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Highly diastereoselective epoxidation of protected α-amino alkenes catalyzed by ruthenium porphyrin/Cl_2PyNO system

Li Sun, Can-Ping Du, Jie Qin, Jing-Song You *, Meng Yang, Xiao-Qi Yu *

Department of Chemistry, Key Laboratory of Green Chemistry and Technology (Ministry of Education), Sichuan University, 29 Wangjiang Road, Chengdu 610064, PR China

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Abstract

Protected α-amino epoxides were successfully obtained in high *threo*-selectivity (up to 99%) under mild reaction conditions by using carbonyl 5,10,15,20-tetra(2,6-dichlorophenyl)porphyrin ruthenium(II) [Ru(TDCPP)CO] as catalyst and 2,6-dichloropyridine N-oxide (Cl_2PyNO) as oxidant.
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Keywords: Diastereoselective Epoxidation; Protected α-amino alkenes; Ruthenium porphyrin carbonyl

1. Introduction

Amino epoxides are not only important building blocks for constructing a variety of densely functionalized compounds such as amino sugars and oxygenated amino acids [1], but also serve as intermediates in the preparation of several dipeptide isosteres, which can be used to synthesize the inhibitors of some key aspartic proteases (e.g., rennin [2] and HIV protease [3]). Scheme 1 shows an example of synthetic route of inhibitors of HIV-1 protease involving the direct coupling of protected α-amino epoxides and amido enolates as an entry into this compound class [3b]. The conversion of L-α-amino acids to a halomethyl ketone, stereoselective reduction with Felkin-Anh control and following ring closure are common ways to access highly stereoselective *threo*-amino epoxides [4]. In addition, Luly et al. [5] prepared the epoxides via the *stoichiometric* epoxidation of protected α-amino alkenes with *m*-chloroperoxybenzoic acid (*m*-CPBA). The development of chemistry that could emanate from a successful catalytic method for such a process has not yet been apparently attained. Herein we develop a conveniently catalytic method for affording *threo*-amino epoxides in high diastereoselectivity (up to 99%) by using carbonyl 5,10,15,20-tetra(2,6-dichlorophenyl)porphyrin ruthenium(II) as catalyst and 2,6-dichloropyridine N-oxide as oxidant under mild reaction conditions.

2. Results and discussion

2.1. Preparation of N-protected α-amino alkenes

The preparation of N-protected α-amino alkenes (*S*)-**5** was developed as outlined in Scheme 2. This route involved initial treatment of natural L-amino acids **1** with thionyl chloride, followed by addition of di-*tert*-butylpyrocarbonate (Boc_2O) or acetic anhydride to afford methyl N-protected amino acid **3**, and the compounds **3** could then be converted to the desired N-protected amino aldehydes **4** by direct reduction with diisobutylaluminum hydride (DIBAH) [4]. Compounds **4** were treated directly with the ylide derived from *n*-BuLi and CH_3PPh_3Br to give N-protected α-amino alkenes (*S*)-**5**. Using the same procedure, we obtained N-protected α-amino alkene (*R*)-**6** from D-leucine

* Corresponding authors. Tel.: +86 28 85415886; fax: +86 28 85412285.
E-mail address: schemorg@mail.sc.cninfo.net (X.-Q. Yu).

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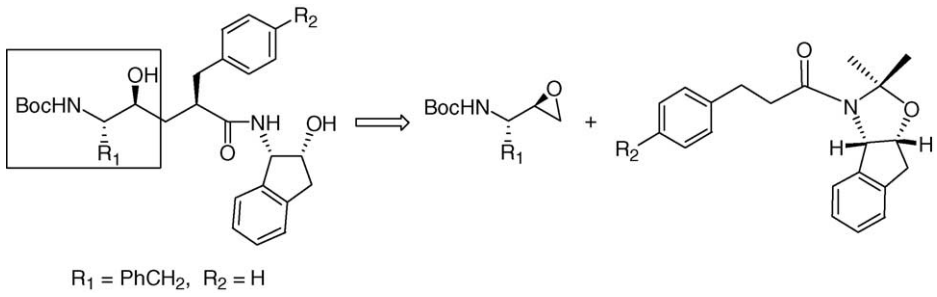
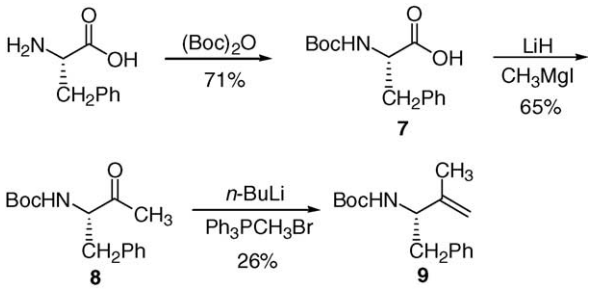


Scheme 1.

