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PFAM catalyzed enantioselective diethylzinc addition to imines

Özdemir DOĞAN*, Eda ÇAĞLI
Department of Chemistry, Middle East Technical University, Ankara, Turkey

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Abstract: Chiral amines are important starting materials for the synthesis of biologically important compounds. Enantioselective addition of dialkylzinc reagents to imines is a reliable method for the synthesis of these compounds. Different chiral catalysts were developed and used for this method. Phosphorous based PFAM catalysts were tried for the first time in the enantioselective synthesis of amines by reacting diethylzinc with N-sulfonyl imines and N-diphenylphosphinoyl imines. Chiral amines were isolated with moderate to acceptable yields and enantioselectivities.

Key words: Chiral amines, diethylzinc addition, sulfonyl imines, phosphinoyl imines

1. Introduction

Chiral amines have a capability of providing high density structural information and H-bonding. Therefore, they are important starting materials for the synthesis of biologically active molecules.[1,2] The catalytic enantioselective addition of dialkylzinc reagents to the C=N double bond of imines is a reliable and important process for the synthesis of optically active amines containing a chirality center at the α-position. However, synthesis of those amines is not easy most of the time.[3,4] Due to the lower reactivity of imine carbon enantioselective addition to C=N double bonds needs to be carried out in the presence of either chiral auxiliaries or chiral ligands. Soai reported the first enantioselective addition of dialkylzinc reagent to the C=N double bond of N-diphenylphosphinoyl imines via the use of a stoichiometric amount of chiral amino alcohol as a promoter and obtained high enantioselectivity.[5] Tomioka was the first to report the same reaction using N-sulfonyl imines with copper-chiral amidophosphine catalyst.[6] Since then different groups have studied this reaction with different chiral catalysts; for example, Gong et al. used copper-bidentate and tridentate bisoxazolines,[7] Wang et al. used copper-chiral ferrocenyl amidophosphines,[8] Shi et al. used copper-chiral binaphthylthiophosphoramides,[9] and Suziki et al. used copper-*N*-heterocyclic carbenes.[10] As examples of diethylzinc addition to N-diphenylphosphinoyl arylaldimine, Charatte et al. used copper-chiral phosphine ligands,[11] Ha et al. used copper-diphosphine and thiophosphoramide ligands,[12] Wang et al. used copper-ferrocenyl amino ketones,[13] Liao et al. used copper-chiral *tert*-butanesulfinylphosphines,[14] and Yus et al. used polymer-supported L-prolinol catalyst.[15] Besides *N*-sulfonyl and *N*-phosphinoyl imines, this reaction was also studied with BOC and formyl protected imines by Aleksakis et al.[16] and Feringa et al.[17] respectively.

However, due to the unavailability of some chiral ligands, impractical reaction conditions, use of excess amounts of dialkylzinc (3–5 equivalents), and the catalytic turnover problems, researchers are still trying to develop a better catalyst system and practical reaction conditions for this reaction. In our previous re-

*Correspondence: dogano@metu.edu.tr

ports, highly enantioselective copper-catalyzed conjugate addition of diethylzinc to substituted chalcones[18] and silver-catalyzed enantioselective 1,3-dipolar cycloaddition reactions of azomethine ylides[19] were accomplished using phosphino ferrocenyl aziridinyl methanol (**PFAM**) and phosphineoxy ferrocenyl aziridinyl methanol (**POFAM**) ligands, respectively. Therefore, we were also interested in investigating the performance of these phosphorous-based ligands for copper-catalyzed enantioselective diethylzinc addition to *N*-sulfonyl and *N*-diphenylphosphinoyl imines.

2. Results and discussion

Various metal salts and chiral ligands were tested for diethylzinc addition to *N*-sulfonyl and *N*-phosphinoyl arylaldimines by different groups. Based on the literature results, copper salts have advantages over the other transition metal salts. Therefore, we tested the performance of chiral phosphino ferrocenyl aziridinyl ketones (**PFAM1-2**) and phosphino ferrocenyl aziridinyl methanols (**PFAM3-6**) with copper salts for diethylzinc addition to aldimines (Figure).

