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Synthesis of Enantiopure (α*S*,*βS*)- or (α*R*,*βS*)-*β*-Amino Alcohols by Complete Regioselective Opening of Aminoepoxides by Organolithium Reagents $LiAlH_4$ or $LiAlD_4$

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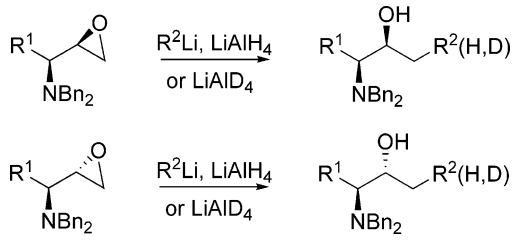

The reaction of chiral (2*R*,1′*S*)- or (2*S*,1′*S*)-2-(1-aminoalkyl)epoxides, **1** or **2** with a variety of organolithium compounds to obtain the corresponding (α*S*,*βS*)- or (α*R*,*βS*)- *β*-amino alcohols in enantiopure form is reported. In both cases, the opening of the oxirane ring at C-3 proceeded with total regioselectivity. Moreover, the ring opening of aminoepoxides **1** or **2** by hydride (utilizing $LiAlH_4$) to obtain the corresponding (2*S*,3*S*)- or (2*R*,3*S*)-3-aminoalkan-2-ols is also described. The reaction of **1** or **2** with $LiAlD_4$ in place of $LiAlH_4$ gave the corresponding (2*S*,3*S*)- or (2*R*,3*S*)-3-amino-1-deuterioalkan-2-ols.

Introduction

Enantiopure *β*-aminoalkanols are important building blocks and have been used to prepare a large number of biologically active natural and synthetic compounds,[1] including unnatural amino acids.[2] The former compounds have also been used as chiral auxiliaries for asymmetric synthesis.[3] Consequently, a large number of syntheses of enantiopure *β*-amino alcohols have been published. The most common and practical method for the synthesis of these compounds was the direct aminolysis of epoxides.[4] The opening of oxiranes by amines was limited by the high temperature, long reaction time, and excess of amine required. In addition, often the method failed when poorly nucleophilic amines or sterically crowded amines or epoxides were used. Finally, the total control of regioselectivity of the ring opening was, generally, unresolved. For this reason, a general synthesis of enantiopure *β*-amino alcohols with complete selectivity, in which several enantiopure diastereoisomers could be available, would be still desirable.

Previously, we reported the efficient synthesis of both enantiopure (2*R*,1′*S*)- or (2*S*,1′*S*)-2-(1-aminoalkyl)epoxides **1** or **2** by total stereoselective reduction of enantiopure α-amino-α′-chloroketones with $LiAlH_4$ or by highly stereoselective addition reaction of iodomethyllithium to α-aminoaldehydes, respectively.[5] Building on these results, we described, more recently,

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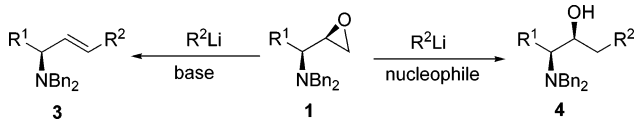

FIGURE 1.

the highly regioselective ring opening of these aminoepoxides with ketones,[6] nitriles,[7] and carboxylic acids.[8]

Very recently, we reported the transformation of aminoepoxides **1** and **2** into enantiopure allylamines **3** by reaction with various organolithium compouds.[9] To investigate the scope of the synthetic applications of the reaction between the same enantiopure aminoepoxides **1** and **2** and organolithium compounds, we report herein the nucleophilic opening of the oxirane ring of **1** or **2** with a variety of organolithium compounds, under different reaction conditions to those previously reported in the synthesis of allylamines.[9] So, we describe the reaction conditions to obtain enantiopure (α*S*,β*S*)- or (α*R*,β*S*)-β-amino alcohols **4** or **5** (instead of the allylamines previously reported) by reaction of various organolithium compounds with compounds **1** or **2**. The ring opening at C-3 proceeded with total regioselectivity, and β-amino alcohols **4** or **5** were readily available, in enantiopure form. To the best of our knowledge, only one example of the ring opening of aminoepoxides **1** or **2** with organolithium compounds was previously described.[10] Moreover, the ring opening of aminoepoxides **1** or **2** by $LiAlH_4$ to obtain the corresponding (2*S*,3*S*)- or (2*R*,3*S*)-3-aminoalkan-2-ols **6** or **7** is also reported. When $LiAlD_4$ was used instead of $LiAlH_4$, the corresponding deuterated amino alcohols **8** and **9** were obtained.

Results and Discussion

Synthesis of (α*S*,β*S*)- or (α*R*,β*S*)-β-Amino Alcohols 4 or 5 Using Organolithium Compounds. In our recent paper,[9] in which we described the synthesis of allylamines **3** by reaction of aminoepoxides **1** with organolithium compounds, the transformation was promoted by the abstraction of a proton of the oxirane ring by the organolithium compound because of its strong basic character. These previous results prompted us to find other different reaction conditions in order to use the organolithium compounds such as nucleophiles instead of bases, so that we could perform the selective ring opening of compounds **1** to obtain the corresponding amino alcohols **4** instead of the allylamines **3** (Figure 1).