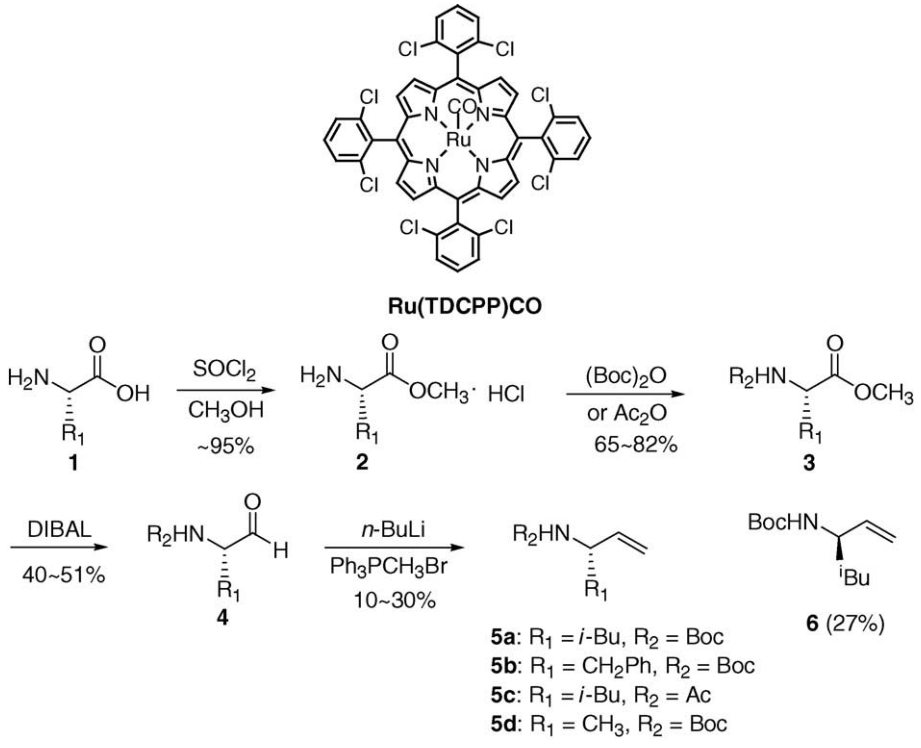


Scheme 3.

(Scheme 2). To compare the effect of substituent on the epoxidation selectivity, N-protected α-amino alkene (*S*)-**9** was prepared through the treatment of N-protected amino acid **7** with LiH and Grignard reagent to afford N-protected amino ketone **8** [6], and followed by Wittig olefination (Scheme 3).

2.2. The selective catalytic epoxidation of N-protected α-amino alkenes

It has been well established, first by Hirobe and coworkers, that substituted pyridine N-oxide such as Cl_2PyNO can efficiently epoxidize alkenes in the presence of homogeneous ruthenium porphyrin catalysts [7]. Very recently, we employed polymer-supported manganese(III) porphyrins to promote the epoxidation of cholest-5-ene derivatives in over 99% β-selectivity [8]. These results prompted us to examine the use of Cl_2PyNO in the diastereoselective epoxidation of N-protected α-amino alkenes. Our initial exploration of reaction conditions for the catalytic epoxidation focused on α-amino alkene (*S*)-**5b** in the presence of ruthenium porphyrin complex [Ru(TDCPP)CO]. Thus, it was gratifying to discover that our catalyst system effectively facilitated this epoxidation to the expected amino epoxides in high diastereoselectivity (up to 99%) in CH_2Cl_2 at 40 °C in the presence



Scheme 2.