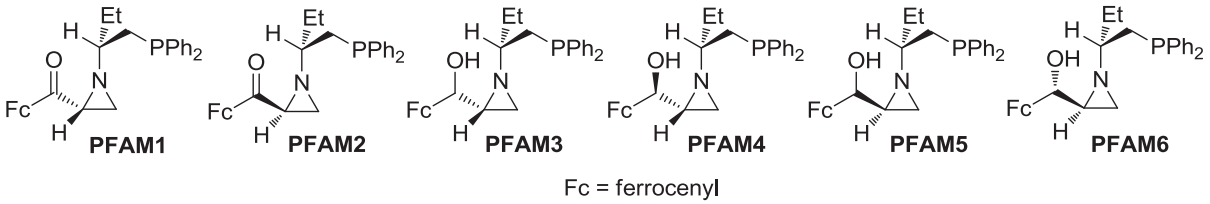
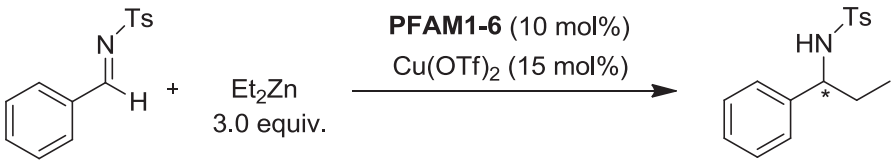


Figure. Structures of **PFAM1-6** ligands.

In order to screen the ligands we chose *N*-sulfonyl imine as the model substrate and adopted the literature procedures[7,13] as the starting point. These studies are summarized in Table 1.

Ligand screening studies (Table 1, entries 1–6) showed that the ligand **PFAM2** formed the product in highest ee. Therefore, further optimizations were done by using this ligand. Changing the reaction solvent from toluene to THF, DCM, or 1,2-dichloroethane (entries 7–9) did not improve enantioselectivity and so we decided to stay with toluene. After ligand and solvent screening, we also looked at the effect of reaction concentration. It was seen that at a concentration lower than 0.05 M (entry 10), the product was obtained in lower yield with no enantioselectivity. At higher concentration (entries 11 and 12), yields were acceptable but the enantioselectivities were low. From the concentration studies, 0.05 M was found to be optimum for this catalyst system. Additives are also commonly used for these reactions. Therefore, in order to see the effect of additives on our catalyst system $^{i}Pr_2NEt$, Et_3N, HMPA, DABCO, MeOH, and TMEDA were tried (entries 15–20). After observing that TMEDA increased the enantioselectivity, it was decided to do further optimization by changing the amount and also the type of copper salt. These studies are summarized in Table 2. Addition of molecular sieves did not change the enantioselectivity but increased the yield more than 10% (compare Table 1 entry 20 with Table 2 entry 1). By increasing the amount of copper salt from 15 mol % to 30 mol %, yield increased further but enantioselectivity decreased (entry 2). When the amount of copper salt was decreased both the yield and enantioselectivity were increased (entry 3). After seeing the effect of molecular sieves, it was decided to dry copper salts by a heat gun under a vacuum line. This resulted in a big jump in enantioselectivity (entry 4). Under dry conditions and a further decrease in the amount of copper salt to 3.5 mol %, enantioselectivity remained almost the same but the yield was low. Therefore, 7 mol % copper salt was determined to be the optimum amount for this catalyst system. We also wanted to find out whether the amount

Table 1. Optimization studies.



Entry	Chiral ligand	Solvent	Conc. (M)	Additive	Temp. (°C)	Yield[a] (%)	ee[b] (%)
1	**PFAM1**	Toluene	0.05	-	–20	60	9
2	**PFAM2**	Toluene	0.05	-	–20	78	17
3	**PFAM3**	Toluene	0.05	-	–20	48	4
4	**PFAM4**	Toluene	0.05	-	–20	< 5	n.d.
5	**PFAM5**	Toluene	0.05	-	–20	30	6
6	**PFAM6**	Toluene	0.05	-	–20	28	4
7	**PFAM2**	THF	0.05	-	–20	20	10
8	**PFAM2**	DCM	0.05	-	–20	60	11
9	**PFAM2**	1,2-DCE	0.05	-	–20	99	5
10	**PFAM2**	Toluene	0.025	-	–20	39	rac
11	**PFAM2**	Toluene	0.1	-	–20	52	6
12	**PFAM2**	Toluene	0.2	-	–20	66	6
13	**PFAM2**	Toluene	0.05	-	0	86	rac
14	**PFAM2**	Toluene	0.05	-	–50	29	rac
15	**PFAM2**	Toluene	0.05	$^{i}Pr_2NEt$	–20	31	15
16	**PFAM2**	Toluene	0.05	Et_3N	–20	59	rac
17	**PFAM2**	Toluene	0.05	HMPA	–20	58	rac
18	**PFAM2**	Toluene	0.05	DABCO	–20	74	rac
19	**PFAM2**	Toluene	0.05	MeOH	–20	70	3
20	**PFAM2**	Toluene	0.05	TMEDA	–20	59	20

