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An improved preparation of epoxides from carbonyl compounds by using diiodomethane/methyllithium: synthetic applications

José M. Concellón,* Humildad Cuervo and Ramón Fernández-Fano

Departamento de Química Orgánica e Inorgánica, Facultad de Química, Universidad de Oviedo, Julián Clavería 8, 33071 Oviedo, Spain

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Abstract—Synthesis of epoxides starting from different carbonyl compounds is easily carried out by using a diiodomethane and methyllithium at 0°C and a short reaction time. Starting from α-aminoaldehydes the reaction affords amino epoxides, with high diastereoselectivity. The products were transformed into 1,3-diaminoalkan-2-ols by treatment with different amines. © 2001 Elsevier Science Ltd. All rights reserved.

1. Introduction

Transformation of carbonyl compounds into epoxides is a reaction of considerable importance, because of the fact that the epoxide obtained can be manipulated further in a variety of synthetically useful reactions. In this sense, an oxirane ring can be opened by a variety of nucleophiles, affording 1,2-difunctionalized systems which can undergo rearrangement reactions, such as chain elongation or ring expansion processes.[1]

The classical reagents to transform carbonyl compounds into epoxides are sulfur ylides.[2] Other epoxidation reagents are the α-heterosubstituted organometallic reagents,[3] including α-halogenated organometallic compounds (principally, chloromethyllithium, generated by lithiation of chloroiodomethane at −78°C).[4] In this sense, we have reported the synthesis of chiral amino epoxides with high diastereoselectivity, by treatment of α-amino aldehydes with halomethyllithium generated in situ at −78°C.[5] However, the use of α-halogenated organolithium compounds (Köbrich reagents) is limited by their thermal instability.[6] For this reason, a number of methods to carry out the addition of Köbrich reagents to carbonyl compounds at higher temperature have been described. Thus, addition of this anions with sonication at −15°C,[7] the use of phase transfer catalysis at room temperature,[8] fluoride catalysis from TMS-containing precursor at 0°C,[9] or replacement of the lithium counterion (normally present) with a variety of transition metals (chiefly, titanium, hafnium and copper) at 0°C[10] have been reported. However, to the best of our knowledge, synthesis of epoxides from carbonyl compounds by using halomethyllithium at 0°C has not been reported in the literature to date.

On the other hand, 1,3-diamines are used as catalyst and chiral auxiliaries in asymmetric synthesis[11] and these compounds (especially chiral ones) are also of great interest in medicinal chemistry, e.g. in cancer[12] or AIDS research (protease inhibitors).[13] However, although the number of reports on the preparation of 1,3-diamines[14] has grown over the last few years, this class of compounds is not receiving the attention which has been devoted to its 1,2-analogues.[15]

Here we wish to report a general, easy and simple transformation of aldehydes and ketones into epoxides using iodomethyllithium generated in situ (from diiodomethane and methyllithium) at 0°C and the transformation of the prepared amino epoxides (from α-aminoaldehydes) into the corresponding 1,3-diaminoalkan-2-ols **4**, by treatment with different amines.

2. Results and discussion

The reaction of different aldehydes or ketones **1** with iodomethyllithium (generated in situ from diiodomethane and methyllithium) at 0°C led to the expected iodinated alkoxide **2**, which undergoes spontaneous epoxidation yielding, after the usual work-up, the corresponding oxirane **3** (Scheme 1). While reactions with aldehydes were carried out using a molar ratio **1**/CH_2I_2/MeLi=1:1.5:2, in the case of ketones a higher molar ratio was necessary (**1**/CH_2I_2/MeLi=1:2:3) due to their lower reactivity in comparison to aldehydes.

Analysis of Table 1 reveals that the reaction is general: oxiranes derived from lineal, branched and cyclic aliphatic, and aromatic aldehydes and linear, cyclic aliphatic and

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Keywords: epoxides; diastereoselection; iodomethyllithium; carbonyl compounds.

* Corresponding author. Tel.: +34-8-510-3457; fax: +34-98-510-3446; e-mail: jmcg@sauron.quimica.uniovi.es

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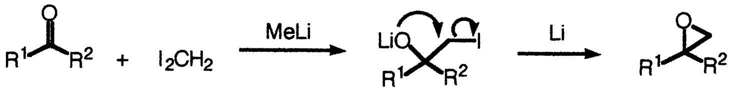


1 **2** **3**

Scheme 1. Transformation of carbonyl compounds into epoxides **3**.