Table 1
Catalytic epoxidation of N-protected α-amino alkene 5b with Cl_2PyNO by ruthenium porphyrin complex [Ru(TDCPP)CO][a]

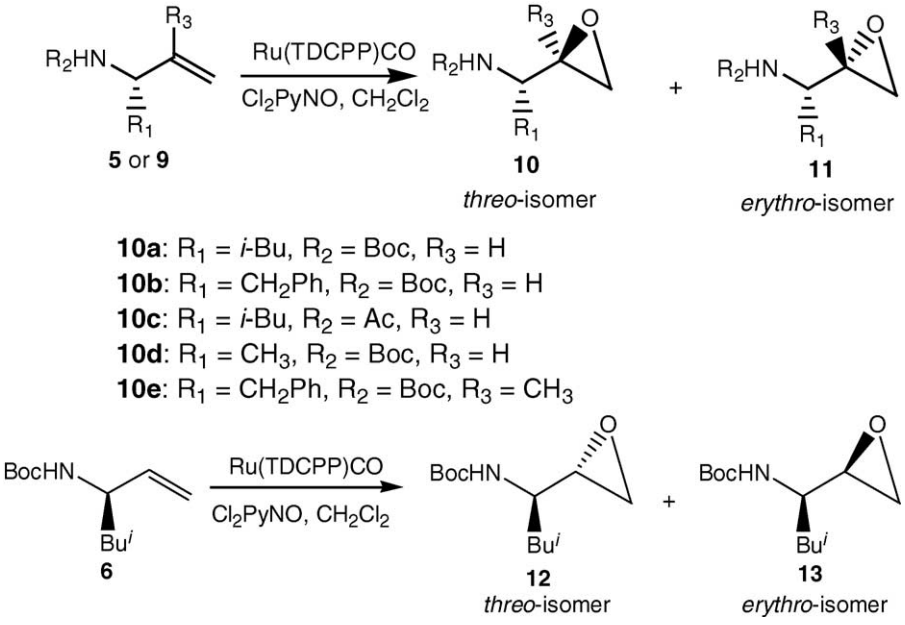
Entry	Catalyst/substrate/oxidant	Reaction time (days)	Yield (%)[b]	Selectivity (*threo*-isomer) (%)[c]
1	1:25:50	4	12	60
2	1:50:100	4	25	>99
3	1:100:200	2	26	84
4	1:100:200	3	47	94
5	1:100:200	4	32	>99
6	1:100:200	5	27	91
7	1:200:400	4	8	49

[a] All reactions were carried out in CH_2Cl_2 at 40 °C in the presence of molecular sieves (4A).
[b] Isolated yields (average of two runs) based on alkene used.
[c] Determined from integrated H_t: ($H_t + H_e$).

of 4A molecular sieves (Table 1). In principle, these products have two isomers including *threo* and *erythro* configurations (Scheme 4), which can be determined by ^{1}H NMR spectra according to the reported methods [5,9]. More interestingly, our protocol shows preference for the *threo* configuration. For this model reaction, we firstly investigated the effect of catalyst loading, and found that the loading could greatly influence the epoxidation yield and diastereoselectivity. The *threo*-selectivities were higher than 99% when 1 and 2% catalysts were employed (entries 2 and 5). However, when the catalyst loading was increased to 4% or reduced to 0.5%, the diastereoselectivities dramatically decreased from 99 to 60 and 49%, respectively (entries 1 and 7). It may be noted that 1% of catalyst loading afforded a higher yield (32%, entry 5) than 2% catalyst (25%, entry 2). Subsequently, the effect of reaction time on the epoxidation of (*S*)-**5b** was studied. We found that the diastereoselectivity appears to be sensitive to the reaction time (entries 2–5). By increasing reaction time, the reaction selectivity could be improved; however, the diastereoselectivity decreased when the reaction time was longer than 5 days.