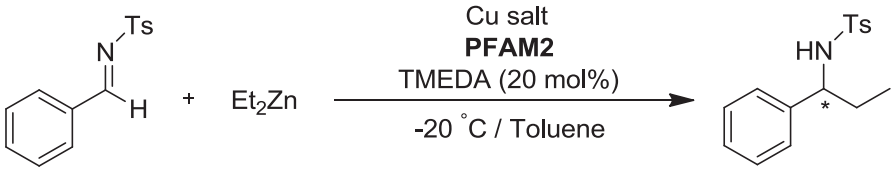
[a]Isolated yield.
[b]Determined by chiral HPLC.

of diethylzinc could be lowered. Running the experiment with 1.5 equivalents of diethylzinc, yield remained the same but the ee improved (entry 7). Reducing the diethylzinc to 1.0 equivalent, however, the product was obtained in lower yield and enantioselectivity (entry 8). We also tried longer and shorter reaction times but the results did not improve (entries 11 and 12). Finally, changing the diethyzinc addition time was also tried but with no success.

After deciding on the optimized conditions (toluene as the solvent, 0.5 M reaction concentration, 1.5 equivalents of diethylzinc, –20 °C as the reaction temperature, TMEDA as the additive, and 7 mol % $Cu(OTf)_2$), substrate screening studies were carried out. The results of these studies are summarized in Table 3. As can be seen, *p*-methoxy, or *p*-bromo phenyl groups on the substrate **1**, formed the product in lower yields and enantioselectivities (entries 2 and 3). Changing the aryl group to 1-naphthyl or 2-naphthyl on the substrate resulted in lower ee (entries 3 and 4). When phosphinoyl imine was used as the substrate, only 2-naphthylphosphinoyl imine formed the product in acceptable yield and ee. For the others (**2g**–**i**) yields were low and so the enantioselectivities were not determined (entries 7–9).

In conclusion, we tested the performance of a new catalyst system, copper-**PFAM**, for enantioselective diethylzinc addition to both sulfonyl and phosphinoyl imines. This catalyst system served better for sulfonyl imines than phosphinoyl imines. It was found that the reaction had to be carried out under dry conditions.

Table 2. Additive and copper salt screening.



Entry	Ligand (mol %)	Et_2Zn (equiv)	Cu salt	Cu salt (mol %)	Yield[a] (%)	ee[b] (%)
1[c]	10	3.0	$Cu(OTf)_2$	15	73	22
2[c]	10	3.0	$Cu(OTf)_2$	30	80	6
3[c]	10	3.0	$Cu(OTf)_2$	7	90	30
4[c,d]	10	3.0	$Cu(OTf)_2$	7	56	73
5[c,d]	10	3.0	$Cu(OTf)_2$	3.5	40	72
6[c,d]	10	3.0	$Cu(OAc)_2$	7	66	65
7	10	1.5	$Cu(OTf)_2$	7	55	77
8	10	1.0	$Cu(OTf)_2$	7	47	53
9	5	1.5	$Cu(OTf)_2$	7	99	25
10	20	1.5	$Cu(OTf)_2$	7	50	53
11[e]	10	1.5	$Cu(OTf)_2$	7	48	70
12[e]	10	1.5	$Cu(OTf)_2$	7	30	65
13[f]	10	1.5	$Cu(OTf)_2$	7	67	56
14[f]	10	1.5	$Cu(OTf)_2$	7	53	72

[a]Isolated yield.

[b]Determined by chiral HPLC.