Table 1. Epoxidation reaction of carbonyl compounds

Entry	Product	R^1	R^2	Yield (%)[a]
1	**3a**	Cyclohexyl	H	72
2	**3b**	Ph	H	70[b]
3	**3b**	Ph	H	83
4	**3b**	Ph	H	80[c]
5	**3c**	*p*ClPh	H	91
6	**3d**	C_7H_{15}	H	83
7	**3e**	Citronellal		95
8	**3f**	Alaninal		93[d]
9	**3g**	Phenylalaninal		91[e]
10	**3h**	C_5H_{11}	Me	45 (72)
11	**3i**	$-(CH_2)_7-$		35 (80)
12	**3j**	Ph	Me	89[b]
13	**3j**	Ph	Me	79
14	**3k**	Ph	Et	79
15	**3l**	Bn	Me	70

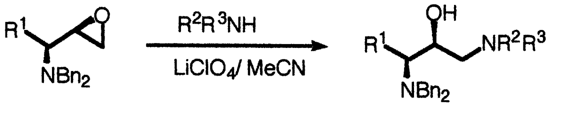
[a] Yield based on the starting carbonyl compound **1**; all epoxides were fully characterized by spectroscopy methods (IR, NMR and MS).
[b] Reaction temperature −78°C.
[c] The reaction was carried out without nitrogen atmosphere.
[d] de=90% (determined by 300 MHz ^{1}H NMR analysis of the crude product.
[e] de=94% (determined by 300 MHz ^{1}H NMR analysis of the crude product.

aromatic ketones have been obtained. In addition, under these reaction conditions, the epoxides **3** obtained from aldehydes, were obtained in high yield, similar to that obtained at −78°C (Table 1, entries 2 and 12), and the isolation of pure epoxides **3** only required the removal of the solvents (purity >95%). The yield of **3**, obtained from ketones, was lower and the epoxide appeared contaminated by small amounts of the corresponding methyl alcohol (addition of methyllithium to the ketone); consequently, purification by column chromatography was necessary.

It is noteworthy that the epoxidation reaction at 0°C take place with high diastereoselectivity. Starting from *N*,*N*-dibenzylalaninal or phenylalaninal, the corresponding *erythro* amino epoxides have been obtained with high diastereoselective excess (90 and 94%, respectively) this being similar to that obtained at −78°C (de=98 and 92%).[3] The diastereoisomeric excess was determined by using 300 MHz ^{1}H NMR and the stereochemistry of amino epoxides was established unambiguously by comparison with the same compounds obtained at −78°C.[5] The observed stereochemistry can be explained by assuming a nonchelation control model in the iodomethylation reaction of carbonyl compounds, due to the steric constraints imparted by the bulky *N*,*N*-dibenzyl protecting group.

The protease inhibitors, such as VX-478 (Vertex-Glaxo Wellcome)[16] and SC-52151 (Searle-Monsanto),[17] involving the hydroxyethylamine moiety are very promising therapeutic agents for the treatment of acquired immunodeficiency syndrome (AIDS).[18] This hydroxyethylamine moiety can be obtained by treatment of amino epoxides with different amines. For this reason and to prove the synthetic usefulness of the amino epoxides obtained, we have prepared 1,3-diaminoalkan-2-ols from **3f** and **3g**.

Treatment of a solution of *erythro* amino epoxides **3f** and **3g** in acetonitrile with different amines in the presence of lithium perchlorate[19] led to the corresponding 1,3-diaminoalkan-2-ols **4** (Scheme 2) in high yield (Table 2). The reaction was completely regioselective with the attack of the nucleophile on the less substituted oxirane carbon.



3f,3g **4**

Scheme 2. Synthesis of 1,3-diaminoalkan-2-ols **4**.

Table 2. Synthesis of 1,3-diaminoalkan-2-ols **4**

Entry	Product	R^1	NHR^2R^3	Yield (%)[a]
1	**4a**[b]	Me	$BnNH_2$	97.5
2	**4b**[c]	Bn	Et_2NH	78
3	**4c**[c]	Bn	$BnNH_2$	80

[a] Yield of **4** based on the starting aminoepoxide **3f** and **3g**.
[b] Reaction time 48 h.
[c] Reaction time 16 h.