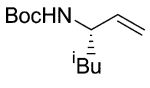
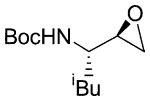
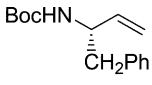
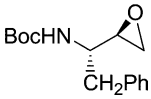
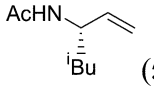
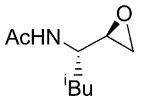
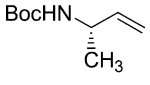
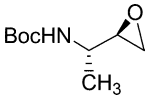
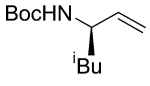
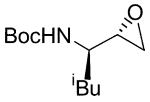
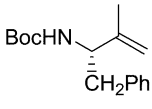
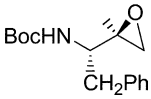
Amino epoxides are useful intermediates in the preparation of natural products, drugs and inhibitors of enzymes such as rennin and HIV protease. Under our optimized reaction conditions, we investigated the epoxidation of six α-amino alkenes derived from amino acids, and were also delighted to find that our methodology could effectively afford amino epoxides with a strong *threo* stereochemical preference (Table 2) [10]. However, these types of alkenes provided somewhat low epoxidation yields presumably because of steric hindrance of substrates. Our protocol also shows a superiority of the Boc group over the Ac group protection. When the protected group of amino group changed from Boc (**5a**) to Ac (**5c**), the *threo* selectivity decreased from 92 to 85% (entries 1 and 3). As is evident in Table 2, the substituents on side-chain of protected α-amino alkenes significantly influence the catalytic stereoselectivity. When the bulk of substituents are diminished in the order of benzyl (**5b**), *iso*-butyl (**5a**) and methyl (**5d**) group, the selectivities are 94, 92 and 87%, respectively (entries 2, 1 and 4). In addition, compared with substrate **5b**, α-amino alkene (**9**) with a methyl substituent at 2-alkene gave a lower *threo* selectivity of 75% (entries 2 and 6). For all of α-amino alkenes investigated, (*S*)-2-(*t*-Boc-amino)-1-phenylbut-3-ene (**5b**) gave the best result in 47% yield and 94% *threo*-selectivity (entry 2).

Finally, we investigated the epoxidation of α-amino alkene **6** derived from D-leucine in which chiral carbon is *R* absolute configuration (Scheme 3). More interestingly, like its enantiomeric substrate (*S*)-**5a**, the epoxidation still gave *threo* epoxide in 90% *threo*-selectivity under our standard



Scheme 4.

Table 2
Epoxidation of N-protected α-amino alkenes with Cl_2PyNO catalyzed by Ru(TDCPP)CO[a]

Entry	Substrates	Products	Yield (%)[b]	Selectivity (*threo*-isomer) (%)[c]
1	 **(5a)**		34	92
2	 **(5b)**		47	94
3	 **(5c)**		29	85
4	 **(5d)**		16	87
5	 **(6)**		20	90
6	 **(9)**		24	75

[a] All reactions were carried out in CH_2Cl_2 at 40 °C in the presence of molecular sieves (4A) for 3 days with a catalyst/substrate/oxidant ratio of 1:100:200.
[b] Isolated yields (average of two runs) based on alkene used.
[c] Determined from integrated H_t: ($H_t + H_e$).

conditions (entry 5). This product (**12**) possesses the *R*, *S* configuration, and in the case of (*S*)-**5a**, the configuration of epoxide (**10**) is the *S*, *R* configuration (Scheme 4).

In summary, we have developed a convenient method for producing *threo* amino epoxides in high diastereoselectivity (up to 99%). The catalytic protocol is facile and effective for the epoxidation of N-protected α-amino alkenes.

3. Experimental section

3.1. General

Ruthenium carbonyl, D- and L-amino acids were commercially available. Solvents were distilled before use according to standard procedures. 2,6-Dichloropyridine N-oxide [9], carbonyl ruthenium(II) 5,10,15,20-tetra(2,6-dichlorophenyl)porphyrin [11], amino acid methyl ester hydrochloride **2**, *N*-Boc-amino acid methyl ester hydrochloride **3** and L-*N*-Boc-Phe-OH 7 [12] were prepared by the reported method. ^{1}H NMR spectra were measured on a Varian INOVA-400 spectrometer (400 MHz) in $CDCl_3$. UV–Vis spectra were measured on a Shimadzu UV-240 spectrophotometer. GC–MS was recorded on Agilent machine N6890-5943. Polarimetric measurements were taken on an automatic polarimeter Perkin-Elmer-341.