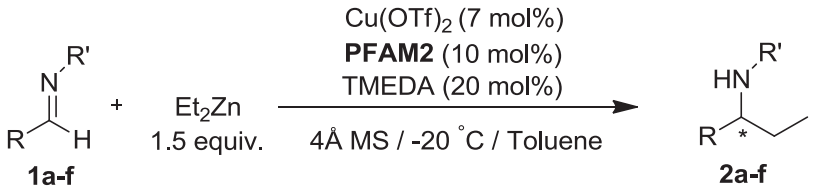
[c]Reaction was carried out with 4 Å molecular sieves.

[d]$Cu(OTf)_2$ was dried via heat gun.

[e]Reaction time for entries 11 and 12 was 42 h and 15 h, respectively; for the rest it was 21 h.

[f]Et_2Zn addition time for entries 13 and 14 was 1 h and 4.5 h, respectively; for the rest it was 3 h.

Table 3. Arylaldimine screening studies.



Entry	R	R'	Product	Yield[a] (%)	ee[b] (%)
1	Ph	Ts	**2a**	55	77
2	4-$MeOC_6H_4$	Ts	**2b**	43	43
3	4-BrC_6H_4	Ts	**2c**	50	56
4	1-Naphtyl	Ts	**2d**	60	26
5	2-Naphtyl	Ts	**2e**	30	12
6	2-Naphtyl	Ph_2PO	**2f**	50	52
7	Ph	Ph_2PO	**2g**	< 5	n.d
8	4-$MeOC_6H_4$	Ph_2PO	**2h**	< 5	n.d
9	4-BrC_6H_4	Ph_2PO	**2i**	< 5	n.d

[a]Isolated yield.

[b]Determined by chiral HPLC. Comparing the HPLC retention times with the literature,[6] absolute configurations of **2a**[6] and **2f**[11] were assigned as *S*.

3. Experimental

3.1. General

All reactions were carried out in flame-dried glassware under reduced pressure and charged with argon or nitrogen unless otherwise stated. Air- and moisture-sensitive imines and chiral phosphorous containing ligands were stored under inert atmosphere. They were transferred via syringe to the reactor after being dissolved in reaction solvent under inert medium. During the work-up procedure for the synthesis of derivatives of imines, TiO_2 was filtered through a Celite pad (Merck Celite 545) and washed with dichloromethane. They were further purified by recrystallization with an appropriate dry mixture of solvents if needed. Commercial copper salts, $Cu(OAc)_2$ and $Cu(OTf)_2$, were benzene-azeotroped or dried under vacuum with a heating gun before use. Toluene was dried with sodium and charged with nitrogen. Tetramethylethylenediamine (TMEDA) was distilled over sodium under vacuum and stored with potassium hydroxide pellets under inert atmosphere. Just before use, 4 Å molecular sieves were activated at high temperature.

Ethyl addition products were purified via flash column chromatography on Silica Gel 60 (E. Merck, particle size: 0.040–0.063 mm, 230–400 mesh ASTM). TLC analyses were performed on 250-μm Silica Gel 60 F254 plates. Enantiomeric excess (ee) was determined by chiral HPLC. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker Spectrospin Avance DPX-400 Ultra shield instrument at 400 and 100 MHz, respectively, relative to TMS. Optical rotations were measured by Rudolph Research Analytical Autopol III Polarimeter. *N*-arylmethylsulfonamides and *N*-aryldiphenylphosphinoylamides were synthesized by the literature procedures.[20,21] Chiral ligands **PFAM1-6** were synthesized as in the literature.[18,19]

