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Addition of Azomethine Ylides to Aldehydes: Mechanistic Dichotomy of Differentially Substituted α-Imino Esters

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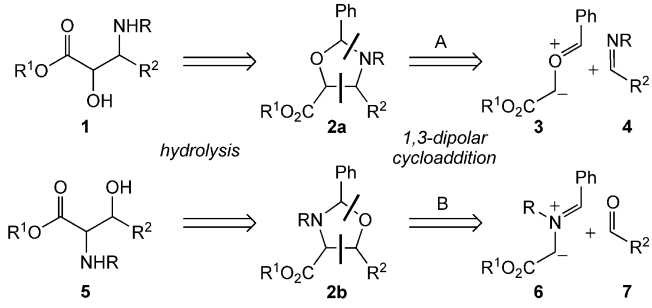
The formal 1,3-dipolar cycloaddition of azomethine ylides and aldehydes is explored, as hydrolysis of the resulting oxazolidine product gives facile access to valuable *syn*-β-aryl-β-hydroxy-α-amino esters. The use of using benzaldehyde-derived imines as the ylide precursor results in 1,3-dipolar cycloaddition with high conversions but low diastereoselectivity. In contrast, the employment of benzophenone-derived imines as the ylide precursor results in an aldol reaction, which gives the intermediate oxazolidine in high diastereoselectivity and requires a weak acid catalyst to achieve higher conversions.

Introduction

Due to their prevalence in natural products, including antibiotics and enzyme inhibitors, and their value as chiral building blocks, β-hydroxy-α-amino acids have become increasingly important synthetic targets.[1] Not only is this structural motif present in vancomycin, sphingofungin, and lactacystin to name but a few natural products, but this scaffold can also be rapidly converted into 2-amino-1,3-diols, β-lactams,[2] aziridines,[3] and β-haloamino acids.[4–6] Thus, numerous methods have been developed to access these compounds.[1]

One of the most efficient strategies to construct these valuable building blocks is the direct addition of a glycine equivalent to an aldehyde. Recently, we disclosed a technology to generate regioisomeric α-hydroxy-β-amino esters **1** from hydrolysis of oxazolidine **2a**, which is the product of a 1,3-dipolar cycloaddition of carbonyl ylide **3** and aldimine **4** (Scheme 1, pathway A).[7,8] Consequently, we envisaged that an analogous retrosynthetic disconnection of oxazolidine **2b** would allow the synthesis of β-hydroxy-α-amino esters through the reaction of azomethine ylide **6**, serving as a glycine equivalent, and aldehyde **7** (Scheme 1, pathway B).[9–15] Interestingly, in the course of this study we uncovered a mechanistic dichotomy between benzophenone-derived imines and benzaldehyde-derived imines. The former undergo aldol reactions with aryl aldehydes, whereas the latter participate in 1,3-dipolar cycloadditions.



Scheme 1. Retrosynthetic analysis of α-hydroxy-β-amino esters and β-hydroxy-α-amino esters.

Results and Discussion

Azomethine ylides have been studied extensively, as they offer an effective method for the construction of nitrogen-containing heterocycles.[16] We began our investigations by using Vedejs' oxazole methodology for mild, low-temperature azomethine ylide generation (Scheme 2).[17,18] Reaction of oxazolium salt **8** with benzaldehyde **7a** under reducing conditions yielded oxazolidine **11a**[19] in 58% yield via ylide **10**. The resulting product was hydrolyzed to β-hydroxy-α-amino ester **12a** as a 2.3:1 mixture of *syn*/*anti* diastereomers in 89% yield.[20]

Pleased with these initial results, we attempted to optimize the reaction, both to improve the diastereoselectivity and the yield (Table 1). A major byproduct was tertiary amine **13**, resulting from reduction of intermediate **10** (Scheme 2). Thus, we tried to alter the solvent (Table 1, Entry 3), reduce the equivalents of $PhSiH_3$ (Table 1, Entry 4), reduce the temperature (Entry 5), and decrease the rate of addition of the reducing agent (Table 1, Entry 5). However, we were unable to preclude the formation of **13** and little improvement of the yield was achieved.

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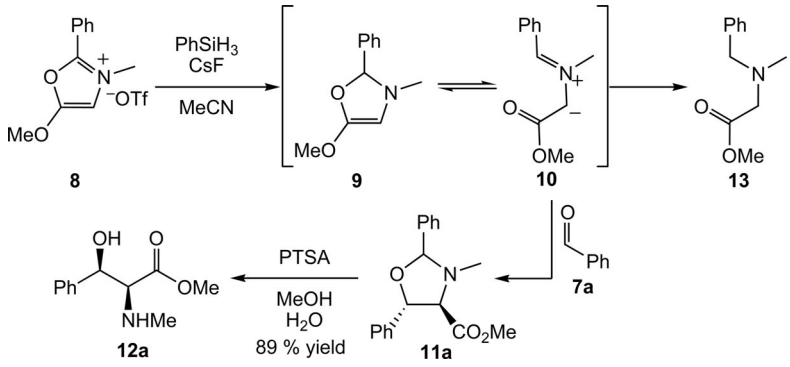
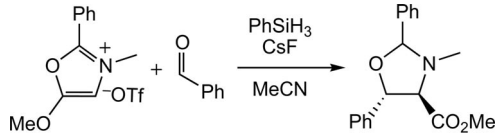


Scheme 2. Azomethine ylide generation from *N*-methyl oxazolium salt **8**, reaction with benzaldehyde (**7a**), and subsequent hydrolysis of oxazolidine **11a**.

