Reaction of α-carbanion of imines with *N*-tosylimines: a facile route to β-aminoaldehydes and 1,3-diamines

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Deprotonation of aldimines by LDA, followed by trapping of the resulting carbanion with *N*-tosylimines and hydrolysis or reduction provides a convenient access to β-aminoaldehydes or 1,3-diamines.

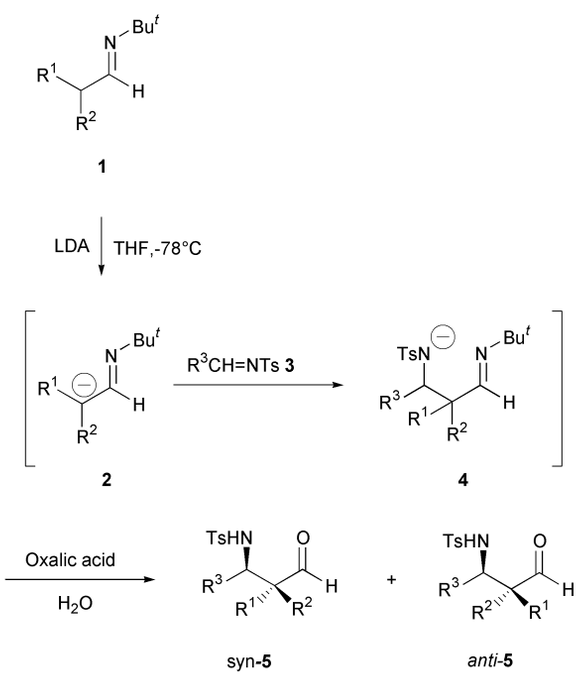
Introduction

Recently, the use of imines as starting materials in the synthesis of nitrogen-containing compounds has attracted a lot of interest from synthetic chemists.[1] A number of reactions of imines, such as aziridination,[2] alkylation, aldol reaction, hetero-Diels–Alder reaction, have been well documented.[2] All of these reactions utilize the carbon of the C=N double bond as an electrophile or the C=N double bond as dienophile. There are only a few examples of the use of α-carbanions derived from imines,[3] although the reactions of α-carbanions of carbonyl compounds, an imine analog, are important in organic synthesis.[4] As a program aimed at the applications of imines,[2a,g,5] we have studied the formation of carbanions from imines and their subsequent reactions. Now we report the deprotonation of imines and the reaction of the thus formed α-carbanion with *N*-tosylimines to provide β-aminoaldehydes and 1,3-diamines.[6]

Results and discussion

In the presence of LDA, deprotonation of imines **1** gave rise to α-carbanions of imines **2**, which reacted with *N*-tosylimines **3** to afford β-amino imines **4**. Direct hydrolysis of amino imines **4** with oxalic acid delivered β-aminoaldehydes **5** (Scheme 1). The results are showed in Table 1.

This is a one-pot reaction. All aldimines **1** reacted with *N*-tosylimines **3** to give rise to the intermediates, β-amino-aldimines **4**, which allowed direct hydrolysis. The β-amino-aldehydes were provided in good yield after three-step reactions. The substituent R^1 can be H and Me and R^2 can be linear and branched alkyl group. The stereoselectivity of the reaction is usually low, but in some cases it is high (Entries 7 and 9). A change of solvent from THF to toluene had little influence on the stereochemistry outcome. The presence of additives, such as molecular sieves, $LiClO_4$, also did not change the *syn–anti* ratio of products. The stereochemistry of products was determined from the *J* value of the aldehyde proton from ^{1}H-NMR and confirmed further by X-ray diffraction analysis of **5g** (Fig. 1).[7]



Scheme 1

Table 1 Synthesis of β-aminoaldehydes from aldimines **1** and **3**[a]

Entry	R^1	R^2	R^3	Product (yield %)[b]	*syn* : *anti*[c]
1	H	Me	4-ClC_6H_4	**5a** (61)	40 : 60
2	Me	Me	4-ClC_6H_4	**5b** (72)	
3	H	Et	Ph	**5c** (81)	23 : 77
4	H	Et	4-$MeOC_6H_4$	**5d** (79)	26 : 74
5	H	Et	4-ClC_6H_4	**5e** (80)	25 : 75
6	H	Pr	Ph	**5f** (63)	25 : 75
7	H	^{i}Pr	Ph	**5g** (82)	8 : 92
8	H	^{i}Pr	4-$MeOC_6H_4$	**5h** (73)	22 : 78
9	H	^{i}Pr	4-ClC_6H_4	**5i** (83)	8 : 92

[a] All reactions were carried out with the ratio of LDA : imine : *N*-tosylimine = 1.6 : 1.5 : 0.75. [b] Isolated yields based on the *N*-tosylimine. [c] Determined by 300 MHz ^{1}H-NMR.

