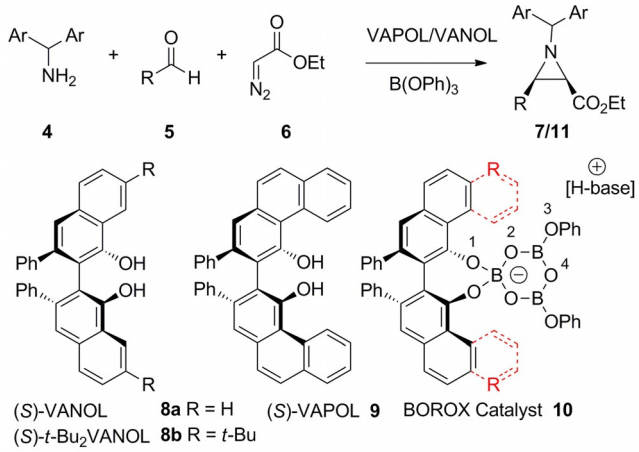
Abstract: A highly diastereoselective and enantioselective method for the multicomponent aziridination of chiral aldehydes has been developed with BOROX catalysts of the VANOL (3,3′-diphenyl-2,2′-bi-1-naphthol) and VAPOL (2,2′-diphenyl-(4-biphenanthrol)) ligands. Very high to perfect catalyst control is observed with most all substrates examined including aldehydes with chiral centers in the α- and β-positions. High catalyst control was also observed for a number of chiral heterocyclic aldehydes allowing for the preparation of epoxy aziridines, bis(aziridines) and ethylene diaziridines. Application of this reaction in the synthesis of β^3-homo-D-alloisoleucine and β^3-homo-L-isoleucine is reported.

The range of asymmetric catalytic tactical methods available to the practicing synthetic organic chemist today could have been scarcely imagined even twenty years ago.[1] Less common are methods that display true catalyst control in setting the stereochemistry of new chiral centers independent of chiral centers already present in the molecule. Typically, one finds matched and miss-matched pairs that allow for the selective formation of one of the possible diastereomers.[2] In this report we describe the first catalyst-controlled multicomponent asymmetric aziridination of aldehydes in which the absolute stereochemistry of the newly formed aziridine is a function of the catalyst and not of the chiral centers in the aldehyde present at either the α- or β-positions (Scheme 1).



Scheme 1. Catalyst-controlled multicomponent aziridination.



Scheme 2. Multicomponent aziridination by BOROX catalysis.

The first multicomponent catalytic asymmetric aziridination of achiral aldehydes was recently reported involving a diarylmethyl amine, an aldehyde, and ethyl diazoacetate to give *cis*-aziridine-2-carboxylates with excellent yields and asymmetric inductions (Scheme 2).[3] This reaction has been used in the synthesis of β-lactams,[4] sphinganines,[5] and β-amino esters.[6] The process is an overall five-component transformation that begins with an amine-induced assembly of the BOROX catalyst **10** from a molecule of either VANOL (3,3′-diphenyl-2,2′-bi-1-naphthol) or VAPOL (2,2′-diphenyl-(4-biphenanthrol)) and three equivalents of $B(OPh)_3$. This is followed by the conversion of the aldehyde and amine into a Schiff base and finally reaction with ethyl diazoacetate to give the aziridine **7**.[3b] The substrate catalyst complex **10** is an ion pair consisting of a chiral boroxinate anion and the substrate in the form of a protonated imine.[7] X-ray analysis reveals that the substrate and catalyst are held together by a number of noncovalent interactions including hydrogen-bonds, π–π stacking, and CH–π interactions.[7] The question that arises in addressing the goals of the present work is whether an imine generated from the chiral aldehyde **1** in Scheme 1 will have a differential binding

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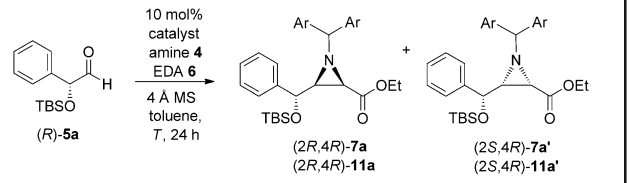
Supporting information and the ORCID identification number(s) for the author(s) of this article can be found under http://dx.doi.org/10.1002/chem.201605955.

between the (*R*)- and (*S*)-BOROX catalysts in the transition state[8] for aziridine formation that would prohibit the realization of the idealized situation for a catalyst-controlled aziridination.