3. Conclusion

We have described a simple and general transformation of carbonyl compounds into epoxides at 0°C, which take place in high yield and with high diastereoselectivity. We believe that the methodology described represents an important improvement in the transformation of carbonyl compounds into epoxides by using iodomethyllithium. We have also described an easy transformation of the amino epoxides, obtained by means of the described methodology, into 1,3-diaminoalkan-2-ols.

4. Experimental

4.1. General

Reactions requiring an inert atmosphere were conducted under dry nitrogen, and the glassware was oven dried (120°C). THF and ether were distilled from sodium/benzophenone ketyl immediately prior to use. All reagents were purchased from Aldrich or Merck and were used without further purification. Silica gel for flash chromatography was purchased from Scharlau or Merck (200–450 mesh), and compounds were visualized on analytical thin layer chromatograms (TLC) by UV light (254 nm). Optical rotations were measured in chloroform ^{1}H NMR spectra were recorded at 200, 300 or 400 MHz. ^{13}C NMR spectra and DEPT experiments were determined at 50 or 75 MHz. Chemical shifts are given in ppm relative to tetramethylsilane (TMS), which is used as an internal standard, and coupling constants (J) are reported in Hz. The diastereomeric excesses were obtained using ^{1}H NMR analysis and GC–MS of crude products. Only the most important IR absorptions (in cm^{-1}) and the molecular ions and/or base peaks in MS are given.

4.2. General procedure for the preparation of epoxides 3 from carbonyl compounds

To a solution of the corresponding aldehyde **1** (2.5 mmol) and diiodomethane (0.3 ml, 3.75 mmol) in THF (10 ml) was added dropwise methyllithium (3.4 ml of 1.5 M solution in diethyl ether, 5 mmol) over 5 min and under nitrogen to a 0°C. After stirring at 0°C for 30 min, the mixture was stirred for one additional hour at room temperature. The resulting mixture was treated with ice and extracted with dichloromethane (3×5 ml). The combined organic layers were dried ($NaSO_4$), filtered and the solvents were removed to provide the corresponding crude epoxides **3**. Column flash chromatography over silica gel (75/1 *n*-hexane/triethyl amine) provided pure epoxides **3**.

4.2.1. 2-Cyclohexyloxirane (3a). R_f=0.61 (hexane/AcOEt 5/1); ^{1}H NMR ($CDCl_3$, 200 MHz) δ 2.63 (2H, t, J=3.08 Hz), 2.45–2.41 (1H, m), 1.85–1.40 (5H, m), 1.20–0.94 (6H, m); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 56.6 (CH), 45.9 (CH_2), 40.2 (CH), 29.6 (CH_2), 28.7 (CH_2), 26.2 (CH_2), 25.6 (CH_2), 25.4 (CH_2); MS (EI) *m/z* (%): 126 (M^+, <1), 96 (70), 55 (100); IR (NaCl) 2924, 2852, 1260.

4.2.2. 2-Phenyloxirane (3b). R_f=0.54 (hexane/AcOEt 5/1); ^{1}H NMR ($CDCl_3$, 200 MHz) δ 7.50–7.30 (5H, m), 3.92 (1H, dd, J=2.56 and 4.10 Hz), 3.19 (1H, dd, J=4.10 and 5.39 Hz), 2.84 (1H, dd, J=2.56 and 5.39 Hz); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 137.3 (C), 128.1 (CH), 127.7 (CH), 125.1 (CH), 52.0 (CH), 50.7 (CH_2); MS (EI) *m/z* (%): 121 (M^++1, 13), 120 (M^+, 84), 89 (100), 77 (53); IR (NaCl) 3042, 2963, 2914, 1260.

4.2.3. 2-(4-Chlorophenyl)oxirane (3c). R_f=0.35 (hexane/AcOEt 10/1); ^{1}H NMR ($CDCl_3$, 200 MHz) δ 7.27–7.10 (4H, m), 3.74 (1H, dd, J=2.56 and 4.11 Hz), 3.05 (1H, dd, J=4.11 and 5.52 Hz), 2.67 (1H, dd, J=2.56 and 5.52 Hz); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 135.7 (C), 133.2 (C), 128.1 (CH), 126.4 (CH), 51.2 (CH), 50.6 (CH_2); MS (EI) *m/z* (%): 154 (M^+, 50), 119 (92), 89 (100); IR (NaCl) 3051, 2990, 2918; Anal. calcd for C_9H_7ClO: C, 62.15; H, 4.56. Found C, 61.90; H, 4.60.