3.2. Preparation of substrates

3.2.1. L-*N*-Ac-Leu-OCH_3 *3c*

To a stirred solution of L-Leu-OCH_3·HCl (8.98 g, 50.0 mmol) in dichloromethane (100 mL) in an ice-bath was added sodium hydrogen carbonate (8.82 g, 105.0 mmol). Acetic anhydride (55.0 mmol) was added dropwise. After being stirred for 2 h at room temperature, the mixture was filtered. The filtrate was washed with saturated sodium hydrogen carbonate (2 × 60 mL) and brine. The dichloromethane layer was dried over sodium sulfate and concentrated to give colorless oil **3c** (7.74 g, 83%). ^{1}H NMR ($CDCl_3$) δ 0.92–0.96 (m, 6H), 1.57–1.59 (m, 1H), 1.60–1.68 (m, 2H), 2.01 (s, 3H), 3.74 (s, 3H), 4.61–4.67 (m, 1H), 6.11–6.13 (s, 1H).

3.2.2. Protected amino aldehydes *4*

To a −78 °C stirred solution of the corresponding protected amino ester **3** (10.0 mmol) in dry tetrahydrofuran (60 mL) was added diisobutylaluminum hydride (20.0 mmol, 1 M in hexane) dropwise over 30 min. Methanol was cautiously added 30 min later, and the mixture was poured into a 0 °C stirred solution of Rochelle salt (33 mL of saturated aqueous solution diluted with 200 mL of H_2O). After being stirred for 60 min at 0 °C, the mixture was filtered through Celite, and the solids were extracted with ether (5 × 100 mL). The aqueous phase was extracted with ether (2 × 100 mL),

and combined organic phase was dried with sodium sulfate, filtered, evaporated and chromatographed on silica gel to give the aldehyde.

(*S*)-2-(*t*-Boc-amino)-4-methylpentanal (**4a**): colorless oil (yield: 44%). ^{1}H NMR ($CDCl_3$) δ 0.91–0.97 (m, 6H), 1.28–1.39 (m, 2H), 1.44 (s, 9H), 1.77–1.79 (m, 1H), 4.24 (s, 1H), 4.91 (s, 1H), 9.58 (s, 1H).

(*S*)-2-(*t*-Boc-amino)-3-phenylpropanal (**4b**): yellow solid (yield: 51%), mp: 86–89 °C. ^{1}H NMR ($CDCl_3$) δ 1.41 (s, 9H), 3.01–3.14 (m, 2H), 4.57–4.59 (m, 1H), 5.12 (s, 1H), 7.15–7.33 (m, 5H), 9.56 (s, 1H).

(*S*)-2-(Acetylamino)-4-methylpropanal (**4c**): yellow oil (yield: 43%). ^{1}H NMR ($CDCl_3$) δ 0.88–0.99 (m, 6H), 1.57–1.59 (m, 1H), 1.61–1.70 (m, 2H), 4.54–4.55 (m, 1H), 6.06 (s, 1H), 9.57 (s, 1H).

(*S*)-2-(*t*-Boc-amino)propanal (**4d**): colorless oil (yield: 40%). ^{1}H NMR ($CDCl_3$) δ 1.33–1.35 (d, 3H, J = 8 Hz), 1.46 (s, 9H), 4.22–4.26 (m, 1H), 5.12 (s, 1H), 9.57 (s, 1H).

(*R*)-2-(*t*-Boc-amino)-4-methylpentanal: colorless oil (yield: 46%). ^{1}H NMR ($CDCl_3$) δ 0.91–0.98 (m, 6H), 1.32–1.36 (m, 2H), 1.46 (s, 9H), 1.61–1.70 (m, 1H), 4.31 (s, 1H), 4.95 (s, 1H), 9.61 (s, 1H).

3.2.3. L-3-(t-Boc-amino)-4-phenylbutan-2-one **8**

A slurry of L-*N*-Boc-Phe-OH **7** (2.65 g, 10.0 mmol) and lithium hydride (179 mg, 22.5 mmol) in 100 mL of dimethoxyethane was heated to 85 °C and kept at this temperature for 4 h. Methylmagnesium iodide (50.0 mmol) diluted in 30 mL of tetrahydrofuran was added to the reaction mixture cooled in an ice bath. The reaction mixture was then heated to 65 °C and kept at this temperature for 16 h. The solution was cooled to ambient temperature and poured rapidly into 100 mL of ice-cold 3 N HCl with vigorous stirring. The solution was extracted three times with 60 mL ethyl acetate and the combined organic layers were washed with saturated sodium bicarbonate solution and brine. The ethyl acetate layer was dried over sodium sulfate and concentrated in vacuo. The product was purified by silica gel chromatography to give 81.71 g as white soild (yield: 65%, mp: 62–63 °C). ^{1}H NMR ($CDCl_3$) δ 1.42 (s, 9H), 2.12 (s, 3H), 2.96–3.12 (m, 2H), 4.52–4.57 (m, 1H), 5.12 (s, 1H), 7.15–7.33 (m, 5H). MS (EI): 263 [M^+].