Table 1. Optimization of 1,3-dipolar cycloaddition of oxazolium salt **8** and benzaldehyde (**7a**).[a]

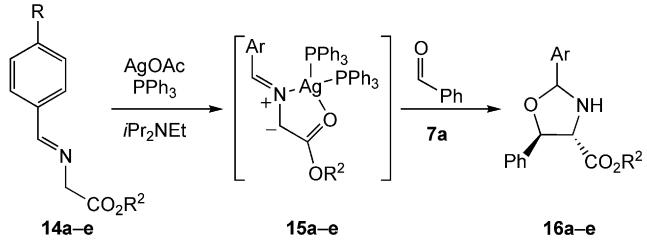


Entry	$PhSiH_3$ [equiv.]	Conditions	*dr*[b]	Yield[c] [%]
1	1.5	In situ formation of **8**	2.3:1	58
2	1.5		2.3:1	60
3	1.5	CH_2Cl_2	n.d.	7
4	1.05		n.d.	53[d]
5	1.05	–40 °C	n.d.	36
6	1.05	$PhSiH_3$ added over 10 h	n.d.	63[d]

[a] The reaction was carried out overnight with 1 equiv. of preformed oxazolium salt **8**. [b] Diastereoselectivity determined after hydrolysis to amino alcohol **12** with PTSA and MeOH/H_2O (95:5). n.d. = not determined. [c] Isolated yield of all diastereomers of **11**. [d] Conversion of the reaction to the product based on integration of the signals in the ^1H NMR spectrum.

To circumvent over-reduction of azomethine ylide **10** to amine **13**, we reconsidered our strategy. An attractive route to the desired species is the generation of a *N*-metallated azomethine ylide.[21–23] A Ag^I/PPh_3 catalyst has been reported to activate α-imino ester **14**, which can lead to metalloazomethine ylide **15** in the presence of a mild base.[24–26] We postulated that this intermediate could then be intercepted by benzaldehyde to produce oxazolidine **16**. To our delight, treatment of glycine-derived imine **14a** and benzaldehyde with AgOAc/PPh_3 catalyst (5 mol-%) in the presence of catalytic amounts of *i*Pr_2NEt yielded desired oxazolidine **16a** in 56% conversion and a 1.6:1 mixture of 4,5-*trans* to 4,5-*cis* diastereomers (Table 2, Entry 1).[27] To improve the conversion, a variety of imine derivatives were examined. The more electron-withdrawing 4-fluoro-substituted imine improved the yield and diastereoselectivity slightly (Table 2, Entry 2), as did increasing the catalyst loading to 10 mol-% (Table 2, Entry 3). We explored two other electron-deficient imines (Table 2, Entries 4 and 5) and determined that the best yields and diastereoselectivities were achieved with imines **14d** and **14e** (Table 2, Entries 5 and 8). Unfortunately, neither cooling the reaction nor changing the solvent enhanced the diastereoselectivity (Table 2, Entries 6–9).

Table 2. Optimization of the 1,3-dipolar cycloaddition of α-imino ester derived azomethine ylide with benzaldehyde.[a]

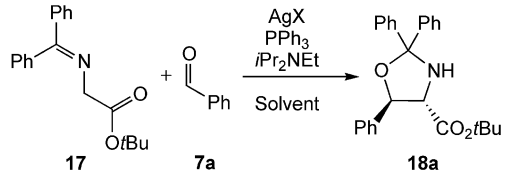


Entry	**14**	R, R^2	Solvent	Conv.[b] [%]	**16**	*dr* **16**[c]
1	**a**	H, Me	THF	56	**a**	1.6:1
2	**b**	F, Et	THF	63	**b**	1.7:1
3	**b**	F, Et	THF	82	**b**	1.7:1
4	**c**	CN, Et	THF	90	**c**	2.3:1
5	**d**	CF_3, Et	THF	96[e]	**d**	2.3:1
6[d]	**c**	CN, Et	THF	80	**c**	1.8:1
7	**d**	CF_3, Et	DCM	93	**d**	2.0:1
8	**e**	CF_3,Me	THF	84[e]	**e**	2.5:1
9	**e**	CF_3, Me	PhMe	78[e]	**e**	2.0:1

[a] Reaction conditions: imine **14** (1.0 equiv.), aldehyde **7a** (2.0 equiv.), *i*Pr_2NEt (0.2 equiv.), AgOAc (10 mol-%), and PPh_3 (20 mol-%) in solvent (c = 0.1 M) at room temperature. In Entries 1 and 2, 5 mol-% of AgOAc and 10 mol-% of PPh_3 were used. [b] Conversion of **14** determined by analysis of the ^1H NMR spectrum of the crude mixture before hydrolysis. [c] The 4,5-*trans* to 4,5-*cis* ratio of oxazolidine **16** was determined by hydrolysis to the corresponding amino alcohol, and analysis of the ^1H NMR spectrum of this mixture yielded the *dr*. Hydrolysis was performed with HCl (1 M) in MeOH/H_2O (95:5). [d] Reaction run at –20 °C. [e] 1-Methyl-naphthalene was used as an internal standard.

