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Scandium triflate as an efficient and useful catalyst for the synthesis of β-amino alcohols by regioselective ring opening of epoxides with amines under solvent-free conditions

Andrew T. Placzek, James L. Donelson, Rushi Trivedi, Richard A. Gibbs and Surya K. De*

Department of Medicinal Chemistry and Molecular Pharmacology, School of Pharmacy, Purdue Cancer Center, Purdue University, West Lafayette, IN 47907, USA

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Abstract—A simple and efficient method has been developed for the synthesis of β-amino alcohols by ring opening of epoxides in the presence of a catalytic amount of $Sc(OTf)_3$ at room temperature under solvent-free conditions. The reaction works well with both aromatic and aliphatic amines. High regio-, and diastereoselectivity can be considered as a noteworthy advantage of this method.
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1. Introduction

β-Amino alcohols are versatile building blocks in the synthesis of a wide range of biologically active natural and synthetic products,[1] unnatural amino acids,[2] and chiral auxiliaries.[3] The classical synthesis of β-amino alcohols involves the ring opening of epoxides with amines;[4] however, these reactions, which are generally carried out with large excess of amines at elevated temperatures, often fail when poorly nucleophilic amines or sterically crowded amines/epoxides are concerned. In addition, as a rule, these reactions are accompanied by poor regioselectivity of ring opening. Subsequently, several activators/promoters such as metal amides (lithium, magnesium, lead, tin),[5] metal alkoxide (DIPAT, $Ti(OiPr)_4$,[6] metal triflates (lithium, Ph_4SbOTf, lanthanide, tin,),[7] metal halides ($TaCl_5$, $ZrCl_4$, VCl_3, $ZnCl_2$, $CeCl_3$),[8] silica under high pressure,[9] ionic liquids,[10] clay,[11] and others[12] have been developed to perform the epoxide ring opening reaction under mild conditions. However, many of these methods often involve the use of expensive and stoichiometric amounts of reagents, air, and/or moisture sensitive catalysts, poor regioselectivity especially with metal amides, long reaction times, and also entail undesirable side reactions such as the rearrangement of epoxides to allyl alcohols under basic conditions or polymerization in strongly acidic conditions resulting in low yields of the desired products. In most cases, the reactions were carried out with aromatic amines only. Moreover, the main disadvantage of almost all existing methods is that the catalysts are destroyed in the work-up procedure and cannot be recovered or reused. To overcome these limitations, we wish to report an efficient method for the ring opening of epoxides with both aromatic and aliphatic amines catalyzed by scandium triflate at room temperature.

Since the beginning of the new century, green chemistry has become a major driving force for organic chemists to develop environmentally benign routes to a myriad of materials. The possibility of performing multi-component reactions under solvent-free conditions with solid catalysts could enhance their efficiency from an economic as well as an ecological point of view, so solvent free chemical reactions have received much attention. These reactions offer several advantages in preparative procedures such as environmental compatibility, simplification of work-ups, formation of cleaner products, enhanced selectivity, reduction of by-products, a reduction in waste produced, and much improved reaction rates. Therefore, there is a need to develop new methods for the synthesis of β-amino alcohols using less hazardous solvents, or better, those that do not need solvents at all. Therefore, there is further scope to search

* Corresponding author. Fax: +765 494 1414; e-mail: skd125@pharmacy.purdue.edu

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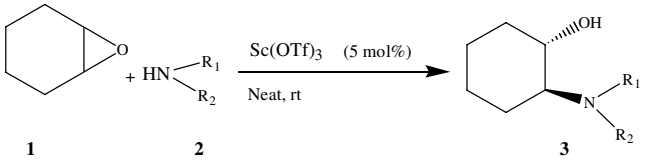
for a better catalyst in terms of operational simplicity, reusability, and economic viability. There is also a need to find out what works best under solvent-free conditions.

In the continuation of our work to develop new organic transformations, we report herein that scandium triflate, which acts as a mild Lewis acid, might be a useful and inexpensive catalyst for the synthesis of β-amino alcohols.