In order to trap the carbanion intermediate **2**, the use of activated imine is crucial. The imines with phenyl and diphenylphosphinoyl groups as substituent on nitrogen failed to react with carbanions **2** to give any product. *N*-Tosylimines, with either electron-donating or electron-withdrawing substituents on the phenyl ring give the desired products. Enamines have been used widely as a synthon of α-carbanions of carbonyl compounds,[8] however, the reaction of enamines derived from cyclohexanone and piperidine with PhCH=NTs or PhCH=NPh–TMSCl did not provide the desired products. The choice of base is also important.[3e] In the presence of LDA, aldimines

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Table 2 Synthesis of 1,3-diamines from aldimines **1** and **3**[a]

Entry	R^1	R^2	R^3	Product (yield %)[b]
1	H	C_2H_5	4-ClC_6H_4	**6a** (79)
2	H	$CH(CH_3)_2$	C_6H_5	**6b** (82)
3	H	$CH(CH_3)_2$	4-ClC_6H_4	**6c** (85)

[a] All reactions were carried out with the ratio of LDA : imine : *N*-tosylimine = 1.6 : 1.5 : 0.75. [b] Isolated yields based on the *N*-tosylimine.

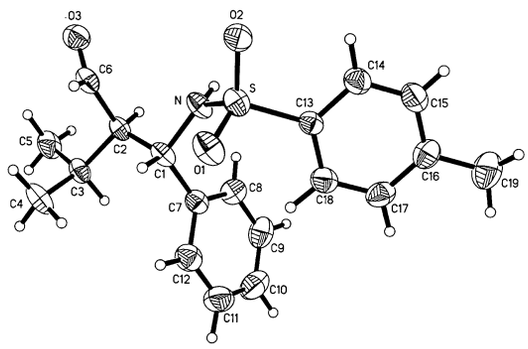
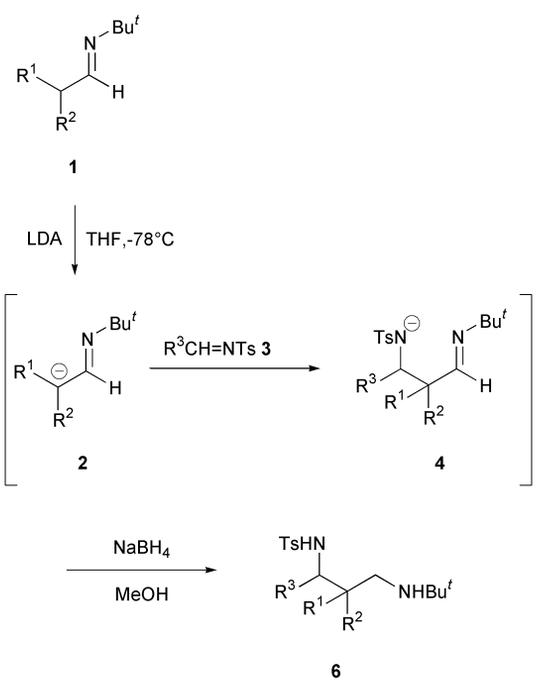


Fig. 1 ORTEP drawing of **5g**.

with hydrogen at the α-position afforded the carbanion, however, no carbanion formed if sodium hydride was used as base. Butyllithium reacted with the C=N double bond.[2e] With lithium bis(trimethylsilyl)amide as base, the product is complex.[9]

If $NaBH_4$–MeOH instead of oxalic acid was used, β-amino aldimines **4** can also be reduced smoothly to give 1,3-diamines in good yield (Scheme 2, Table 2). The NMR of **6** showed that



Scheme 2

there were two –N*H*Ts peaks with a ratio of around 1 : 1. Thus the epimerization should take place during the reduction reaction because both reactions have the same intermediate **4** and hydrolysis of **4** gave the products with *syn* : *anti* ratio of 40 : 60 to 8 : 92.