3.2.4. Protected allylic amines **5**, **6** and **9**

To a −78 °C stirred suspension of methyltriphenylphosphonium bromide (7.14 g, 20.0 mmol) in dry tetrahydrofuran (100 mL) under argon was added *n*-BuLi (21.0 mmol, 2.88 M in hexane). The mixture was warmed to room temperature for 30 min. The solution was then cooled to −78 °C and added to a −78 °C stirred solution of protected amino aldehyde **4** (10.0 mmol) in tetrahydrofuran (30 mL) over the course of 3 h, quenched with water (100 mL), and extracted with hexane (4 × 100 mL). The combined organic phase was washed with brine, dried with sodium sulfate, filtered, evaporated, and chromatographed on silica gel.

(*S*)-3-(*t*-Boc-amino)-5-methylhex-1-ene (**5a**): yellow oil (yield: 29%). ^{1}H NMR ($CDCl_3$) δ 0.88–0.94 (m, 6H), 1.25–1.40 (m, 2H), 1.45 (s, 9H), 1.49–1.73 (m, 1H), 4.15 (s, 1H), 4.40 (s, 1H), 5.05–5.18 (dd, 2H, J = 8, J = 16 Hz), 5.69–5.77 (m, 1H).

(*S*)-2-(*t*-Boc-amino)-1-phenylbut-3-ene (**5b**): white solid (yield: 27%). ^{1}H NMR ($CDCl_3$) δ 1.29 (s, 9H), 2.82–2.84 (d, 2H, J = 8 Hz), 4.42 (s, 2H), 5.06–5.12 (m, 2H), 5.75–5.83 (m, 1H), 7.17–7.31 (m, 5H).

(*S*)-3-(Acetylamino)-5-methylhex-1-ene (**5c**): yellow oil (yield: 23%). ^{1}H NMR ($CDCl_3$) δ 0.92–0.97 (m, 6H), 1.35–1.39 (m, 2H), 1.60–1.70 (m, 1H), 1.99–2.02 (m, 3H), 4.49–4.56 (m, 1H), 5.06–5.19 (dd, 2H, J = 13.2, 25.6 Hz), 5.45–5.46 (d, 1H, J = 4 Hz), 5.70–5.79 (m, 1H). MS (EI): 155 [M^+].

(*S*)-3-(*t*-Boc-amino)but-1-ene (**5d**): colorless oil (yield: 10%). ^{1}H NMR ($CDCl_3$) δ 1.19–1.21 (d, 3H, J = 6.8 Hz), 1.44 (s, 9H), 4.21 (s, 1H), 4.44 (s, 1H), 5.04–5.16 (dd, 2H, J = 10.4, 17.2 Hz), 5.77–5.85 (m, 1H). MS (EI): 156 [$M - CH_3$]$^+$.

(*R*)-3-(*t*-Boc-amino)-5-methylhex-1-ene (**6**): colorless oil (yield: 25%). ^{1}H NMR ($CDCl_3$) δ 0.93–0.95 (m, 6H), 1.34 (m, 2H), 1.46 (s, 9H), 1.62–1.72 (m, 1H), 4.17 (s, 1H), 4.43 (s, 1H), 5.06–5.09 (d, 1H, J = 12.8 Hz), 5.15–5.19 (d, 1H, J = 17.2 Hz), 5.71–5.79 (m, 1H). MS (EI): 214 [$M + 1$]$^+$.

(*S*)-2-(*t*-Boc-amino)-1-phenyl-3-methylbut-3-ene (**9**): white solid (yield: 26%, mp: 80–84 °C). ^{1}H NMR ($CDCl_3$) δ 1.37 (s, 9H), 1.77 (s, 3H), 2.75–2.93 (dd, 2H, J = 20.4, 20 Hz), 4.31 (s, 1H), 4.54 (s, 1H), 4.79–4.84 (d, 2H, J = 18.8 Hz), 7.16–7.42 (m, 5H). MS (EI): 261 [M^+].