The multicomponent aziridination of the α-silyloxyphenylacetaldehyde (*R*)-**5a** with amine **4a** and ethyl diazoacetate **6** gave a 96:4 selectivity for the *anti*-aziridine **7a** over the *syn*-aziridine **7a′** with the catalyst derived from (*S*)-VAPOL (Table 1, entry 1, the prime in **7a′** indicates that it is the favored isomer with the (*R*)-ligand). This reaction reveals that there is a very high level of catalyst control, since the aziridination with the (*R*)-VAPOL catalyst gives a 97:3 ratio of aziridines in favor of the *syn*-isomer of **7a′** (entry 2). The aziridination with non-chiral aldehydes with a catalyst derived from the (*S*)-ligand gives addition to the *Si*-face of the imine and thus to an aziridine carboxylate with a (2*R*)-configuration.[3] The selectivities can be improved slightly by lowering the temperature to −10 °C (entries 5 and 6). The catalysts generated from the (*R*)- and (*S*)-VANOL ligands are comparable to those of VAPOL and give complete catalyst control at −10 °C (entries 7 and 8). The catalyst control is not quite as high with benzhydryl amine **4b** (entries 9 and 10) and thus the investigation was moved forward with the MEDAM (3,5-dimethyldianisylmethylamine) **4a**. The absolute and relative stereochemistry of the aziridines **7a** was determined by an X-ray diffraction analysis on the (2*S*,4*R*)-isomer **7a′** (see Supporting Information).

Table 1. Catalyst-controlled aziridination of (*R*)-2-silyloxy-2-phenylacetaldehyde **5a**.[a]



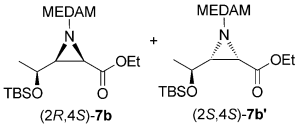
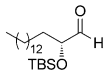
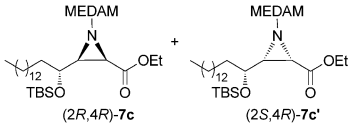
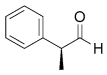
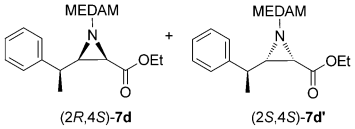
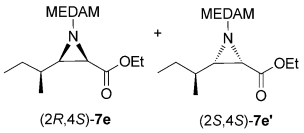
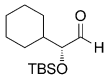
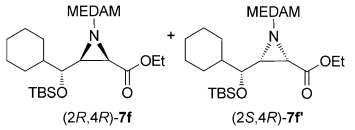
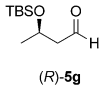
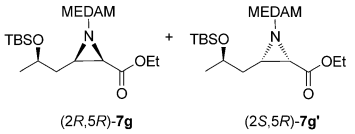
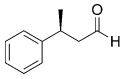
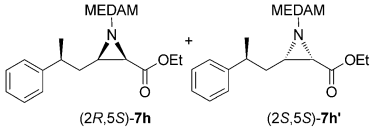
Entry	Amine **4**	Ligand	*T* [°C]	Total yield[b] [%]	Azir.	(2*R*)/(2*S*)[c]
1		(*S*)-VAPOL	25	90	**7a**	96:4
2		(*R*)-VAPOL	25	90	**7a**	3:97
3		(*S*)-VAPOL	0	92	**7a**	97:3
4		(*R*)-VAPOL	0	90	**7a**	1:99
5	MEDAMNH$_2$	(*S*)-VAPOL	−10	90	**7a**	98:2
6	**4a**	(*R*)-VAPOL	−10	90	**7a**	1:99
7		(*S*)-VANOL	−10	85	**7a**	98:2
8		(*R*)-VANOL	−10	85	**7a**	2:98
9		(*S*)-VAPOL	−10	50	**11a**	>99:1
10	**4b**	(*R*)-VAPOL	−10	65	**11a**	6:94