To improve the outcome of the reaction, benzophenone-derived imine **17** was examined. This reagent has previously been used in several direct aldol reactions under phase-transfer conditions to access *anti*-β-alkyl-β-hydroxy-α-amino esters.[28–32] Oxazolidine **18a** was obtained as a 3.8:1 mixture of 4,5-*trans* to 4,5-*cis* diastereomers (Table 3, Entry 1).[32,33] A further screen revealed toluene to be the opti-

Table 3. Reaction of benzophenone-derived imines with benzaldehyde.[a]



17 7a 18a

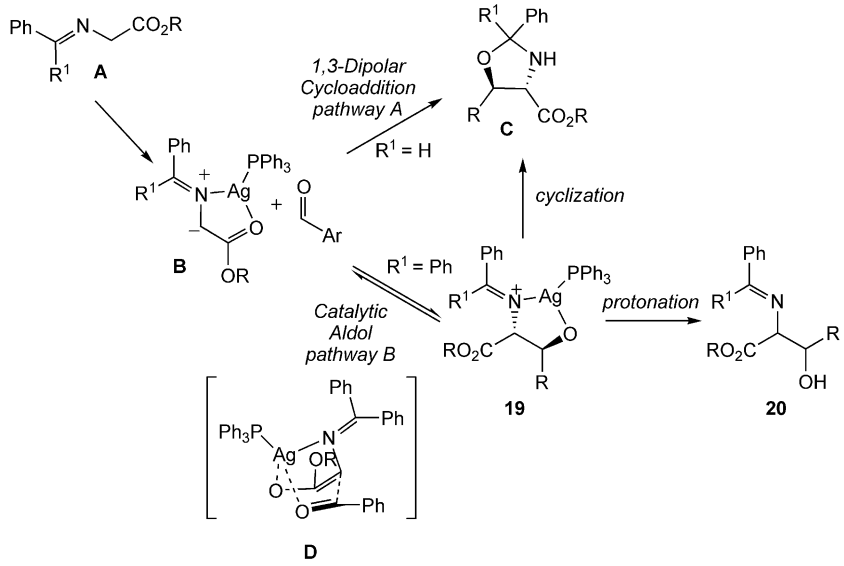
Entry	AgX	PPh_3 [mol-%]	Solvent	DIEA [mol-%]	Acid[b] [mol-%]	Conv. **17**[c] [%]	*dr*[d] **18**
1	AgOAc	20	THF	20	–	64	3.8:1
2	AgOAc	20	DCM	20	–	48	7.3:1
3	AgOAc	20	PhMe	20	–	61	10:1
4	AgOAc	10	PhMe	20	–	75[e]	30:1
5	AgOTf	10	PhMe	20	–	78[f]	36:1
6	$Ag(MeCN)_4BF_4$	10	PhMe	20	–	80	24:1
7	AgOTf	10	PhMe	20	–	30	9:1
8	AgOTf	10	PhMe	10	10	89	17:1
9	AgOTf	10	PhMe	10	5	72	36:1
10	AgOTf	10	PhMe	20	5	83	58:1
11	AgOTf	10	PhMe	–	10	–	–

[a] Reaction conditions: imine **17** (1.0 equiv.), aldehyde **7a** (2.0 equiv.), iPr_2NEt (20 mol-%), PPh_3, and AgX (10 mol-%) in solvent (c = 0.1 M) at room temperature. [b] Benzoic acid was used. In these cases the benzaldehyde was distilled. [c] Determined by analysis of the ^1H NMR spectrum of the crude mixture. [d] The 4,5-*trans* to 4,5-*cis* ratio of the oxazolidine was determined by analysis of the ^1H NMR spectrum of the crude reaction mixture. [e] The *syn* diastereomer of the amino alcohol was isolated in 70% yield after hydrolysis. [f] The *syn* diastereomer of the amino alcohol was isolated in 72% yield after hydrolysis.

mal solvent, significantly increasing the diastereoselectivity; although this occurred at the price of the conversion (Table 3, Entries 2 and 3). Interestingly, decreasing the amount of PPh_3 as compared to Ag^I increased the yield and diastereoselectivity (Table 3, Entries 3–5). Examining Ag^I salts, AgOTf was revealed to induce the highest yields and diastereoselectivities (Table 3, Entries 3–6).[34]

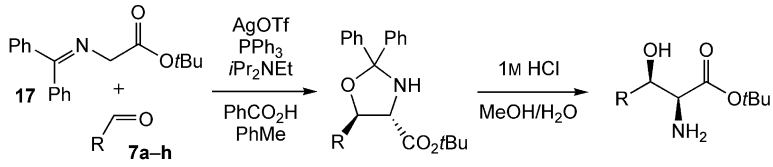
At this point we encountered an interesting setback; in our use of freshly distilled benzaldehyde, both the yield and diastereoselectivity were drastically decreased (Table 3, Entry 7).[35] Knowing the major impurity in the benzaldehyde to be benzoic acid, we tried using a 1:1 mixture (10 mol-% each) of *N*,*N*-diisopropylethylammonium benzoate, which resulted in an increased yield and diastereoselectivity (Table 3, Entry 8). Decreasing the amount of benzoic acid increased the diastereoselectivity (Table 3, Entry 9). The optimal combination proved to be 20 mol-% of iPr_2NEt and 5 mol-% benzoic acid (Table 3, Entry 10).[36]

Intrigued by the increased yield and diastereoselectivity in the presence of a proton source, we probed the mechanism of the reaction. Two potential reaction manifolds from the α-imino ester to the corresponding oxazolidine can be envisioned (Scheme 3). In the first, a straightforward 1,3-dipolar cycloaddition of metalloazomethine ylide intermediate **B** to aldehyde would lead directly to oxazolidine product **C** (pathway A). Alternatively, ylide **B** could participate



Scheme 3. Proposed mechanisms for the reaction of azomethine ylides with aryl aldehydes.