2. Results and discussion

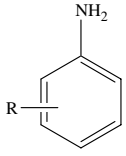
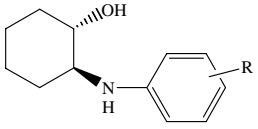
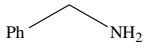
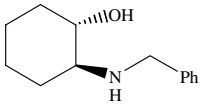
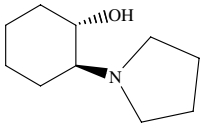
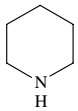
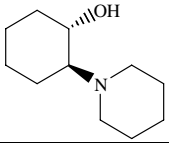
Most recently, there has been considerable interest growing in the use of scandium triflate as a potential Lewis acid in various organic reactions[13] because the catalyst is quite stable in water and reusable. The scandium triflate is commercially available and can be used for the synthesis of β-amino alcohols by the regioselective ring opening of epoxides with amines. In the case of cyclohexene oxide (Scheme 1 and Table 1), only one isomer was formed. The aryl oxiranes underwent cleavage by a variety of amines in a regioselective fashion with preferential attack at the benzylic position in the presence of scandium triflate at room temperature in neat (Schemes 1 and 2). Interestingly, the sterically bulky 2,6-diisopropylaniline underwent the cleavage to afford the corresponding β-amino alcohol in good yield. Unlike previously reported methods, the present method gave in good yields with aliphatic amines (entries 4, 5, 6 in Table 1 and entries 6, 7, 8 in Table 2). Aliphatic oxiranes gave the major product with the opposite regiochemistry of the aromatic substrate (entry 3, 4, 5 in Table 3). Therefore, we suggest that the attack of nucleophile is governed by the nature of oxirane and the stability of carbonium ion. In aryl oxirane, the positive charge on oxygen appears to be localized on the more highly substituted benzylic carbon leading to the major product (Scheme 3). For aliphatic oxirane gave the opposite regiochemistry of the aromatic substrates, possibly steric factors predominate over electronic factors. In case of propylene oxide (Table 3, entry 3) or glycidyl *tert*-butyl ether (Table 3, entry 4), the selective nucleophilic attack at the less hindered carbon of epoxide was observed. The cyclic aliphatic amines such as morpholine, piperidine, pyrrolidine reacted with aliphatic epoxide to afford in good yields. However, simple acyclic aliphatic amines such as pentyl amine and hexyl amine did not give any satisfactory yields (20%) in the reaction of aliphatic epoxide (propylene oxide). We isolated starting materials and/or undesired products (Scheme 4). The scope and generality of this method is



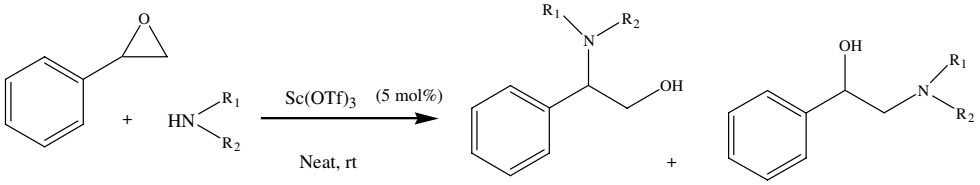
a: R_1 = H, R_2 = C_6H_5; b: R_1 = H, R_2 = 4-MeC_6H_4;
c: R_1 = H, R_2 = 4-ClC_6H_4; d: R_1 = R_2 = $C_6H_5CH_2$;
e: R_1= R_2= $(CH_2)_4$; f: R_1 = R_2 = $(CH_2)_5$

Scheme 1. $Sc(OTf)_3$-catalyzed epoxide ring opening of **1** with various amines.

Table 1. Reactions of **1** with various amines under neat conditions at room temperature catalyzed by $Sc(OTf)_3$

Entry	Amine	Product	Time (h)	Yield[a] (%)
				
1	R = H	R = H	1	92
2	R = 4-Me	R = 4-Me	3	88
3	R = 4-Cl	R = 4-Cl	2	91
4			3	86
5			2	90
6			2	92

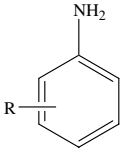
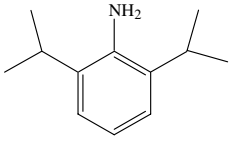
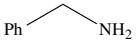
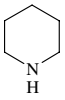
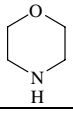
[a] Isolated yields of the corresponding amino alcohol and in all cases one isomer was observed.



a: R_1 = H, R_2 = C_6H_5; b: R_1 = H, R_2 = 4-MeC_6H_4;
c: R_1= H, R_2 = 4-$MeOC_6H_4$; d: R_1 = H, R_2 = 2,6-disopropyl-C_6H_3;
e: R_1 = H; R_2 = $C_6H_5CH_2$; f: R_1 = R_2 = $(CH_2)_5$;
g: R_1= R_2 = $(CH_2)_2O(CH_2)_2$.