In conclusion, new and one-pot procedures have been developed to prepare β-aminoaldehydes and 1,3-diamines from α-carbanions of imines in a convenient way. The usefulness of α-carbanions of imines in organic synthesis is also demonstrated. The investigation on the reactions of α-carbanions of imines with other kinds of nucleophiles and the asymmetric version of them are in progress.

Experimental

General

All the reactions were performed under a dry argon atmosphere. The commercially available reagents were used without further purification. THF was freshly distilled under nitrogen from a purple solution of sodium and benzophenone. ^{1}H-NMR spectra were recorded on a Bruker AMX-300 (300 MHz) spectrometer and the chemical shifts were referenced to $CHCl_3$ (δ 7.27) in $CDCl_3$, IR spectra were recorded in neat solutions, and measured in cm^{-1}, using a Shimadzu IR-440 infrared spectrophotometer. Mass spectra were taken using HP5989A. Elemental analyses were performed on a Foss–Heraeus Vario EL instrument.

General procedure for the preparation of β-aminoaldehydes 5

To a solution of aldimine **1** (1.5 mmol) in THF (5 mL) was added LDA (1 mL, 1.6 M in THF) at 0 °C under argon, the resulting mixture was stirred for 2 h, then was cooled to −78 °C and *N*-tosylimine **2** (0.75 mmol) was added. The mixture was stirred at this temperature for 7 h. A solution of oxalic acid (144 mg, 1.6 mmol) in water (2 mL) was added dropwise and the mixture was stirred for 12 h. The mixture was extracted with Et_2O (10 mL × 3). The organic layer was combined, washed with brine (10 mL × 2) and dried over Na_2SO_4. The solvent was removed in vacuum and the residue was purified by flash column chromatography by using petroleum ether (60–90 °C) and ethyl acetate (3 : 1) as the eluent to obtain the corresponding β-aminoaldehydes **5** as a *syn–anti* mixture.

3-(*N*-Tosylamino)-3-(4′-chlorophenyl)-2-methylpropanal 5a. ^{1}H-NMR: δ ($CDCl_3$–Me_4Si) *syn*-**5a**: 1.09 (d, J = 7.4 Hz, 3H), 2.37 (s, 3H), 2.76 (m, 1H), 4.69 (dd, J = 6.1 and 9.1 Hz, 1H), 6.05 (d, J = 9.4 Hz, 1H), 6.94 (m, 2H), 7.09 (m, 4H), 7.46 (m, 2H), 9.53 (d, J = 1.2 Hz, 1H); *anti*-**5a**: 0.96 (d, J = 7.2 Hz, 3H), 2.35 (s, 3H), 2.76 (m, 1H), 4.52 (dd, J = 8.4 and 9.0 Hz, 1H), 6.16 (d, J = 9.1 Hz, 1H), 6.94 (m, 2H), 7.09 (m, 4H), 7.46 (m, 2H), 9.62 (d, J = 2.7 Hz, 1H); MS: *m*/*z* (%) 351 (M^+, 1.98), 260 (90), 91 (100); IR (cm^{-1}) 3273, 2977, 2925, 2720, 1721, 1598, 1495, 1455; Anal. Calcd $C_{17}H_{18}ClNO_3S$: C, 58.03; H, 5.16; N, 3.98. Found: C, 57.76; H, 5.43; N, 3.96%.

3-(*N*-Tosylamino)-3-(4′-chlorophenyl)-2,2-dimethylpropanal 5b. ^{1}H-NMR: δ ($CDCl_3$–Me_4Si) 1.01 (s, 3H), 1.05 (s, 3H), 2.33 (s, 3H), 4.46 (d, J = 9.9 Hz, 1H), 6.21 (d, J = 9.9 Hz, 1H), 6.85 (d, J = 8.3 Hz, 2H), 7.02 (d, J = 8.6 Hz, 4H), 7.41 (d, J = 8.3 Hz, 2H), 9.49 (s, 1H); MS: *m*/*z* (%) 365 (M^+, 0.25), 293 (100); IR(cm^{-1}) 3263, 2928, 2843, 2710, 1735, 1598, 1461, 1437; Anal. Calcd. $C_{18}H_{20}ClNO_3S$: C, 59.09; H, 5.51; N, 3.83. Found: C, 59.04; H, 5.78; N, 3.57%.