Table 4. Scope of silver-catalyzed direct aldol.[a]



	7/18/21	R	Conv.[b] [%]	*dr* **18** (*trans*/*cis*)[b]	Yield[c] [%]	*dr* **21** (*syn*/*anti*)[d]
1	**a**	Ph	89	58:1	68 (79)[e]	18:1
2	**b**	4-FC_6H_4	89	26:1	62 (77)[e]	16:1
3	**c**	4-BrC_6H_4	83	18:1	52	3:1
4	**d**	4-$MeOC_6H_4$	<10	–	–	–
5	**e**	3-BrC_6H_4	95	18:1	48	11:1
6	**f**	3-$MeOC_6H_4$	78	46:1	76	13:1
7	**g**	2-MeC_6H_4	51	36:1	49	3:1
8	**h**	furyl	94	1.5:1	91[f]	1.3:1

[a] Reaction conditions: imine **17** (1.0 equiv.), aldehyde **7** (2.0 equiv.), *i*Pr_2NEt (20 mol-%), benzoic acid (5 mol-%), and AgOTf/PPh_3 (1:1) in THF (c = 0.1 M) at room temperature. [b] Determined by analysis of the ^{1}H NMR spectrum of the crude reaction mixture before hydrolysis. [c] Isolated yield of *syn* diastereomer. [d] Determined by analysis of the ^{1}H NMR spectrum of the crude mixture after hydrolysis. [e] Yield in parenthesis is the yield of both diastereomers. [f] Isolated as a 1.3:1 mixture of diastereomers.

in an aldol-type reaction to produce intermediate **19**, which could then cyclize to oxazolidine product **C** (pathway B).[37–41] Imine **14** (R^1 = H) is believed to participate in a 1,3-dipolar cycloaddition with the aryl aldehyde. However, we cannot exclude a fast stepwise formation of the oxazoline. In contrast, imine **17** (R^1 = Ph) appears to react through an aldol-type pathway. The main indications for this are: (i) The *dr* improved with the use of the 1:1 Ag/PPh_3 catalyst (Table 3, Entry 4), which forms a coordinatively unsaturated silver salt.[42] This can then coordinate to both the azomethine ylide, as well as to the aldehyde, resulting in an increased reactivity and a more ordered transition state (structure **D**). (ii) The observation of side product **20**,[43] which could result from protonation of intermediate **19**, which was not observed when using imine **14**. In line with this, re-exposure of the minor diastereomer of **20** (*anti*) to the reaction conditions resulted in both *cis*- and *trans*-**18**, as well as aldehyde **7a** and imine **17**. However, if *cis*-**18**, the minor stereoisomer formed in the reaction, is re-exposed to the reaction there is no change in the diastereomeric ratio. Taken together, this indicates that the formation of intermediate **19**, or the equivalent, and alcohol **20** is reversible, whereas the cyclization to form oxazolidine **18** appears to be irreversible under the reaction conditions. In this scenario, the beneficial effect of catalytic amounts of an ammonium salt can be rationalized by its protonation of compound **19** at the imine or the alkoxy moiety, assisting in regeneration of the Ag^I catalyst.

Armed with both mechanistic insight and optimized conditions, this protocol was then applied to the preparation of oxazolidines **18a**–**h**. Good to excellent diastereoselectivities and conversions were obtained for many aryl aldehydes, including heteroatom-substituted aryl aldehydes and electron-withdrawing aryl aldehydes (Table 4, Entries 1–3, 5, and 6). To the best of our knowledge, this represents a significant increase in diastereoselectivity for aryl substrates in a direct aldol reaction, especially because these products are not available under phase-transfer conditions.[30] Notable exceptions are *p*-anisaldehyde (**7d**), which reduced the silver catalyst to Ag^0, and hindered 2-tolualdehyde (**7g**). 2-Furaldehyde (**7h**) gave an excellent yield, but the diastereoselectivity was poor.

The hydrolysis of oxazolidines **18a**–**h**, which was reported for the analogous alkyl derivatives,[32] did not proceed as anticipated. It was quickly discovered that both epimerization (Table 4, *dr* **18** vs. **21**) and decomposition of substrates **18a**–**h** occurred. Interestingly, in other reports of access to β-aryl-β-hydroxy-α-amino esters, the deprotection of the benzophenone-derived oxazolidine results in 50% yield, or is not disclosed.[38,39,44] In our case, we believe that protonation of the oxygen atom in **18** can lead to a retro-aldol reaction, resulting in either epimerization to the thermodynamic product[45,38] or to further breakdown to the original starting materials.[9,12,46] Several weaker acids and reaction conditions were screened, resulting in incomplete conversion to the desired amino ester, epimerization, or decomposition. Thus, it seems that the aldol product is poised to undergo epimerization even under mild conditions.

Conclusions

In conclusion, an unusual mechanistic dichotomy in the reaction of azomethine ylides with aldehydes has been disclosed. Depending on the imine substituents, the reaction can proceed through a 1,3-dipolar cycloaddition, affording the corresponding oxazolidines in modest selectivity, or alternatively, the reaction can proceed through an aldol-type mechanism, giving good to excellent diastereoselectivities and conversions with aryl aldehydes. The latter reaction provides mild access to oxazolidines at room temperature and is acid catalyzed. However, manipulation of this pro-

tected amino ester into the deprotected *syn*-β-hydroxy-α-amino ester proved difficult. As the formation of the N,O-acetal is the driving force in the direct aldol reaction, this highlights the need for creative access to this class of molecules circumventing this motif.