Scheme 2. Regioselectivity in $Sc(OTf)_3$-catalyzed epoxide ring opening of **4** with various amines.

Table 2. Regioselectivity during the reaction of **4** with various amines in the presence of $Sc(OTf)_3$ at room temperature under neat conditions

Entry	Amine	Time (h)	Yield[a] (%)	Ratio **5**:**6**[b]
				
1	R = H	2	95	95:5
2	R = 4-Me	4	88	90:10
3	R = 4-OMe	3	90	94:6
4	R = 4-NO_2	4	91	92:8
5		5	82	88:12
6		2	89	15:85
7		2	91	25:75
8		3	92	30:70

[a] Isolated yields.
[b] Regioisomers were determined using ^1H NMR.

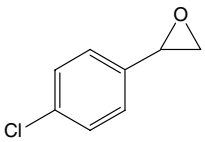
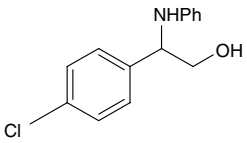
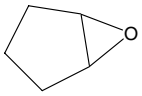
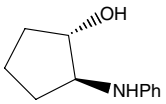
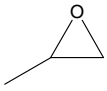
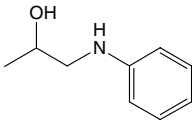
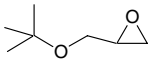
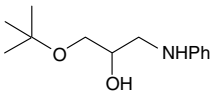
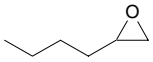
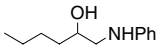
illustrated with respect to various epoxides and amines and the results are presented in Tables 1–3.

In comparison with other catalysts such as $CoCl_2$, $BiCl_3$, $TaCl_5$, $CeCl_3$, VCl_3, $Cu(OTf)_2$, which are recently reported in the ring opening of styrene oxide with amines, $Sc(OTf)_3$ employed here shows more catalytic reactivity than the others in terms of the amount of catalyst required, reaction times, and yields of the product (Table 2). The efficacy of other catalysts such as $InCl_3$, $RuCl_3$, $Nd(OTf)_3$, $Lu(OTf)_3$, $Yb(OTf)_3$, was studied for this reaction. Among these catalysts, $Sc(OTf)_3$ was found to be superior in terms of conversion and reaction times (Table 4). The reported catalysts as shown in Table 4 are not effective for reaction with benzyl amines. Possibly these catalysts are strong Lewis acids making the formation of complex metal salts with aliphatic amines cause the catalyst destruction and make these metal salts ineffective for use as catalysts for the opening of epoxide rings by aliphatic amines. Due to the mild acidic nature of $Sc(OTf)_3$, it works effectively for epoxide ring opening reaction with aliphatic amines. The results summarized in Tables 1–3 reveal that the present method is applicable for aromatic as well as aliphatic amines.

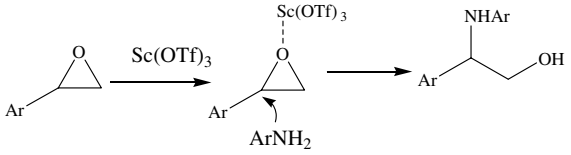
3. Conclusion

In conclusion, $Sc(OTf)_3$ is a new, highly efficient, and reusable catalyst for the opening of epoxides with amines leading to the generation of β-amino alcohols.

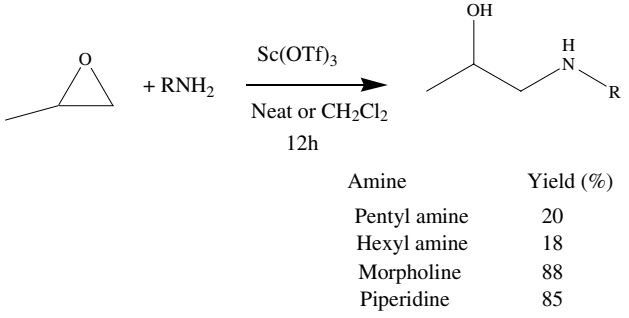
Table 3. Reaction of various epoxides with aniline in the presence of $Sc(OTf)_3$ at room temperature under neat conditions

Entry	Epoxide	Product (major)	Time (h)	Yield[a] (%)
1			2	92[b]
2			2	94
3			3	95
4			4	87
5			5	85[b]

[a] Isolated yields.
[b] Other regioisomer 5–10% was determined using ^1H NMR.