[a] Unless otherwise specified, all reactions were performed with amine **4** (0.2 mmol, 1.0 equiv), with EDA **6** (1.2 equiv), and aldehyde (*R*)-**5a** (1.05 equiv) at 0.4 M in toluene for 24 h at the indicated temperature in the presence of powdered 4 Å molecular sieves, and reached 100% completion. The BOROX catalyst was assembled by heating a solution of **4** (100 mol%), ligand (10 mol%), and B(OPh)$_3$ (30 mol%) in toluene at 80 °C for 0.5 h. After cooling to the indicated temperature, (*R*)-**5a** and 4 Å molecular sieves were added, followed directly by **6**. [b] Yield of (2*R*,4*R*)-**7a**/**11a** and (2*S*,4*R*)-**7a′**/**11a′** isolated together after silica gel chromatography. [c] Determined from the ^{1}H NMR spectrum of the crude reaction mixture.

The results from a number of different aldehydes with chiral centers at the α- and β-carbon atoms are collected in Table 2. Replacement of the phenyl group in aldehyde **5a** with a methyl group still results in very good catalyst control for the aldehyde (*S*)-**5b** (entries 1 and 2). However, replacement of the *tert*-butyldimethylsilyloxy (TBS) group in aldehyde **5a** with a methyl group results in a drop of the selectivity for aldehyde (*S*)-**5d** to a 22:78 ratio of isomers in the miss-matched case. This turns out not to be a simple reflection of the selection difference between an OTBS and a methyl group at the α-chiral center, but rather that the intermediate imine is undergoing racemization. In addition, the reaction of (*S*)-**5d** is slightly slower with (*R*)-VAPOL catalyst than with the (*S*)-VAPOL catalyst and as a result racemization proceeds to a slightly higher degree with the former. The reaction of the imine derived from aldehyde (*S*)-**5d** with the (*R*)-VAPOL catalyst gives a 22:78 mixture of (2*R*,4*S*)-**7d** and (2*S*,4*S*)-**7d′**, in which the *ee* of the former is 43% and that of the latter is 99.9%. The reaction of the imine derived from aldehyde (*S*)-**5d** with the (*S*)-VAPOL catalyst gives a 96:4 mixture of (2*R*,4*S*)-**7d** and (2*S*,4*S*)-**7d′**, in which the *ee* of the former is 99% and that of the latter is 80%. Correcting for racemization, the ratio of (2*R*,4*S*)-**7d** to (2*S*,4*S*)-**7d′** should be 94:6 with the (*S*)-VAPOL catalyst and 12:88 with the (*R*)-VAPOL catalyst. The degree of racemization was found to be somewhat greater at higher concentration (see Supporting Information). This is the only chiral aldehyde for which we found that racemization was a problem.

The chiral aldehyde (*S*)-**5e** with an α-branched aliphatic chain did not give a high degree of catalyst control with the VAPOL ligand. However, a higher degree of catalyst control was realized with a catalyst with the *t*Bu$_2$VANOL ligand (entries 10 and 11) at −10 °C and perfect catalyst control at −40 °C (entries 12 and 13). It was demonstrated for the aldehyde (*S*)-**5e** (4.8 mmol) that this reaction could be carried out on gram scale, giving 1.72 g of aziridines **7e** in 95% yield and 96:4 diastereoselectivity (Table 2, entry 12). The only substrate for which we found a strongly miss-matched reaction was with the aldehyde (*R*)-**5f**, in which a *tert*-butylsilyloxy group is pitted against a cyclohexyl group. For this aldehyde both the (*S*)-VAPOL and (*S*)-VANOL catalysts were miss-matched with very slow reactions that did not go to completion giving both low yields and selectivities (entries 14 and 17). The corresponding (*R*)-ligands, on the other hand, gave very high yields and exclusively a single stereoisomer of the aziridine **7f′** (entries 15 and 18) and no drop-off in performance of the (*R*)-VAPOL catalyst was observed when the catalyst loading was decreased from 10 to 5 mol% (entry 16). Greatly improved catalyst control in the aziridination of aldehyde (*R*)-**5f** was realized with a catalyst derived from the 7,7′-di-*tert*-butyl VANOL ligand **8b** (Scheme 2). Here the selectivity in the miss-matched case could be increased to 82:18 and the yield increased from 15 to 75% (entries 17 vs. 19). The reaction of the non-chiral catalyst Yb(OTf)$_3$ (10 mol%) was examined with the pre-formed imine of aldehyde (*R*)-**5f** at room temperature and gave **7f** and **7f′** in a 1:10 ratio, which is to be compared to 1:18 and 2:1 ratios