Experimental Section

General Methods: ^{1}H and ^{13}C NMR spectra were recorded at 500 and 125 MHz, respectively, in $CDCl_3$ by using the residual peak of $CHCl_3$ (^{1}H NMR δ = 7.26 ppm) and $CDCl_3$ (^{13}C NMR δ = 77.0 ppm) as internal standard. Chemical shifts are reported in the δ-scale with multiplicity (br. = broad, s = singlet, d = doublet, t = triplet, q = quartet, and m = multiplet), coupling constant (Hz), and integration. Dichloromethane (CH_2Cl_2) and tetrahydrofuran (THF) were dried by passing through a solvent column composed of activated alumina. Anhydrous acetonitrile (MeCN) was bought from Sigma–Aldrich Co and stored over activated 4 Å molecular sieves under an atmosphere of N_2. Air- and moisture-sensitive reactions were carried out in flame-dried, septum-capped flasks under an atmospheric pressure of nitrogen. All liquid reagents were transferred by oven-dried syringes. Commercially available compounds were used without further purification unless otherwise indicated. TLC analyses were run on Merck TLC alumina sheets (Silica gel 60 F_{254}) and were visualized by using UV and a solution of phosphomolybdic acid in ethanol (5 wt.-%), $KMNO_4$ stain, or *p*-anisaldehyde stain. Flash chromatography was performed by using silica gel 60 (35–63 μm). Melting points were measured with a Stuart Scientific melting point apparatus (SMP3). IR was recorded with an ATI Mattson Infinity 60 Mi Plus.

3,5-Dimethyl-2-phenyloxazol-3-ium (8): To a solution of 5-methoxy-2-phenyloxazole[47] (169.5 mg, 0.97 mmol) in CH_2Cl_2 (10 mL) was added methyl triflate (159.0 μL, 1.45 mmol), and the resulting solution was stirred at room temperature for 2 h. The mixture was concentrated under reduced pressure to give analytically pure **8** (327.1 mg, 99.6%) as a white solid. M.p. 74.5–75.3 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.87 (d, J = 7.5 Hz, 2 H), 7.77 (t, J = 7.5 Hz, 1 H), 7.68 (t, J = 7.8 Hz, 2 H), 7.54 (s, 1 H, NC*H*=), 4.20 (s, 6 H, NCH_3, OCH_3) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ = 159.8, 152.7, 134.5, 130.1, 129.8, 119.3, 99.2, 60.9, 37.7 ppm. IR (film): $\tilde{\nu}$ = 3496 (br.), 3129, 1666, 1457, 1297, 1151, 1037 cm^{-1}.

Methyl (2*S,3*R**)-3-Hydroxy-2-(methylamino)-3-phenylpropanoate (12a):** To a suspension of anhydrous cesium fluoride (34.6 mg, 0.228 mmol) in dry acetonitrile (1.5 mL) was added a mixture of **8** (38.7 mg, 0.114 mmol), benzaldehyde (38.2 μL, 0.376 mmol), and phenylsilane (21.3 μL, 0.171 mmol) in dry acetonitrile (2 mL) at room temperature. The resulting suspension was stirred at room temperature overnight and then concentrated under reduced pressure. The ratio of **11a/8** was determined by analysis of the ^{1}H NMR spectrum of the crude reaction mixture. The residue was filtered through silica gel to give crude oxazole **11a**.[48] The residue was then dissolved in MeOH/H_2O (2 mL, 95:5), and to this solution was added PTSA (1.5 M in MeOH, 88 μL, 0.133 mmol). The resulting mixture was stirred until no **11a** remained (TLC; heptane/EtOAc, 4:1). The reaction mixture was concentrated under reduced pressure, neutralized with a solution of saturated $NaHCO_3$, and the residue was extracted with CH_2Cl_2 (5×), dried ($MgSO_4$), and concentrated under reduced pressure. Flash chromatography (heptane/EtOAc, 1:2) of the residue gave **12a** (13.8 mg, 58%) as a clear oil. Data for the *syn* isomer: ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.35–7.20 (m, 5 H), 4.59 (d, J = 7.6 Hz, 1 H), 3.55 (s, 3 H), 3.22 (d, J = 7.6 Hz, 1 H), 2.38 (s, 3 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ = 173.5, 140.1, 128.3, 128.0, 126.3, 74.2, 70.6, 51.8, 35.2 ppm. IR (film): $\tilde{\nu}$ = 3430 (br.), 2925, 2854, 1729, 1643 cm^{-1}. HRMS (ESI+): calcd. for $C_{11}H_{15}NO_3$ [M + H]$^+$ 210.11247; found 210.11240. Data for the *anti* isomer: ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.35–7.20 (m, 5 H), 4.98 (d, J = 5.4 Hz, 1 H), 3.65 (s, 3 H), 3.54 (d, J = 5.4 Hz, 1 H), 2.41 (s, 3 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ = 172.6, 140.0, 128.3, 127.9, 125.9, 72.8, 68.6, 51.7, 35.3 ppm.

Imines 14a–e: Prepared by using the method described by Longmire.[25,26]

Ethyl 2-(4-cyanobenzylideneamino)ethanoate (14c): The resulting imine was used crude and was a white powder (quantitative yield). M.p. 81.6–82.7 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.38 (s, 1 H), 7.89 (d, J = 8.4 Hz, 2 H), 7.72 (d, J = 8.4 Hz, 2 H), 4.49 (d, J = 1.3 Hz, 2 H), 4.30 (q, J = 7.1 Hz, 2 H), 1.36 (t, J = 7.1 Hz, 3 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ = 169.6, 163.4, 139.3, 132.4, 128.9, 118.4, 114.5, 61.9, 61.3, 14.2 ppm. IR (film): $\tilde{\nu}$ = 3430, 2227, 1731, 1651, 1209 cm^{-1}. HRMS (ESI+): calcd. for $C_{12}H_{12}N_2O_2$ [M + H]$^+$ 217.09715; found 217.09712.