4.2.4. 2-Heptyloxirane (3d). R_f=0.4 (hexane/AcOEt 10/1); ^{1}H NMR ($CDCl_3$, 200 MHz) δ 2.82–2.80 (1H, dd J=2.56 and 3.85), 2.62 (1H, dd, J=3.85 and 5.13 Hz), 2.35 (1H, dd, J=2.56 and 5.13 Hz), 1.41–1.33 (12H, m) 0.78 (3H, t, J=4.61 Hz); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 52.1 (CH), 46.8 (CH_2), 32.3 (CH_2), 31.6 (CH_2), 29.2 (CH_2), 29.0 (CH_2), 25.8 (CH_2), 22.4 (CH_2), 13.8 (CH_3); MS (EI) *m/z* (%): 142 (M^+, 8), 127 (40), 57 (100); IR (NaCl) 2927, 2856, 1265.

4.2.5. 2-(2,6-Dimethylhept-5-enyl)oxirane (3e). R_f=0.25 (hexane/AcOEt 10/1); ^{1}H NMR ($CDCl_3$, 200 MHz) δ 5.06 (1H, t, J=7.08 Hz), 2.92–2.89 (1H, m), 2.76–2.70 (1H, m), 2.44–2.38 (1H, m), 1.98–1.95 (2H, m), 1.65 (6H, s), 1.48–1.34 (5H, m), 0.98 and 0.96 (3H, 2d, J=3.08 Hz); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 131.1 (C), 124.3 (CH), 47.3 (CH), 46.8 (CH_2), 39.6 (CH_2), 37.1 (CH_2), 36.7 (CH_2), 30.9 (CH), 30.4 (CH), 25.5 (CH_3), 25.2 (CH_2), 19.7 (CH_3), 19.3 (CH_3), 17.4; IR (NaCl) 3050, 2960.

4.2.6. (1*S*)-[1′(*S*)-(Dibenzylamino)ethyl]oxirane (3f). $[\alpha]_D^{25}$=+9.3° (*c* 0.65, $CHCl_3$); R_f=0.51 (hexane/AcOEt 10/1); ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.45–7.22 (10H, m), 3.87 (2H, d, J=14.00 Hz), 3.64 (2H, d, J=14.00 Hz), 3.15–3.10 (1H, m), 2.92–2.77 (1H, m), 2.72 (1H, dd, J=4.11 and 4.87 Hz), 2.44 (1H, dd, J=2.90 and 4.80 Hz), 1.07 (3H, d, J=7.00 Hz); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 140.1 (C), 128.2 (CH), 128.1 (CH), 126.6 (CH), 55.2 (CH), 53.9 (CH_2), 53.4 (CH), 44.5 (CH_2), 8.5 (CH_3); MS (EI) *m/z* (%): 267 (M^+, 2), 224 (8), 91 (100); IR (NaCl) 3066; Anal. calcd for $C_{18}H_{21}NO$: C, 80.86, H, 7.92, N, 5.24. Found: C, 80.60, H, 7.98, N, 5.26.

4.2.7. (1*S*)-[1′(*S*)-(Dibenzylamino)-2-phenylethyl]oxirane (3g). $[\alpha]_D^{25}$=+6.5° (*c* 0.82, $CHCl_3$); R_f=0.36 (hexane/AcOEt 15/1); ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.39–7.16 (15H, m), 3.95 (2H, d, J=13.70 Hz), 3.80 (2H, d, J=13.70 Hz), 3.25–3.20 (1H, m), 3.12–3.05 (1H, m), 2.95–2.87 (2H, m), 2.85 (1H, dd, J=4.11 and 5.10 Hz), 2.56 (1H, dd, J=2.80 and 5.12 Hz); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 139.3 (C), 139.2 (C), 129.4 (CH), 128.3 (CH), 128.2 (CH), 128.0 (CH), 126.7 (CH), 126.0 (CH), 60.2 (CH), 54.1 (CH_2), 51.9 (CH), 46.1 (CH_2), 33.6 (CH_2); MS (EI) *m/z* (%): 344 (M^++1, <1), 343 (M^+, <1), 91 (100); IR (NaCl) 3051; Anal. calcd for $C_{24}H_{25}NO$: C, 83.93, H, 7.34, N, 4.08. Found: C, 83.71, H, 7.40, N, 4.07.