Scheme 3.



Amine	Yield (%)
Pentyl amine	20
Hexyl amine	18
Morpholine	88
Piperidine	85

Scheme 4.

The advantages of this method include (a) short reaction times, (b) applications on both aromatic and aliphatic amines, (c) high yields of products, (d) excellent regio-, and stereoselectivity, (e) the use of relatively cheap commercially available reusable reagents, and (f) solvent-free conditions.

4. Experimental

NMR spectra were recorded on a Bruker ARX 299 (300 MHz) instrument. Low resolution mass spectra (CI, EI) were recorded on a Finnigan 4000 mass spectrometer. High resolution mass spectra (HRMS, EI, CI, ESI) were recorded on a Finnigan MAT XL95 mass spectrometer. The reactions were monitored by TLC, and visualized with UV light followed by development using 15% phosphomolybdic acid in ethanol. All solvents and reagents were purchased from Aldrich with high grade quality, and used without any purification. All products are known and were identified by comparison with those reported in the literature.

Table 4. Ring opening of styrene oxide (**4**) with benzyl amine: effects of catalysts

Entry	Catalyst	Yield (%)
1	$CoCl_2$	0 (Ref. 14)
2	$BiCl_3$	0 (Ref. 15)
3	TaCls	0 (Ref. 8a)
4	VCl^3	0 (Ref. 8d)
5	$CeCl_3$	0 (Ref. 8f)
6	$Cu(OTf)_2$	0 (Ref. 16)
7	HFIP	0 (Ref. 17)
8	Zr-salt	0 (Ref. 18)
9	$InCl_3$	0
10	$RuCl_3$	0
11	$Lu(OTf)_3$	72
12	$Nd(OTf)_3$	61
13	$Yb(OTf)_3$	79
14	$Sc(OTf)_3$	95

5. Typical procedure for reaction of epoxide with amine

To a mixture of cyclohexene oxide (490 mg, 5 mmol) and aniline (465 mg, 5 mmol), $Sc(OTf)_3$ (123 mg, 0.25 mmol) was added at room temperature. After the

completion of reaction [TLC, $R_f = 0.4$ (30% ethyl acetate in hexane)], the reaction mixture was partitioned between ether (60 mL) and water (20 mL). The aqueous layer containing the catalyst has been recovered and reused for three times with a modest loss in activity (reaction yields: 87%, 69%, 55%). The organic layer was washed with water (40 mL), aqueous $NaHCO_3$ solution (20 mL), and brine (40 mL), respectively, dried ($MgSO_4$), and concentrated. The residue was purified by column chromatography (30% ethyl acetate in hexane) to give a pure *trans*-2(phenylamino)cyclohexanol. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.02–1.43 (m, 4H), 1.71–1.82 (m, 2H), 2.11–2.23 (m, 2H), 2.82 (brs, 1 H, NH), 3.14 (ddd, $J = 4.1$, 10.1, 10.2 Hz, 1H), 3.36 (ddd, $J = 4.2$, 10.2, 10.4 Hz, 1H), 6.71–7.23 (m, 5H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 24.3, 25.4, 31.7, 33.2, 60.3, 74.5, 114.5, 118.5, 129.4, 147.9; EIMS *m*/*z* 191 (M^+), 174, 99, 82, 77, 41.

5.1. Selected spectral data

5.1.1. 2-Anilino-2-phenyl-1-ethanol (entry 1, Table 2). Liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.73 (dd, $J = 10$, 7 Hz, 1H), 3.94 (dd, $J = 10$, 4 Hz, 1H), 4.53 (dd, $J = 10.8$, 6.8 Hz, 1H), 6.52 (d, $J = 7.2$ Hz, 2H), 6.72 (t, $J = 7.2$ Hz, 1H), 7.11 (t, $J = 7.2$ Hz, 2H), 7.31–7.45 (m, 5H); EIMS *m*/*z* 213 (M^+), 195, 107, 77, 57.