3-(*N*-Tosylamino)-3-phenyl-2-ethylpropanal 5c. ^{1}H-NMR: δ ($CDCl_3$–Me_4Si) *syn*-**5c**: 0.92 (t, J = 7.3 Hz, 3H), 1.37–1.60 (m, 2H), 2.32 (s, 3H), 2.58 (m, 1H), 4.59 (dd, J = 8.0 and 8.9 Hz, 1H), 5.55 (d, J = 8.9 Hz, 1H), 6.95–7.11 (m, 7H), 7.45 (m, 2H), 9.46 (d, J = 2.3 Hz, 1H); *anti*-**5c**: 0.89 (t, J = 7.8 Hz, 3H), 1.37–1.60 (m, 2H), 2.30 (s, 3H), 2.58 (m, 1H), 4.59 (dd, J = 8.0 and 8.9 Hz, 1H), 5.72 (d, J = 9.5 Hz, 1H), 6.95–7.11 (m, 7H), 7.45 (m, 2H), 9.56 (d, J = 3.2 Hz, 1H); MS: *m*/*z* (%) 314 (M^+ − 17, 6.23), 260 (100); IR(cm^{-1}) 3240, 3028, 2968, 2697, 1726, 1598, 1494, 1455, 1385, 1324; Anal. Calcd. $C_{18}H_{21}NO_3S$: C, 65.23; H, 6.38; N, 4.22. Found: C, 65.03; H, 6.29; N, 4.35%.

3-(*N*-Tosylamino)-3-(4′-methoxyphenyl)-2-ethylpropanal 5d. ^{1}H-NMR: δ ($CDCl_3$–Me_4Si) *syn*-**5d**: 0.90 (t, J = 7.4 Hz, 3H),

1.33 (m, 1H), 1.51 (m, 1H), 2.33 (s, 3H), 2.54 (m, 1H), 3.71 (s, 3H), 4.54 (dd, J = 9.1 and 8.8 Hz, 1H), 5.77 (d, J = 9.0 Hz, 1H), 6.63 (m, 2H), 6.88 (m, 2H), 7.06 (m, 2H), 7.46 (m, 2H), 9.44 (d, J = 2.3 Hz, 1H); *anti*-**5d**: 0.84 (t, J = 7.8 Hz, 3H), 1.33 (m, 1H), 1.51 (m, 1H), 2.31 (s, 3H), 2.54 (m, 1H), 3.72 (s, 3H), 4.54 (dd, J = 9.1 and 8.8 Hz, 1H), 5.90 (d, J = 8.9 Hz, 1H), 6.63 (m, 2H), 6.88 (m, 2H), 7.06 (m, 2H), 7.46 (m, 2H), 9.58 (d, J = 3.9 Hz, 1H); MS: *m*/*z* (%) 290 (M^+ − 71, 100); IR(cm^{-1}) 3261, 2960, 2923, 2710, 1726, 1613, 1515, 1461; Anal. Calcd. $C_{19}H_{23}NO_4S$: C, 63.14; H, 6.41; N, 3.88. Found: C, 62.81; H, 6.50; N, 3.57%.

3-(*N*-Tosylamino)-3-(4′-chlorophenyl)-2-ethylpropanal 5e. ^{1}H-NMR: δ ($CDCl_3$–Me_4Si) *syn*-**5e**: 0.89 (m, 3H), 1.36–1.67 (m, 2H), 2.34 (s, 3H), 2.55 (m, 1H), 4.57 (m, 1H), 5.85–6.06 (m, 1H), 6.91–7.06 (m, 6H), 7.43 (m, 2H), 9.44 (d, J = 2.1 Hz, 1H); *anti*-**5e**: δ 0.89 (m, 3H), 1.36–1.67 (m, 2H), 2.33 (s, 3H), 2.55 (m, 1H), 4.57 (m, 1H), 5.85–6.06 (m, 1H), 6.91–7.06 (m, 6H), 7.43 (m, 2H), 9.56 (d, J = 3.5 Hz, 1H); MS: *m*/*z* (%) 294 (M^+ − 71, 43), 155 (30), 91 (100); IR(cm^{-1}) 3261, 2960, 2923, 2710, 1726, 1613, 1515, 1461, 1323; Anal. Calcd. $C_{18}H_{20}ClNO_3S$: C, 59.08; H, 5.50; N, 3.82. Found: C, 59.12; H, 5.43; N, 3.67%.