4.2.8. 2-Methyl-2-pentyloxirane (3h). R_f=0.26 (hexane/AcOEt 10/1); ^{1}H NMR ($CDCl_3$, 200 MHz) δ 2.52 (1H, d, J=5.0 Hz), 2.48 (1H, d, J=5.0 Hz) 1.48–1.10 (8H, m), 1.22 (3H, s), 0.81 (3H, t, J=6.41 Hz); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 56.8 (C), 53.6 (CH_2), 36.5 (CH_2), 31.6 (CH_2), 24.7 (CH_2), 22.4 (CH_2), 20.6 (CH_3), 13.7 (CH_3); MS (EI) *m/z* (%): 128 (M^+, 4), 123 (42), 41 (100); IR (NaCl) 2930, 2857, 1260.

4.2.9. 1-Oxa-spiro[2.7]decane (3i). R_f=0.24 (hexane/AcOEt 10/1); ^{1}H NMR ($CDCl_3$, 200 MHz) δ 2.61 (2H, s), 1.50–1.11 (14H, m); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 59.6 (C), 55.3 (CH_2), 34.2 (CH_2), 26.1 (CH_2), 25.0 (CH_2), 23.9 (CH_2); MS (EI) *m/z*(%): 140 (M^+, 10); IR (NaCl) 2925, 2867, 1262.

4.2.10. 2-Methyl-2-phenyloxirane (3j). R_f=0.22 (hexane/AcOEt 10/1); ^{1}H NMR ($CDCl_3$, 200 MHz) δ 7.42–7.34 (5H, m), 2.99 (1H, d, J=5.39), 2.82 (1H, d, J=5.39 Hz), 1.76 (3H, s); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 140.8 (C), 127.9 (CH), 127.1 (CH), 124.9 (CH), 56.6 (CH_2), 56.3 (C), 21.4 (CH_3); IR (NaCl) 3050, 3048, 2972, 2924, 1266; Anal. calcd for $C_9H_{10}O$: C, 80.56, H, 7.51. Found: C, 80.34, H, 7.62.

4.2.11. 2-Ethyl-2-phenyloxirane (3k). R_f=0.5 (hexane/AcOEt 10/1); ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.39–7.28 (5H, m), 2.98 (1H, d, *J*=5.39 Hz), 2.74 (1H, d, *J*=5.39 Hz), 1.83 (2H, q, *J*=7.44 Hz), 0.97 (3H, t, *J*=7.44 Hz); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 139.8 (C), 128.1 (CH), 127.9 (CH), 127.2 (CH), 125.8 (CH), 60.8 (C), 55.2 (CH_2), 28.1 (CH_2), 8.9 (CH_3); MS (EI) *m/z* (%): 148 (M^+, 22), 119 (58), 84 (100); IR (NaCl) 3053, 3051, 2980, 2940, 1260; Anal. calcd for $C_{10}H_{12}O$: C, 81.04, H, 8.16. Found: C, 80.84, H, 8.22.

4.2.12. 2-Benzyl-2-methyloxirane (3l). R_f=0.45 (hexane/AcOEt 10/1); ^{1}H NMR ($CDCl_3$, 200 MHz) δ 7.32–7.26 (5H, m), 2.89 (1H, d, *J*=4.87 Hz), 2.69 (1H, d, *J*=4.87 Hz), 1.52–1.30 (2H, m), 1.38 (3H, s); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 137.0 (C), 129.3 (CH), 128.1 (CH), 126.3 (CH), 57.0 (C), 53.1 (CH_2), 42.8 (CH_2), 20.6 (CH_3); MS (EI) *m/z* (%): 148 (M^+, <1), 106 (65), 91 (77), 59 (100); IR (NaCl) 3040, 2988, 1264; Anal. calcd for $C_{10}H_{12}O$: C, 81.04, H, 8.16. Found: C, 80.80, H, 8.10.