Ethyl 2-[4-(Trifluoromethyl)benzylideneamino]ethanoate (14d): The resulting imine was used crude and was an off white powder (890 mg, 94% yield). M.p. 58.2–58.6 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.35 (s, 1 H), 7.90 (d, J = 8.1 Hz, 2 H), 7.69 (d, J = 8.2 Hz, 2 H), 4.44 (d, J = 1.2 Hz, 2 H), 4.25 (q, J = 7.1 Hz, 2 H), 1.31 (t, J = 7.1 Hz, 3 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ = 169.8, 163.9, 138.7 (d, $J_{C,F}$ = 1 Hz), 132.8 (q, $J_{C,F}$ = 32.5 Hz), 128.7, 125.6 (q, $J_{C,F}$ = 3.8 Hz), 123.9 (d, $J_{C,F}$ = 272 Hz), 62.0, 61.3, 14.2 ppm. IR (film): $\tilde{\nu}$ = 3438, 2989, 1749, 1648, 1164, 1124 cm^{-1}. HRMS (ESI+): calcd. for $C_{12}H_{12}F_3NO_2$ [M + H]$^+$ 260.08929; found 260.08929.

Methyl 2-[4-(Trifluoromethyl)benzylideneamino]ethanoate (14e): The resulting imine (0.500 mg, 3.6 mmol) was an off white powder and was obtained in quantitative yield. M.p. 81.6–82.7 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.35 (s, 1 H), 7.90 (d, J = 8.1 Hz, 2 H), 7.68 (d, J = 8.2 Hz, 2 H), 4.46 (d, J = 0.7 Hz, 2 H), 3.79 (s, 3 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ = 170.3, 164.0, 138.6 (q, $J_{C,F}$ = 1.1 Hz), 132.8 (q, $J_{C,F}$ = 32.5 Hz), 128.7, 125.6 (q, $J_{C,F}$ = 3.8 Hz), 123.9 (q, $J_{C,F}$ = 272 Hz), 61.9, 52.3 ppm. IR (film): $\tilde{\nu}$ = 3473, 2998, 2956, 1740, 1651 cm^{-1}. HRMS (ESI+): calcd. for $C_{11}H_{10}F_3NO_2$ [M + H]$^+$ 246.07346; found 246.07365.

General Procedure A (Silver-Catalyzed Reaction): A solution of AgOAc (Ag^I salt) (2.8 mg, 0.017 mmol) and PPh_3 (4.5 mg, 0.017 mmol) in the indicated solvent (0.5 mL) was stirred for 45 min, after which time imine **14** (50.2 mg, 0.17 mmol) in the indicated solvent (1.2 mL) and the aldehyde (0.34 mmol) was added. iPr$_2$NEt (5.9 μL, 0.034 mmol) was added, and the resulting mixture was wrapped in aluminum foil and stirred at room temperature overnight. The reaction mixture was filtered through basic alumina (pipette) and concentrated under reduced pressure. The conversion of the 1,3-dipolar cycloaddition was determined by analysis of the ^{1}H NMR spectrum of the crude reaction mixture (integration of the *tert*-butyl ester signals in remaining **14** and oxazoline **16**). The residue was dissolved in MeOH/H_2O (2 mL, 95:5), and to the solution was added PTSA (65 mg, 0.34 mmol). The resulting mixture was stirred until no **16** remained (TLC; heptane/EtOAc, 4:1). The reaction mixture was quenched by the addition of saturated solution of $NaHCO_3$ and concentrated under reduced pressure. The residue was extracted with CH_2Cl_2 (5×), dried ($MgSO_4$), and concentrated under reduced pressure. The diastereomeric ratio was determined by analysis of the ^{1}H NMR spectrum of the crude reaction mixture.

General Procedure B (Benzoic Acid/DIEA Salt): To a solution of AgOTf (4.4 mg, 0.017 mmol) in toluene (0.5 mL) (flask was covered with aluminum foil) was added freshly prepared PPh_3/toluene (0.17 M solution, 100 μL), and the resulting mixture was stirred for 20 to 30 min. Subsequently, imine **17** (50 mg, 0.17 mmol) was added in toluene by cannula (0.5 mL + 0.5 mL rinse) followed by the aldehyde (0.34 mmol). *i*Pr_2NEt (5.9 μL, 0.034 mmol) and benzoic acid (1.03 mg, 0.0085 mmol) were combined in a separate flask and toluene was added to make a standard solution (0.34 M *i*Pr_2NEt and 0.085 M benzoic acid). The salt solution (100 μL) was added, and the resulting mixture was stirred at room temperature overnight. The reaction was concentrated, and the conversion of the aldol addition was determined by analysis of the ^{1}H NMR spectrum of the crude reaction mixture (integration of the *tert*-butyl ester signals in remaining **17** and oxazolidine **18**). The reaction mixture was filtered through basic alumina (pipette) and concentrated under reduced pressure. The residue was dissolved in MeOH (4 mL) and water (0.4 mL), and to the solution was added 0.5 M HCl (0.2 mL). The resulting mixture was stirred until no **18** remained (TLC; heptane/EtOAc, 1:1). The reaction mixture was quenched by the addition of a saturated solution of $NaHCO_3$ and concentrated under reduced pressure. The residue was extracted with CH_2Cl_2 (5×), dried ($MgSO_4$), and concentrated under reduced pressure. The diastereomeric ratio was determined by analysis of the ^{1}H NMR spectrum of the crude reaction mixture.