5.1.2. 2-(4-Methylphenyl)amino-2-phenyl-1-ethanol. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.16 (s, 3H), 3.69 (dd, $J = 7.4$, 11.2 Hz, 1H), 3.87 (dd, $J = 4.2$, 11.2 Hz, 1H), 4.45 (dd, $J = 4.2$, 7.4 Hz, 1H), 6.48 (d, $J = 8$ Hz, 2H), 6.92 (d, $J = 8$ Hz, 2H); ^{13}C NMR (75 MHz, CDCl3) δ 20.5, 60.3, 66.9, 114.2, 119.6, 126.6, 127.3, 128.5, 129.6, 140.2, 144.8; EIMS *m*/*z* 227 (M^+), 196, 77.

5.1.3. 2-(2,6-Diisopropylphenyl)amino-2-phenyl-1-ethanol. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.18–1.27 (m, 12H), 3.04–3.27 (m, 4H), 4.95 (dd, $J = 7.9$, 3.6 Hz, 1H), 7.02–7.46 (m, 8H); ^{13}C NMR (75 MHz, $CDCl_3$) 24.4, 27.6, 58.5, 73.4, 123.6, 124.2, 125.9, 127.8, 128.7, 142.2, 142.3, 142.8; EIMS *m*/*z* 297 (M^+), 190, 175, 160, 107.

5.1.4. 2-*N*-Benzylamino-1-phenyl-ethanol. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.58 (br s, 2H), 2.77 (dd, $J = 8.8$, 12.1 Hz, 1H), 2.96 (dd, $J = 3.7$, 12.2 Hz, 1H), 3.82 (d, $J = 13.2$ Hz, 1H), 3.88 (d, $J = 13.2$ Hz, 1H), 4.74 (dd, $J = 3.7$, 8.8 Hz, 1H), 7.24–7.41 (m, 10H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 53.8, 56.7, 72.1, 126.2, 127.4, 127.8, 128.4, 128.7, 128.8, 140.2, 142.8; EIMS *m*/*z* 227 (M^+).

5.1.5. 2-(1-Piperidino)-1-phenylethanol. Mp 55–56 °C; 1H NMR (300 MHz, $CDCl_3$) δ 1.51(m, 2H), 1.60 (m, 4H), 2.41 (m, 4H), 2.71 (m, 2H), 4.71 (dd, $J = 3.7$, 6.6 Hz, 1H), 7.20–7.32 (m, 5H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.2, 24.4, 26.2, 54.4, 66.9, 68.7, 125.5, 125.8, 127.5, 142.6.

5.1.6. 1-(4-Methylphenylamino)hexan-2-ol. Liquid; ^{1}H NMR(300 MHz, $CDCl_3$) δ 0.92 (t, $J = 6.9$ Hz, 3H), 1.24–1.63 (m, 6H), 2.21 (s, 3H), 2.94 (dd, $J = 12.4$, 8.2 Hz, 1H), 3.26 (dd, $J = 12.4$, 3.4 Hz, 1H), 3.82 (m, 1H), 6.62 (d, $J = 8$ Hz, 2H), 6.89 (d, $J = 8$ Hz, 2H); EIMS *m*/*z* 207 (M^+), 190, 178, 135, 121, 107, 84, 57.

5.1.7. *trans*-2-(Phenylamino)cyclohexanol. Mp 61–63 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.02–1.43 (m, 4H), 1.71–1.82 (m, 2H), 2.11–2.23 (m, 2H), 2.82 (brs, 1 H, NH), 3.14 (ddd, $J = 4.1$, 10.1, 10.2 Hz, 1H), 3.36 (ddd, $J = 4.2$, 10.2, 10.4 Hz, 1H), 6.71–7.23 (m, 5H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 24.3, 25.4, 31.7, 33.2, 60.3, 74.5, 114.5, 118.5, 129.4, 147.9; EIMS *m*/*z* 191 (M^+), 174, 99, 82, 77, 41.

5.1.8. *trans*-2-(Benzylamino)cyclohexanol. Mp 73–74 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.92–1.05 (m, 1H), 1.12–1.32 (m, 3H), 1.65–1.72 (m, 2H), 1.96 (m, 1H), 2.12–2.29 (m, 2H), 2.84 (s, 1H), 3.18 (m, 1H), 3.62 (d, $J = 12.8$ Hz, 1H), 3.91 (d, $J = 12.8$ Hz, 1H), 7.18–7.38 (m, 5H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 24.6, 25.3, 30.7, 33.6, 51.1, 63.4, 74.1, 127.2, 128.4, 128.6, 140.9; EIMS *m*/*z* 205 (M^+).

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