3-(*N*-Tosylamino)-3-(4′-methoxyphenyl)-2-propylpropanal 5f. ^{1}H-NMR: δ ($CDCl_3$–Me_4Si) *syn*-**5f**: 0.85 (m, 3H), 1.31–1.53 (m, 4H), 2.33 (s, 3H), 2.69 (m, 1H), 4.58 (m, 1H), 5.49 (d, J = 8.5 Hz, 1H), 6.94–7.14 (m, 7H), 7.47 (m, 2H), 9.47 (d, J = 2.3 Hz, 1H); *anti*-**5f**: 0.85 (m, 3H), 1.31–1.53 (m, 4H), 2.32 (s, 3H), 2.69 (m, 1H), 4.58 (m, 1H), 5.67 (d, J = 9.4 Hz, 1H), 6.94–7.14 (m, 7H), 7.47 (m, 2H), 9.54 (d, J = 3.0 Hz, 1H); MS: *m*/*z* (%) 328 (M^+ − 17, 1.48), 260 (32), 155 (34), 91 (100), 77 (10); IR (cm^{-1}): 3269, 2959, 2870, 2710, 1729, 1457, 1381, 1322, 1289; Anal. Calcd. $C_{19}H_{23}NO_3S$: C, 66.06; H, 6.71; N, 4.05. Found: C, 65.86; H, 6.80; N, 3.94%.

3-(*N*-Tosylamino)-3-phenyl-2-isopropylpropanal 5g. ^{1}H-NMR: δ ($CDCl_3$–Me_4Si) *syn*-**5g**: 1.01 (m, 6H), 1.80 (m, 1H), 2.32 (s, 3H), 2.54 (m, 1H), 4.71 (dd, J = 9.1 and 8.8 Hz, 1H), 5.58 (d, J = 8.8 Hz, 1H), 7.01 (m, 7H), 7.42 (m, 2H), 9.45 (d, J = 3.4 Hz, 1H); *anti*-**5g**: 1.01 (m, 6H), 1.80 (m, 1H), 2.29 (s, 3H), 2.54 (m, 1H), 4.82 (dd, J = 8.9 and 8.0 Hz, 1H), 5.96 (d, J = 9.4 Hz, 1H), 7.01 (m, 7H), 7.42 (m, 2H), 9.74 (d, J = 3.4 Hz, 1H); MS: *m*/*z* (%) 314 (M^+ − 31, 2.23), 260 (70), 91 (100); IR(cm^{-1}) 3247, 2966, 2770, 1708, 1600, 1490, 1456, 1329; Anal. Calcd. $C_{19}H_{23}NO_3S$: C, 66.06; H, 6.71; N, 4.05. Found: C, 65.80; H, 6.66; N, 3.76%.

3-(*N*-Tosylamino)-3-(4′-methoxyphenyl)-2-isopropylpropanal 5h. ^{1}H-NMR: δ ($CDCl_3$–Me_4Si) *syn*-**5h**: 1.00 (m, 6H), 1.75 (m, 1H), 2.33 (s, 3H), 2.53 (m, 1H), 3.70 (s, 3H), 4.69 (dd, J = 8.7 and 9.0 Hz, 1H), 6.00 (d, J = 9.1 Hz, 1H), 6.60 (m, 2H), 6.89 (m, 2H), 7.03 (m, 2H), 7.41 (m, 2H), 9.44 (d, J = 3.4 Hz, 1H); *anti*-**5h**: 0.97 (d, J = 7.7 Hz, 3H), 0.99 (d, J = 7.7 Hz, 3H), 1.75 (m, 1H), 2.31 (s, 3H), 2.53 (m, 1H), 3.72 (s, 3H), 4.78 (dd, J = 8.9 and 9.1 Hz, 1H), 6.00 (d, J = 9.1 Hz, 1H), 6.60 (m, 2H), 6.89 (m, 2H), 7.03 (m, 2H), 7.41 (m, 2H), 9.76 (d, J = 4.0 Hz, 1H); MS: *m*/*z* (%) 314 (M^+ − 31, 2.23), 260 (70), 155 (43), 91 (100); IR(cm^{-1}) 3247, 2966, 2770, 1708, 1600, 1490, 1456, 1329; Anal. Calcd. $C_{20}H_{25}NO_4S$: C, 63.97; H, 6.71; N, 3.73. Found: C, 64.14; H, 7.03; N, 3.50%.