3.2. General procedures for asymmetric diethylzinc addition to arylaldimines

In a 10-mL flame-dried Schlenk tube purged with nitrogen, $Cu(OTf)_2$ (7.0 mg, 0.020 mmol) and 4 Å molecular sieves were added and dried under reduced pressure via heating. Toluene (3.5 mL) was added at room temperature. Then, chiral ligand (13.0 mg, 0.026 mmol) was dissolved in toluene (0.5 mL) and the reaction was stirred for 1 h at room temperature. After 1 h, imine (68.0 mg, 0.230 mmol) and TMEDA (8.0 μL, 0.04 mmol) were added consecutively. After complete dissolution of imine, the reaction mixture was cooled to –20 °C. Et_2Zn in 1.1 M toluene (0.47 mL, 0.34 mmol) was added dropwise at regular time intervals over 3 h and the reaction mixture was stirred overnight. At this point, before work-up, the reaction mixture was analyzed by taking the ^{1}H NMR, which showed no imine proton at 9 ppm corresponding to the starting material. The reaction was quenched with saturated NH_4Cl aqueous solution (4.0 mL). The organic phase was extracted twice with DCM (2 × 10.0 mL) and the combined organic phase was dried over Na_2SO_4. The crude product was purified by flash column chromatography on silica gel (pentane:acetone 10:1). The pure product was obtained in moderate to good yields. The enantioselectivity was determined by HPLC using a Chiralcel.

3.2.1. *N*-[1-phenylpropan-1-yl]-4-methylbenzenesulfonamide (2a)

R_f = 0.40 pentane/acetone 5:1; $[\alpha]_D^{21}$ = –84.2 (c 0.25, CH_2Cl_2) for 77% ee and –96.2 (c 0.25, CH_2Cl_2) for 81% ee. Lit.[9] $[\alpha]_D^{25}$ = –52.2 (c 2.97, $CHCl_3$) for 86% ee. ^{1}H NMR δ 6.99–7.52 (m, 9H), 4.81 (d, J = 7.5 Hz, 1H), 4.18 (q, J = 7.4 Hz, 1H), 2.31 (s, 3H), 1.75 (dq, J =15.8 Hz, J = 7.9 Hz, 2H), 0.78 (t, J = 7.5 Hz, 3H); ^{13}C NMR δ 142.9, 141.5, 139.9, 137.6, 129.3, 128.8, 128.7, 128.3, 128.1, 128.0, 127.3, 126.0, 55.0, 26.7, 21.3, 10.4. HPLC: Chiralcel OD column, UV detection at 254 nm, eluent: hexane/iPrOH 10:1, flow 0.7 mL min^{-1}, t_R = 15.1 min (minor) and 21.3 min (major).

3.2.2. *N*-[1-(4-methoxyphenyl)propan-1-yl]-4-methylbenzenesulfonamide (2b)

R_f = 0.43 pentane/acetone 5:1; $[\alpha]_D^{21}$ = –7.6 (c 0.25, CH_2Cl_2) for 43% ee. Lit.[9] $[\alpha]_D^{25}$ = – 80.1 (c 1.50, $CHCl_3$) for 90% ee. ^{1}H NMR δ 7.54 (d, J = 7.9 Hz, 2H), 7.13 (d, J = 7.4 Hz, 2H), 6.92 (d, J = 7.9 Hz, 2H), 6.68 (d, J = 8.2 Hz, 2H), 4.90 (d, J = 7.0 Hz, 1H), 4.13 (q, J = 7.2 Hz, 1H), 3.74 (s, 3H), 2.37 (s, 3H), 1.74 (dq, J = 45.4 Hz, J =8.3 Hz, 2H), 0.76 (t, J = 7.2 Hz, 3H); ^{13}C NMR δ 142.9, 132.8, 129.2, 127.7, 127.2, 114.8, 113.7, 59.6, 55.3, 30.5, 21.4, 10.4. HPLC: Chiralcel OD-H column, UV detection at 254 nm, eluent: hexane/iPrOH 10:1, flow 0.6 mL min^{-1}, t_R = 29.8 min (minor) and 34.4 min (major).

3.2.3. *N*-[1-(4-bromophenyl)propan-1-yl]-4-methylbenzenesulfonamide (2c)

R_f = 0.41 pentane/acetone 5:1; $[\alpha]_D^{21}$ = –20.9 (c 0.25, CH_2Cl_2) for 56% ee. Lit.[9] $[\alpha]_D^{25}$ = –25.5 (c 1.25, $CHCl_3$) for 92% ee. ^{1}H NMR δ 7.49 (d, J = 7.9 Hz, 2H), 7.26 (d, J = 7.3 Hz, 2H), 7.13 (d, J = 7.9 Hz, 2H), 6.88 (d, J = 7.3 Hz, 2H), 4.80 (d, J = 7.0 Hz, 1H), 4.17 (q, J = 7.2 Hz, 1H), 2.38 (s, 3H), 1.70 (dq, J = 45.0 Hz, J = 7.6 Hz, 2H), 0.78 (t, J = 7.5 Hz, 3H); ^{13}C NMR δ 141.2, 131.4, 129.3, 128.4, 126.9, 121.2, 59.2, 30.3, 21.4, 10.4. HPLC: Chiralpak AS-H column, UV detection at 254 nm, eluent: hexane/iPrOH 75:25, flow 0.7 mL min^{-1}, t_R = 30.7 min (minor) and 54.8 min (major).