3.3. General procedure for the catalytic epoxidation of protected α-amino alkenes

In the presence of 200 mg 4A molecular sieves, a mixture of alkene (0.4 mmol), Cl_2PyNO (0.8 mmol) and Ru(TDCPP)CO (5 mg) in CH_2Cl_2 (5 mL) was stirred at 40 °C under nitrogen atmosphere. After 3 days the reaction proved complete by TLC analysis. Drying and evaporating the mixture provided the corresponding epoxides, which were examined by ^{1}H NMR to give the *threo*:*erythro* data reported in Table 2. Separation of this mixture by careful chromatography (silica gel, 8:1 petroleum ether-ethyl acetate) afforded the *threo*-isomer.

(1*R*,*S*)-[1′*S*-(*t*-Boc-amino)-3-methylbutyl]oxirane (**10a**): yellow oil. $[\alpha]_D = -10.7°$ (2.8 g/L, CH_2Cl_2). ^{1}H NMR ($CDCl_3$) δ_{threo} 0.96–1.08 (m, 6H), 1.25 (s, 2H), 1.43 (s, 9H), 1.55–1.66 (m, 1H), 2.60 (s, 1H), 2.72–2.76 (m, 1H), 2.99 (s, 1H), 3.97–3.98 (d, 1H, J = 4.4 Hz), 4.31 (s, 1H).

(1*R*,*S*)-[1′*S*-(*t*-Boc-amino)-2-phenylethyl]oxirane (**10b**): white solid. ^{1}H NMR ($CDCl_3$) δ_{threo} 1.38–1.40 (s, 9H), 2.59–3.03 (m, 5H), 4.13 (s, 1H), 4.50 (s, 1H), 7.21–7.33 (m, 5H).

(1*R*,*S*)-(1′*S*-Acetylamino-3-methylbutyl)oxirane (**10c**): yellow oil. ^{1}H NMR ($CDCl_3$) δ_{threo} 0.91–0.95 (m, 6H), 1.35–1.38 (m, 2H), 1.60–1.65 (m, 1H), 1.95–2.04 (m, 3H), 3.54–3.69 (m, 1H), 3.71–3.93 (d, 1H, J = 8.6 Hz), 4.08–4.19

(m, 1H), 4.55–4.60 (m, 1H), 5.33–5.4 (m, 1H). MS (EI): 172 $[M+1]^+$.

(1*R*,*S*)-[1′*S*-(*t*-Boc-amino)ethyl]oxirane (**10d**): white oil. ^{1}H NMR ($CDCl_3$) δ_{threo} 1.12–1.29 (m, 3H), 1.44 (s, 9H), 2.59–2.61 (m, 1H), 2.73–2.79 (m, 1H), 2.97–3.00 (dd, 1H, J = 2.4, 2.8 Hz), 3.99 (s, 1H), 4.43 (s, 1H). MS (EI): 189 $[M+2]^+$.

(1*R*,*S*)-[1′-Methyl-1′*S*-(*t*-Boc-amino)-2-phenylethyl]oxirane (**10e**): colorless oil. ^{1}H NMR ($CDCl_3$) δ_{threo} 1.33 (s, 9H), 1.67 (s, 3H), 2.52–3.08 (m, 4H), 4.05 (s, 1H), 4.45 (s, 1H), 7.01–7.46 (m, 5H). MS (EI): 277 [M^+].

(1*S*,*R*)-[1′*R*-(*t*-Boc-amino)-3-methylbutyl]oxirane (**12**): yellow oil. $[\alpha]_D = +5.1^\circ$ (8.5 g/L, CH_2Cl_2). ^{1}H NMR ($CDCl_3$) δ_{threo} 0.91–1.00 (m, 6H), 1.20 (s, 2H), 1.43 (s, 9H), 1.67–1.73 (m, 1H), 2.59 (s, 1H), 2.72–2.74 (m, 1H), 2.98–2.99 (d, 1H, J = 1.2 Hz), 3.97–3.98 (d, 1H, J = 4.4 Hz), 4.31 (s, 1H). MS (EI): 231 $[M+2]^+$.

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