3-(*N*-Tosylamino)-3-(4′-chlorophenyl)-2-isopropylpropanal 5i. ^{1}H-NMR: δ ($CDCl_3$–Me_4Si) *syn*-**5i**: 1.00 (m, 6H), 1.76 (m, 1H), 2.35 (s, 3H), 2.51 (m, 1H), 4.67 (dd, J = 9.5 and 8.2 Hz, 1H), 5.90 (d, J = 8.7 Hz, 1H), 6.91 (m, 2H), 7.03 (m, 4H), 7.39 (m, 2H), 9.45 (d, J = 3.2 Hz, 1H); *anti*-**5i**: 1.00 (m, 6H), 1.76 (m, 1H), 2.34 (s, 3H), 2.51 (m, 1H), 4.80 (dd, J = 9.2 and 8.4 Hz, 1H), 6.21 (d, J = 9.6 Hz, 1H), 6.91 (m, 2H), 7.03 (m, 4H), 7.39 (m, 2H), 9.74 (d, J = 3.7 Hz, 1H); MS: *m*/*z* (%) 290 (M^+ − 85, 2.23), 155 (22), 92 (22), 91 (100); IR(cm^{-1}) 3249, 2961, 2710, 1720, 1612, 1597, 1561; Anal. Calcd. $C_{19}H_{22}ClNO_3S$: C, 60.07; H, 5.83; N, 3.69. Found: C, 59.82; H, 5.82; N, 3.59%.

General procedure for the preparation of 1,3-diamines 6

To a solution of aldimine **1** (1.5 mmol) in THF (5 mL) was added LDA (1 mL, 1.6 M in THF) at 0 °C under argon, the resulting mixture was stirred for 2 h, then was cooled to −78 °C, and *N*-tosylimine (**2**, 0.75 mmol) was added, the mixture was stirred at this temperature for 7 h. Then $NaBH_4$ (10 mmol) in MeOH (4 mL) was added and the temperature was allowed to rise to room temperature. After stirring for 3 h at room temperature, HCl (15 mL, 2 M) was added and the mixture was extracted with Et_2O (3 × 20 mL). Subsequently, the aqueous phase was basified by the addition of NH_3, and the 1,3-diamines **6** were extracted with CH_2Cl_2 (3 × 20 mL). The combined organic phase was dried over Na_2SO_4. The solvent was removed in vacuum and the residue was purified by preparative TLC with a mixture of light petroleum (60–90 °C) and ethyl acetate (3 : 1) as the eluent to give pure product **6** as a *syn–anti* mixture.

3-(*N*-Tosylamino)-3-(4′-chlorophenyl)-2-ethyl-*N*-(*tert*-butyl)-propylamine 6a. ^{1}H-NMR: δ ($CDCl_3$–Me_4Si) *syn*-**6a**: 0.89 (t, J = 7.6 Hz, 3H), 1.13 (s, 9H), 0.90–1.68 (m, 4H), 2.36 (s, 3H), 2.42 (d, J = 10.3 Hz, 1H), 2.56 (m, 1H), 4.61 (d, J = 4.1 Hz, 1H), 7.05 (m, 6H), 7.45 (m, 2H); *anti*-**6a**: 0.79 (t, J = 7.3 Hz, 3H), 1.15 (s, 9H), 0.90–1.68 (m, 4H), 2.35 (s, 3H), 2.56 (m, 1H), 2.74 (dd, J = 1.8 and 12.0 Hz, 1H), 4.36 (d, J = 6.8 Hz, 1H), 7.05 (6H, m), 7.45 (2H, m); MS: *m*/*z* (%) 423 (M^+ + 1, 11.85), 407 (32.24), 91 (27.28), 86 (100); IR (cm^{-1}) 3302, 2967, 2876, 1599, 1492, 1323, 1290, 1215; Anal. Calcd. $C_{22}H_{31}ClN_2O_2S$: C, 62.46; H, 7.39; N, 6.62. Found: C, 62.43; H, 7.52; N, 6.43%.