3.2.4. *N*-[1-(1-naphthylmethylene)propan-1-yl]-4-methylbenzenesulfonamide (2d)[9]

R_f = 0.52 pentane/acetone 5:1. ^{1}H NMR δ 7.82–7.23 (m, 9H), 6.88 (d, J = 8.0 Hz, 2H), 5.23 (d, J = 7.7 Hz, 1H), 5.04 (q, J = 7.2 Hz, 1H), 2.45 (s, 3H), 1.94 (dq, J =14.6 Hz, J =4.3 Hz, 2H), 0.85 (t, J = 7.5 Hz, 3H); ^{13}C NMR δ 142.8, 142.6, 133.7, 131.3, 131.2, 130.6, 129.8, 129.1, 128.9, 128.7, 127.8, 127.3, 126.8, 126.7, 126.1, 126.0, 125.5, 125.2, 125.1, 123.9, 123.2, 122.6, 55.8, 45.4, 30.4, 21.2, 10.7. HPLC: Chiralpak AD column, UV detection at 254 nm, eluent: hexane/iPrOH 85:15, flow 0.7 mL min^{-1}, t_R = 19.1 min (minor) and 24.2 min (major).

3.2.5. *N*-[1-(2-naphthylmethylene)propan-1-yl]-4-methylbenzenesulfonamide (2e)[7]

R_f = 0.47 pentane/acetone 5:1, $[\alpha]_D^{21}$ = –8.8 (c 0.25, CH_2Cl_2) for 12% ee. ^{1}H NMR δ 7.69–7.27 (m, 8H), 7.06 (dd, J = 8.6 Hz, J =1.84 Hz, 1H), 6.84 (d, J = 8.1 Hz, 2H), 4.94 (d, J = 7.6 Hz, 1H), 4.28 (q, J = 7.3 Hz, 1H), 2.09 (s, 3H), 1.78 (dq, J = 40.4 Hz, J = 7.53 Hz, 2H), 0.76 (t, J = 7.4 Hz, 3H); ^{13}C NMR δ 142.8, 142.6, 135.8, 128.5, 128.4, 127.7, 127.5, 127.0, 126.5, 126.4, 126.0, 125.9, 124.9, 124.8, 124.7, 124.0, 122.6, 59.9, 46.4, 30.4, 21.5, 10.8. HPLC: Chiralpak AD column, UV detection at 254 nm, eluent: hexane/iPrOH 85:15, flow 0.7 mL min^{-1}, t_R = 21.5 min (minor) and 29.0 min (major).

3.2.6. *N*-[1-(2-naphthyl)propyl]-P,P-diphenylphosphinic amide (2f)[11]

R_f = 0.30 pentane/acetone 5:1. ^{1}H NMR δ 7.91–7.75 (m, 3H), 7.65 (d, J = 7.7 Hz, 2H), 7.47–7.22 (m, 10H), 6.91 (d, J = 8.0 Hz, 2H), 5.04 (q, J = 7.0 Hz, 1H), 4.97 (d, J = 7.3 Hz, 1H), 1.96 (dq, J = 14.4 Hz, J =3.5 Hz, 2H), 0.86 (t, J = 7.5 Hz, 3H); ^{13}C NMR δ 133.2, 133.1, 133.1, 133.0, 131.7, 128.9, 128.8, 128.7, 58.1, 31.4 (J_{C-P} = 4.8 Hz), 18.2 (J_{C-P} = 5.3 Hz), 10.8. HPLC: Chiralpak AD column, UV detection at 254 nm, eluent: hexane/iPrOH 80:20, flow 1.0 mL min^{-1}, t_R = 10.3 min (minor) and 12.7 min (major).

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