Table 2. Catalyst-controlled aziridination of α- and β-chiral aldehydes.[a]

Entry	Substrate	Cat. [mol%]	Ligand	Products	Total yield[b] [%]	(2*R*)/(2*S*)
1	 (*S*)-**5b**	10	(*S*)-VAPOL	 (2*R*,4*S*)-**7b** (2*S*,4*S*)-**7b'**	85	91:9
2		10	(*R*)-VAPOL		87	4:96
3		10	(*R*)-VANOL		82	5:95
4	 (*R*)-**5c**	10	(*S*)-VAPOL	 (2*R*,4*R*)-**7c** (2*S*,4*R*)-**7c'**	88	90:10
5		10	(*R*)-VAPOL		94	<1:99
6[d,e]	 (*S*)-**5d**	10	(*S*)-VAPOL	 (2*R*,4*S*)-**7d** (2*S*,4*S*)-**7d'**	92[f]	96:4
7[d,e]		10	(*R*)-VAPOL		90[g]	17:83
8[h]	 (*S*)-**5e**	5	(*S*)-VAPOL	 (2*R*,4*S*)-**7e** (2*S*,4*S*)-**7e'**	60	75:25
9[h]		5	(*R*)-VAPOL		50	33:67
10[d]		5	(*S*)-*t*Bu$_2$VANOL		>99	92:8
11[d]		5	(*R*)-*t*Bu$_2$VANOL		80	17:83
12[d,i]		10	(*S*)-*t*Bu$_2$VANOL		95	96:4
13[d,i,j]		10	(*R*)-*t*Bu$_2$VANOL		90	4:96
14	 (*R*)-**5f**	10	(*S*)-VAPOL	 (2*R*,4*R*)-**7f** (2*S*,4*R*)-**7f'**	30[k]	52:48
15		10	(*R*)-VAPOL		85	<1:>99
16		5	(*R*)-VAPOL		83	<1:>99
17		10	(*S*)-VANOL		15[k]	60:40
18		10	(*R*)-VANOL		80	<1:>99
19		5	(*S*)-*t*Bu$_2$VANOL		75	82:18
20		5	(*R*)-*t*Bu$_2$VANOL		93	<1:>99
21	 (*R*)-**5g**	10	(*S*)-VAPOL	 (2*R*,5*R*)-**7g** (2*S*,5*R*)-**7g'**	83	98:2
22		10	(*R*)-VAPOL		85	<1:>99
23	 (*S*)-**5h**	10	(*S*)-VAPOL	 (2*R*,5*S*)-**7h** (2*S*,5*S*)-**7h'**	80	94:6
24		10	(*R*)-VAPOL		85	<1:>99