3-(*N*-Tosylamino)-3-phenyl-2-isopropyl-*N*-(*tert*-butyl)propyl-amine 6b. ^{1}H-NMR: δ ($CDCl_3$–Me_4Si) *syn*-**6b**: 0.87 (d, J = 6.4 Hz, 3H), 0.95 (d, J = 6.3 Hz, 3H), 1.14 (s, 9H), 1.27–1.53 (m, 3H), 2.33 (s, 3H), 4.70 (d, J = 3.9 Hz, 1H), 7.03–7.11 (m, 7H), 7.43 (m, 2H); *anti*-**6b**: 0.51 (d, J = 6.6 Hz, 3H), 0.83 (d, J = 7.0 Hz, 3H), 1.15 (s, 9H), 1.27–1.53 (m, 3H), 2.32 (s, 3H), 4.59 (d, J = 7.0 Hz, 1H), 7.03–7.11 (m, 7H), 7.43 (m, 2H); MS: *m*/*z* (%) 387 (M^+ − 15, 20.82), 260 (11.09), 91 (100); IR (cm^{-1}) 3298, 2958, 2872, 1601, 1493, 1454, 1346; Anal. Calcd. $C_{23}H_{34}N_2O_2S$: C, 68.61; H, 8.51; N, 6.96. Found: C, 68.33; H, 8.36; N, 6.56%.

3-(*N*-Tosylamino)-3-(4′-chlorophenyl)-2-isopropyl-*N*-(*tert*-butyl)propylamine 6c. ^{1}H-NMR: δ ($CDCl_3$–Me_4Si) *syn*-**6c**: 0.85 (d, J = 6.8 Hz, 3H), 0.94 (d, J = 6.8 Hz, 3H), 1.17 (s, 9H), 1.14–1.58 (m, 4H), 2.53 (s, 3H), 2.39–2.55 (m, 1H), 2.71 (t, J = 10.3 Hz, 1H), 4.69 (d, J = 3.8 Hz, 1H), 7.03 (m, 6H), 7.41 (m, 2H); *anti*-**6c**: 0.52 (d, J = 6.7 Hz, 3H), 0.81 (d, J = 6.7 Hz, 3H), 1.13 (s, 9H), 1.14–1.58 (m, 4H), 2.34 (s, 3H), 2.39–2.55 (m, 1H), 2.71 (t, J = 10.3 Hz, 1H), 4.50 (d, J = 7.6 Hz, 1H), 7.03 (m, 6H), 7.41 (m, 2H); MS: *m*/*z* (%) 437 (M^+ + 1, 17.02), 421 (23.03), 86 (100); IR (cm^{-1}) 3303, 2957, 2872, 1598, 1491, 1412, 1312, 1248, 1214. Anal. Calcd. $C_{23}H_{33}ClN_2O_2S$: C, 63.21; H, 7.61; N, 6.40. Found: C, 63.08; H, 7.67; N, 6.16%.

X-Ray diffraction analysis data of 3-*N*-tosylamino-3-phenyl-2-isopropylpropanal 5g †

Chemical formula: $C_{19}H_{23}NO_3S$; formula weight: 345.44; crystal system: triclinic; unit-cell dimensions and volume with estimated standard deviations: cell length *a*: 9.4619(11); cell length *b*: 9.8687(12); cell length *c*: 10.1330(12); cell angle alpha: 100.097(2); cell angle beta: 99.529(2); cell angle gamma: 91.423(2); cell volume: 917.26(19); data collection temperature: 293 K; No. of formula units in unit cell (*Z*): 2; linear absorption coefficient(μ): 0.192 mm^{-1}; Number of reflections measured and/or number of independent reflections, 3371 (*R*int) = 0.0401; Final *R* indices [$I > 2$sigma(I)]: *R*1 = 0.0406, w*R*2 = 0.0507.

† CCDC reference number 177332. See http://www.rsc.org/suppdata/p1/b2/b200198e/ for crystallographic files in .cif or other electronic format.

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