***tert*-Butyl (4*R**,5*S**)-2,2,5-Triphenyloxazolidine-4-carboxylate (18a):** Prepared according to general procedure by using AgOTf (2.2 mg, 0.0085 mmol), imine **17** (25 mg, 0.085 mmol), and aldehyde **7a** (31.3 mg, 0.17 mmol). Flash chromatography (heptane/EtOAc, 24:1) of the residue gave *trans*-**18a** as a white solid. Data for the *trans* isomer: m.p. 113.0–114.3 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.80–7.70 (m, 2 H), 7.62–7.57 (m, 2 H), 7.39–7.23 (m, 8 H), 7.15 (dd, J = 7.0, 2.4 Hz, 2 H), 4.88 (d, J = 8.4 Hz, 1 H), 3.73 (d, J = 8.4 Hz, 1 H), 3.43 (s, 1 H), 1.36 (s, 9 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ = 169.6, 144.9, 144.4, 139.4, 128.3, 128.3, 128.1, 128.0, 127.8, 127.5, 127.3, 127.0, 126.4, 126.2, 125.6, 101.1, 84.4, 82.3, 69.3, 27.9 ppm. IR (film): $\tilde{\nu}$ = 3313, 3062, 3031, 2979, 1730, 1450, 1157, 700 cm^{-1}. HRMS (ESI+): calcd. for $C_{14}H_{21}NO_4$ [M + H]$^+$ 402.20637; found 402.20639.

***tert*-Butyl (2*S**,3*R**)-2-Amino-3-hydroxy-3-phenylpropanoate (21a):** Prepared according to general procedure b by using aldehyde **7a** (34.6 μL, 0.34 mmol) in PhMe. For the diastereomeric ratio, see Table 4 (analysis of the ^{1}H NMR spectrum of the crude reaction mixture). Flash chromatography (heptane/EtOAc, 1:1 → 1:2) of the residue gave *syn*-**21a** (27.9 mg, 68%) as a clear oil. Data for the *syn* isomer: ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.37–7.26 (m, 5 H), 4.73 (d, J = 5.5 Hz, 1 H), 3.51 (d, J = 5.5 Hz, 1 H), 1.34 (s, 9 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ = 172.5, 140.9, 128.3, 127.8, 126.5, 81.7, 74.6, 61.0, 27.8 ppm. IR (film): $\tilde{\nu}$ = 3370 (br.), 3305, 2980, 2933, 1727, 1369, 1155 cm^{-1}. HRMS (ESI+): calcd. for $C_{13}H_{19}NO_3$ [M + H]$^+$ 238.14377; found 238.14363. Data for the *anti* isomer: ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.36–7.24 (m, 5 H), 4.93 (d, J = 5.5 Hz, 1 H), 3.72 (d, J = 5.5 Hz, 1 H), 1.38 (s, 9 H) ppm.

***tert*-Butyl (2*S**,3*R**)-2-Amino-3-(4-fluorophenyl)-3-hydroxypropanoate (21b):** Prepared according to general procedure b by using aldehyde **7b** (31.3 mg, 0.17 mmol). for the diastereomeric ratio, see Table 4 (analysis of the ^{1}H NMR spectrum of the crude reaction mixture). Flash chromatography (heptane/EtOAc, 1:1 → 1:2) of the residue gave *syn*-**21b** (25.2 mg, 62%) as a white solid. Data for the *syn* isomer: M.p. 68.2–70.3 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.40–7.31 (m, 2 H), 7.09–6.96 (m, 2 H), 4.67 (d, J = 6.0 Hz, 1 H), 3.45 (d, J = 6.0 Hz, 1 H), 1.34 (s, 9 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ = 172.4, 162.4 (d, $J_{C,F}$ = 245 Hz), 136.6 (d, $J_{C,F}$ = 3.1 Hz), 128.2 (d, $J_{C,F}$ = 8.1 Hz), 115.1 (d, $J_{C,F}$ = 21 Hz), 81.9, 74.1, 61.2, 27.9 ppm. IR (film): $\tilde{\nu}$ = 3386 (br.), 2960, 2931, 1728, 1509, 1286, 1155 cm^{-1}. HRMS (ESI+): calcd. for $C_{13}H_{18}FNO_3$ [M + H]$^+$ 256.13435; found 256.13440.

***tert*-Butyl (2*S**,3*R**)-2-Amino-3-(4-bromophenyl)-3-hydroxypropanoate (21c):** Prepared according to general procedure b by using aldehyde **7c** (31.3 mg, 0.17 mmol). For the diastereomeric ratio, see Table 4 (analysis of the ^{1}H NMR spectrum of the crude reaction mixture). Flash chromatography (heptane/EtOAc, 1:1 → 1:2) of the residue gave *syn*-**21c** (26.2 mg, 52%) as a clear oil. Data for the *syn* isomer: ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.48 (m, 2 H), 7.24 (m, 2 H), 4.70 (d, J = 5.5 Hz, 1 H), 3.46 (d, J = 5.5 Hz, 1 H), 1.36 (s, 9 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ = 172.3, 140.1, 131.4, 128.3, 121.7, 82.1, 73.9, 60.9, 27.9 ppm. IR (film): $\tilde{\nu}$ = 3496 (br.), 3305, 2977, 2927, 1727, 1155 cm^{-1}. HRMS (ESI+): calcd. for $C_{13}H_{18}BrNO_3$ [M + H]$^+$ 316.05428; found 316.05420.

***tert*-Butyl (2*S**,3*R**)-2-Amino-3-(3-bromophenyl)-3-hydroxypropanoate (21e):** Prepared according to general procedure b by using aldehyde **7e** (19.8 μL, 0.17 mmol). For the diastereomeric ratio, see Table 4 (analysis of the ^{1}H NMR spectrum of the crude reaction mixture). Flash chromatography (heptane/EtOAc, 1:1 → 1:2) of the residue gave *syn*-**21e** (25.8 mg, 48%) as a clear oil. Data for the *syn* isomer: ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.51 (t, J = 1.7 Hz, 1 H), 7.42 (ddd, J = 7.9, 1.9, 1.1 Hz, 1 H), 7.30 (m, 1 H), 7.22 (t, J = 7.8 Hz, 1 H), 4.68 (d, J = 5.5 Hz, 1 H), 3.47 (d, J = 5.6 Hz, 1 H), 1.37 (s, 9 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ = 172.2, 143.5, 130.8, 129.9, 129.7, 125.2, 122.4, 82.1, 73.8, 60.9, 27.9 ppm. IR (film): $\tilde{\nu}$ = 3363 (br.), 3303, 2977, 2931, 1727, 1369, 1155 cm^{-1}. HRMS (ESI+): calcd. for $C_{13}H_{18}BrNO_3$ [M + H]$^+$ 316.05428; found 316.05423.