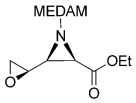
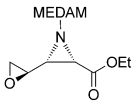
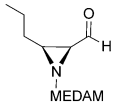
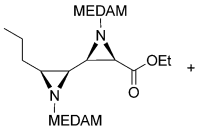
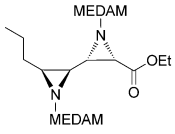
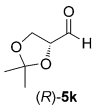
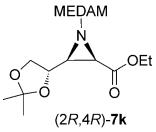
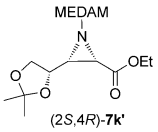
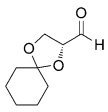
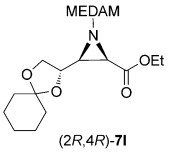
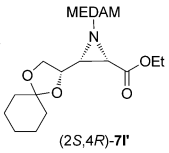
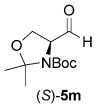
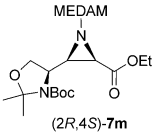
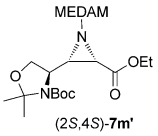
[a] Unless otherwise specified, all reactions were performed as described in Table 1 at −10 °C with 10 mol% catalyst. The concentration was 0.4 M in amine except for entries 4 and 5, which were 0.2 M. [b] Yield of **7**-(2*R*) and **7**′-(2*S*) isolated together after silica gel chromatography. [c] Determined from the ^{1}H NMR spectrum of the crude reaction mixture. [d] 4 equiv EDA. [e] Reaction at 0.04 M instead of 0.4 M. [f] The *ee* of **7 d**-(2*R*,4*S*) is 99% and that of **7 d**′-(2*S*,4*S*) is 80% from (*S*)-VAPOL. [g] The *ee* of **7 d**-(2*R*,4*S*) is 43% and that of **7 d**′-(2*S*,4*S*) is 99.9% from (*R*)-VAPOL. [h] 2 equiv EDA. [i] Reaction at −40 °C. [j] Reaction for 48 instead of 24 h. [k] Reactions did not reach completion.

with the BOROX catalysts of (*R*)-VAPOL and (*S*)-VAPOL, respectively (see Supporting Information).

High catalyst control was realized with aldehydes (*R*)-**5 g** and (*S*)-**5 h**, each bearing a chiral center at the β-carbon atom. In comparing aldehydes (*R*)-**5 g** with (*S*)-**5 b**, one would expect that the level of catalyst control would be higher the greater the distance of the chiral center from the reaction center and this indeed was found to be the case. As a control, it should be noted that the enantioselectivity for the multicomponent aziridination of a non-chiral unbranched aliphatic aldehyde is 95% *ee* for the VANOL BOROX catalyst and 98% *ee* for the 7,7′-di-*tert*-butyl VANOL BOROX catalyst at room temperature.[5] In related chemistry, the VANOL BOROX catalyst can also give high catalyst control in the amino allylation of aldehydes bearing a β-chiral center, but not an α-chiral center.[9]

Several chiral heterocyclic aldehydes were examined as well and most of the reactions indicated in Table 3 were performed with 5 mol% catalyst. The multicomponent aziridination of (*S*)-epoxypropanal **5 i** exhibited very high yields and essentially complete levels of catalyst control for the VANOL ligand, since essentially the same selectivity was observed for each enantiomer of the ligand. The determinant in the selectivity between

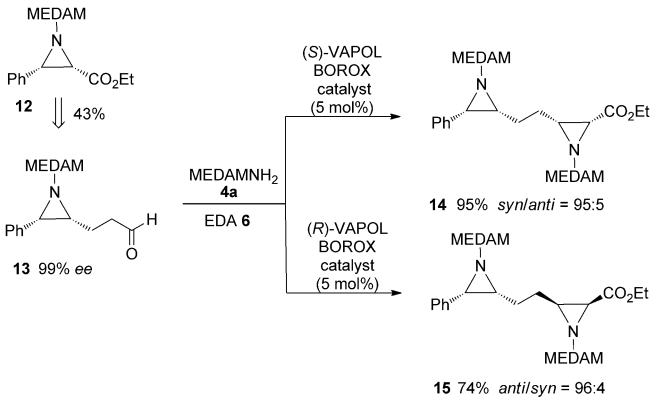
Table 3. Catalyst-controlled aziridination of chiral heterocyclic carboxaldehydes.[a]