4.3. General procedure for the preparation of 1,3-diaminoalkan-2-ols 4 from amino epoxides 3

To a room temperature solution of the corresponding aminoepoxide **3f** or **3g** (3 mmol) in acetonitrile (10 ml) was added lithium perchlorate (0.32 g, 3 mmol) and the corresponding amine (3.6 mmol) under nitrogen. After stirring at room temperature for 16 or 48 h (see Table 1), the resulting mixture was treated with H_2O (2 ml) and extracted with dichloromethane (3×5 ml). The combined organic layers were dried ($NaSO_4$), filtered and the solvents were removed to provide, the corresponding crude 1,3-diaminoalkan-2-ols **4**. The excess of amine was distilled at reduced pressure providing pure 1,3-diaminoalkan-2-ols **4** (purity >95%).

4.3.1. (2*R*,3*S*)-3-Dibenzylamino-1-benzylamino-butan-2-ol (4a). $[\alpha]_D^{25}=+11.6°$ (*c* 0.76, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 200 MHz) δ 7.28–7.41 (15H, m), 3.71–3.93 (1H, m), 3.75 (2H, d, *J*=13.1 Hz), 3.41 (2H, d, *J*=13.1 Hz) 3.36–2.97 (2H, m), 2.68–2.61 (2H, m), 2.58–2.42 (1H, m), 1.24 (3H, d, *J*=6.5 Hz); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 139.6 (C), 128.0 (C), 127.9 (CH), 127.8 (CH), 127.7 (CH), 126.6 (CH), 126.5 (CH), 126.4 (CH), 70.8 (CH), 55.4 (CH), 53.6 (CH_2), 53.2 (CH_2), 51.8 (CH_2), 8.0 (CH_3); Anal. calcd for $C_{25}H_{50}N_2O$: C, 80.17, H, 8.07, N, 7.48. Found: C, 79.88, H, 7.40, N, 4.27.

4.3.2. (2*R*,3*S*)-3-Dibenzylamino-1-diethylamino-4-phenylbutan-2-ol (4b). $[\alpha]_D^{25}=+13.6°$ (*c* 0.51, $CHCl_3$); R_f=0.45 (AcOEt); ^{1}H NMR ($CDCl_3$, 200 MHz) δ 7.24–7.40 (15H, m), 4.01–4.10 (1H, m), 3.85 (2H, d, *J*=14.3 Hz), 3.75 (2H, d, *J*=14.3 Hz) 3.02–3.22 (2H, m), 2.88–2.95 (1H, m), 2.44–2.79 (5H, m), 2.18 (1H, dd, *J*=10.8, 12.4 Hz), 1.07 (6H, t, *J*=7.3 Hz);^{13}C NMR ($CDCl_3$, 50 MHz) δ 141.4 (C), 140.0 (C), 129.6 (CH), 128.8 (CH), 128.6 (CH), 127.9 (CH), 126.5 (CH), 125.5 (CH), 65.6 (CH), 62.3 (CH), 57.7 (CH_2), 54.3 (CH_2), 46.7(CH_2), 32.5 (CH_2), 11.9 (CH_3); MS (EI) *m/z* (%): 401 (M^+, <1), 91 (100); IR (NaCl) 3420. Anal. calcd for $C_{28}H_{36}N_2O$: C, 80.73, H, 8.71, N, 6.72. Found: C, 80.21, H, 8.56, N, 6.73.

4.3.3. (2*R*,3*S*)-3-Dibenzylamino-1-benzylamino-4-phenylbutan-2-ol (4c). $[\alpha]_D^{25}=-10.7°$ (*c* 1.00, $CHCl_3$); $R_{f>}$=0.33 (AcOEt/hexane 2/1); ^{1}H NMR ($CDCl_3$, 200 MHz) δ 7.36–7.15 (m, 20H), 4.01–3.84 (m, 1H), 3.77 (s, 2H), 3.68 (d, 2H, *J*=6.4 Hz), 3.10–2.81 (m, 5H), 2.62–2.52 (m, 2H); ^{13}C NMR ($CDCl_3$, 50 MHz) δ 142.9 (C), 141,3 (C), 139.7 (C), 129.4 (CH), 128.3 (CH), 127.9 (CH), 127.1 (CH), 126.9 (CH), 125.6 (CH), 69.3 (CH), 61.9 (CH), 54.4 (CH_2), 53.2 (CH_2), 52.1 (CH_2), 32.5 (CH_2); IR (NaCl) 3356, 3061, 3026, 2926, 1494, 1453: Anal. calcd for $C_{31}H_{54}N_2O$: C, 82.63, H, 7.61, N, 6.22. Found: C, 82.45, H, 7.49, N, 6.21.

Acknowledgements

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