***tert*-Butyl (2*S**,3*R**)-2-Amino-3-(3-methoxyphenyl)-3-hydroxypropanoate (21f):** Prepared according to general procedure b by using **7f** (31.3 mg, 0.17 mmol). For the diastereomeric ratio, see Table 4 (analysis of the ^{1}H NMR spectrum of the crude reaction mixture). Flash chromatography (heptane/EtOAc, 1:1 → 1:2) of the residue gave *syn*-**21f** (33.1 mg, 76%) as a clear oil. Data for the *syn* isomer: ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.26 (t, J = 8.1 Hz, 1 H), 6.94–6.93 (dd, J = 4.6, J = 2.7 Hz, 2 H), 6.82 (ddd, J = 8.1, J = 2.5, J = 1.0 Hz, 1 H), 4.75 (d, J = 5.2 Hz, 1 H), 3.18 (s, 3 H), 3.54 (d, J = 5.2 Hz, 1 H), 1.37 (s, 9 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ = 172.4, 159.7 142.7, 129.4, 129.4, 118.8, 113.3, 112.0, 81.9, 74.3, 60.9, 55.3, 27.9 ppm. IR (film): $\tilde{\nu}$ = 3379 (br.), 2978, 2931, 1728, 1257, 1155 cm^{-1}. HRMS (ESI+): calcd. for $C_{14}H_{21}NO_4$ [M + H]$^+$ 268.15433; found 268.15439.

***tert*-Butyl (2*S**,3*R**)-2-Amino-3-(2-methylphenyl)-3-hydroxypropanoate (21g):** Prepared according to general procedure b by using aldehyde **7g** (31.3 mg, 0.17 mmol). For the diastereomeric ratio, see Table 4 (analysis of the ^{1}H NMR spectrum of the crude reaction mixture). Flash chromatography (heptane/EtOAc, 1:1 → 1:2) of the residue gave *syn*-**21g** (19.6 mg, 49%) as a clear oil. Data for the *syn* isomer: ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.42 (dd, J = 7.5, 1.2 Hz, 1 H), 7.21 (dt, J = 7.5, 1.8 Hz, 1 H), 7.17 (dt, J = 7.5, 1.5 Hz, 1 H), 7.12 (d, J = 7.0 Hz, 1 H), 5.05 (d, J = 4.5 Hz, 1 H), 3.54 (d, J = 4.5 Hz, 1 H), 2.34 (s, 3 H), 1.36 (s, 9 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ = 170.7, 137.5, 138.0, 128.7, 125.8, 124.6, 124.3, 80.0, 69.3, 57.4, 26.1, 17.4 ppm. IR (film): $\tilde{\nu}$ = 3370 (br.), 2977, 2931, 1728, 1367, 1155 cm^{-1}. HRMS (ESI+): calcd. for $C_{14}H_{21}NO_3$ [M + H]$^+$ 252.15942; found 252.15961.

***tert*-Butyl (2*S**,3*R**)-2-Amino-3-(furan-2-yl)-3-hydroxypropanoate (21h):** Prepared according to general procedure b by using aldehyde **7h** (28.2 μL, 0.34 mmol). For the diastereomeric ratio, see Table 4

(analysis of the ^{1}H NMR spectrum of the crude reaction mixture). Flash chromatography (heptane/EtOAc, 1:1 → 1:2) of the residue gave an inseparable mixture of *syn*/*anti*-**21h** (34.5 mg, 91%) as a clear oil. Data for the *syn* isomer: ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.38 (dd, J = 1.7, 0.8 Hz, 1 H), 6.33 (dd, J = 3.2, 1.8 Hz, 1 H), 6.31 (d, J = 3.3 Hz, 1 H), 4.77 (d, J = 5.1 Hz, 1 H), 3.76 (d, J = 5.1 Hz, 1 H), 1.40 (s, 9 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ = 172.0, 153.9, 142.1, 110.2, 107.7, 81.9, 68.7, 58.1, 27.9 ppm. Data for the *anti* isomer: ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.32 (dd, J = 1.7, 0.7 Hz, 1 H), 6.31 (m, 1 H), 6.27 (d, J = 3.2 Hz, 1 H), 4.95 (d, J = 5.2 Hz, 1 H), 3.74 (d, J = 5.2 Hz, 1 H), 1.39 (s, 9 H) ppm.^{13}C NMR (125 MHz, $CDCl_3$): δ = 171.8, 153.6, 142.0, 110.1, 107.6, 82.0, 68.0, 58.6, 27.9 ppm. Data for the major isomer: IR (film): $\tilde{\nu}$ = 3434 (br.), 2929, 1729, 1641, 1155 cm^{-1}. HRMS (ESI+): calcd. for $C_{11}H_{17}NO_4$ $[M + H]^+$ 228.12303; found 228.12299.

Supporting Information (see footnote on the first page of this article): ^{1}H and ^{13}C NMR spectra of **8**, **11a**, **12a**, **14c**, **14d**, **14e**, **21a**–**h**.

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