Entry	Substrate	Ligand	Products	Total yield[b] [%]	(2*R*)/(2*S*)
1	 (*S*)-**5i**	(*S*)-VANOL	 (2*R*,4*S*)-**7i** +  (2*S*,4*S*)-**7i'**	99[d]	13:87
2		(*R*)-VANOL		>99[d]	88:12
3		(*S*)-*t*Bu$_2$VANOL		96[d]	8:92
4		(*R*)-*t*Bu$_2$VANOL		>99[d]	89:11
5	 (2*S*,3*S*)-**5j**	(*S*)-VAPOL	 (2*R*,4*S*)-**7j** +  (2*S*,4*S*)-**7j'**	84[e]	>99:<1
6		(*R*)-VAPOL		80[e]	<1:>99
7	 (*R*)-**5k**	(*S*)-VAPOL	 (2*R*,4*R*)-**7k** +  (2*S*,4*R*)-**7k'**	73	84:16
8		(*R*)-VAPOL		90	6:94
9		(*S*)-VANOL		67	84:16
10		(*R*)-VANOL		86	8:92
11		(*S*)-*t*Bu$_2$VANOL		99	97:3
12		(*R*)-*t*Bu$_2$VANOL		99	3:97
13	 (*R*)-**5l**	(*S*)-VANOL	 (2*R*,4*R*)-**7l** +  (2*S*,4*R*)-**7l'**	>99[d]	91:9
14		(*R*)-VANOL		>99[d]	3:97
15		(*S*)-*t*Bu$_2$VANOL		>99[d]	97:3
16		(*R*)-*t*Bu$_2$VANOL		>99[d]	2:98
17	 (*S*)-**5m**	(*S*)-VAPOL	 (2*R*,4*S*)-**7m** +  (2*S*,4*S*)-**7m'**	70[e]	>99:<1
18		(*R*)-VAPOL		60[e]	1:99

[a] Unless otherwise specified, all reactions were performed as described in Table 1 at −10 °C with 5 mol% catalyst. [b] Yield of **7**-(2*R*) and **7**′-(2*S*) isolated together after silica gel chromatography. [c] Determined from the ^{1}H NMR spectrum of the crude reaction mixture. [d] 2 equiv EDA instead of 1.2. [e] 10 mol% catalyst.

the diastereomers of the α,β,γ,δ-epoxy aziridine **7i** is the chirality of the ligand and not that of the epoxide. A slight improvement in the selectivity level was observed for the *t*Bu$_2$VANOL ligand **8b** (Scheme 2), but here the slightest hint of a matched/miss-matched pair was seen. Similar high levels of stereocontrol were found in the reactions of the aziridines **5j** giving the *syn*- and *anti-bis*-aziridines **7j** each as a single diastereomer and enantiomer. The aziridination of (*S*)-glyceraldehyde acetonide **5k** occurs with good catalyst control for both VANOL and VAPOL catalysts; in the miss-matched case with the (*S*)-ligands the selectivity was usable (84:16), but certainly less than ideal. As with the reactions of the cyclohexyl-substituted aldehyde (*R*)-**5f**, the selectivity could be improved with the use of a catalyst derived from the 7,7′-di-*tert*-butylVANOL ligand **8b**, and here for aldehyde (*S*)-**5k** the improvement is to the level of perfect catalyst control (equal selectivities seen with both the (*R*)- and (*S*)-**8b**). Slightly improved selectivities were observed for the cyclohexanone-protected (*S*)-glyceraldehyde **5l** with the VANOL ligand. Perfect catalyst control and perfect selectivities were observed with Garner's aldehyde (*S*)-**5m** with the VAPOL ligand.

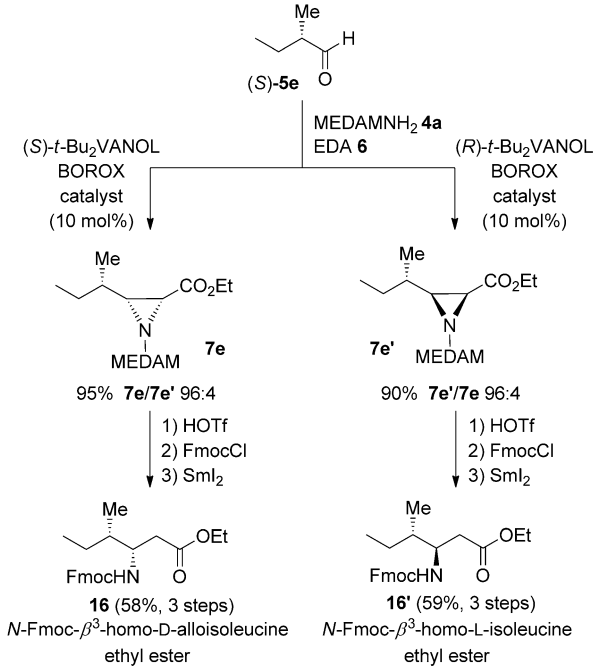
The multicomponent aziridination of the γ,δ-aziridinyl aldehyde **13** was examined with both (*R*)- and (*S*)-VAPOL catalysts (Scheme 3). Aziridine **13** was prepared in 43% yield from the aziridine **12**, which we have previously reported, can be prepared in 98% yield and 99.8% *ee* with the (*R*)-VAPOL-BOROX catalyst.[10] The reaction of **13** with MEDAM amine **4a** and ethyl diazoacetate **6** with 5 mol% (*S*)-VAPOL catalyst gave the ethylene diaziridine **14** as a 95:5 mixture of *syn*- and *anti*-diastereomers in 95% yield. The *anti*-diastereomer **15** is preferen-



Scheme 3. Catalyst-controlled synthesis of ethylene diaziridines.

tially generated with the catalyst prepared from (*R*)-VAPOL in 74% yield with a 96:4 selectivity. This is not quite as high a selectivity as observed with the α,β-aziridinyl aldehyde **5j** (Table 3, entries 5 and 6), which suggests that the steric bulk in the near vicinity is important to selectivity.

In the last twenty years or more there has been significant interest in the synthesis of β-amino acids as a consequence of the fact that they are needed in the synthesis of β-peptides and β-lactams.[11,12] As an illustration of the synthetic utility of the catalyst controlled asymmetric aziridination of chiral aldehydes, we have carried out a concise synthesis of protected forms of β^3-homo-D-alloisoleucine and β^3-homo-L-isoleucine (Scheme 4). The aldehyde (*S*)-**5e** can be readily prepared from



Scheme 4. A concise route to β^3-homo-L-isoleucine and β^3-homo-D-alloisoleucine.

the commercially available (*S*)-2-methyl-1-butanol (see Supporting Information). The aziridination of (*S*)-**5e** with amine **4a** and ethyl diazoacetate **6** occurs with the BOROX catalyst prepared from *t*Bu$_2$VANOL **8b** with complete catalyst control giving a 96:4 selectivity with each isomer of the catalyst (see Table 2). The key reductive ring-opening step was effected with samarium(II) diiodide using a protocol that we have recently reported.[6] For *cis*-aziridines, it was found that the N-substituent needs to be activating or else both C–C and C–N cleavage occurs. Thus the MEDAM group was removed and replaced by Fmoc subsequent to reduction, which gave the β-amino esters **15** and **16** in good yields and as single diastereomers and enantiomers.

In conclusion, we have found that BOROX catalysts are highly effecting in dominating the stereochemical outcome of multicomponent aziridination of aldehydes that have chiral centers either in the α- or β-position to the aldehyde carbon. BOROX catalysts derived the VANOL and VAPOL ligands are equally effective, but for the toughest substrates the 7,7′-di-*tert*-butylVANOL BOROX catalyst is the best. The methodology developed here will allow the synthesis of complex organic scaffolds with more than two chiral centers in which a definitive stereochemical relationship is desired.

Acknowledgements

This work was supported by a grant from the National Institute of General Medical Sciences (GM094478).

Keywords: asymmetric catalysis · aziridines · borox catalyst · catalyst control · vapol

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Manuscript received: December 21, 2016
Accepted Article published: December 22, 2016
Final Article published: January 27, 2017