Previously, in some of the synthesis of allylamines we detected the formation of β-amino alcohols **4**, as a minor reaction product, when less reactive organolithium compounds were used, such as phenyllithium or methyllithium. This inconvenience was then overcome, and the allylamines were isolated as the only product by performing the transformation with longer reaction times. On the basis of these results, and to find the reaction conditions to obtain β-amino alcohols **4** as only product, our initial attempts were carried out utilizing *syn*-aminoepoxides **1** and methyllithium and with short reaction times. Thus, treatment of a solution of aminoepoxides **1a**−**c** derived from alanine, leucine, and phenylalanine (R^1 = Me, *i*-Bu, and Bn) in THF with methyllithium (3 equiv) at 0 °C with short reaction time (30 min) provided a 3:1 mixture of the corresponding allylamine **3** and the β-amino alcohol **4**. No β-amino alcohols were obtained, as only product, by using shorter reaction times.

SCHEME 1. Synthesis of (α*S*,β*S*)-β-Amino Alcohol 4

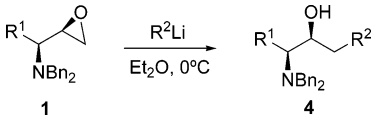

TABLE 1. Synthesis of (α*S*,β*S*)-β-Amino Alcohol 4

entry	4	R^1	R^2	yield (%)[a]
1	**4a**	Me	Me	78
2	**4b**	Me	Allyl	80
3	**4c**	Me	Ph	72
4	**4d**	*i*-Bu	Me	71
5	**4e**	*i*-Bu	Allyl	84
6	**4f**	*i*-Bu	Ph	76
7	**4g**	Bn	Me	70
8	**4h**	Bn	Allyl	89
9	**4i**	Bn	Ph	78

[a] Isolated yields after column chromatography based on the starting aminoepoxide **1**.

Taking into account the influence of the solvent in the nucleophilicity and basicity of the organolithium compounds,[11] we decided to perform the reaction using diethyl ether instead of THF. Thus, treatment of a solution of aminoepoxides **1** in diethyl ether with methyl, allyl, or phenyllithium at 0 °C afforded, after hydrolysis, the corresponding (α*S*,β*S*)-β-amino alcohol **4** in high yield (Scheme 1 and Table 1).

The reaction with allyllithium could also be performed in THF instead of Et_2O obtaining the corresponding amino alcohol **4**, as the sole product, in similar yields. This result could be obtained as consequence of the higher nucleophilicity of this organolithiun compound.

When aminoepoxides **1** were treated with other organolithium reagents, such as *i*-propyl, *n*-butyl, *sec*-butyl, or *tert*-butyl in diethyl ether, no β-amino alcohols were obtained, and the corresponding allylamines were the only isolated product.

The reaction of aminoepoxides **1** derived from alanine, leucin, and phenylalanine with these organolithium reagents was totally regioselective. So, no regioisomers were observed by ^1H and ^{13}C NMR analysis (300 MHz) of crude reaction products **4**, within the limits of NMR assay. In addition, ^1H and ^{13}C NMR spectra of compounds **4** showed the opening of the oxirane at the C-3 position. The preservation of the absolute configuration of the oxirane in the course of the ring opening and, consequently, the structure of compounds **4** has been proposed on the basis of previous reactions of aminoepoxides **1** with ketones,[6] nitriles,[7] and carboxylic acids[8] and on the single-crystal X-ray analysis of **5i** (see below).

To extend the scope of this reaction, we performed the reaction of *anti*-aminoepoxides **2** with the same organolithium

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(10) For the only example of the reaction of an organolithium compound with aminoepoxide, see the following: Mena, M.; Bonjoch, J. *Tetrahedron* **2005**, *61*, 8264−8270.

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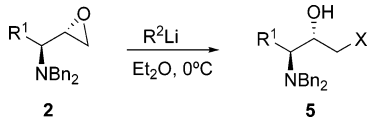
SCHEME 2. Synthesis of (α*R*,*βS*)-*β*-Amino Alcohol 5



2 **5**

TABLE 2. Synthesis of (α*R*,*βS*)-*β*-Amino Alcohol 5

entry	5	R^1	R^2	de (%)[a]	yield (%)[b]
1	**5a**	Me	Me	>98 (>98)	80
2	**5b**	Me	allyl	>98 (>98)	79
3	**5c**	Me	Ph	>98 (>98)	72
4	**5d**	*i*-Bu	Me	89 (91)	68
5	**5e**	*i*-Bu	allyl	90 (91)	75
6	**5f**	*i*-Bu	Ph	91 (91)	70
7	**5g**	Bn	Me	91 (92)	69
8	**5h**	Bn	allyl	92 (92)	80
9	**5i**	Bn	Ph	91 (92)	77

[a] Diastereoisomeric excess determined by ^{1}H NMR analysis of the crude products **5**; de of the starting aminoepoxides **2** is given in parentheses [b] Isolated yields after column chromatography based on the starting aminoepoxide **2**.

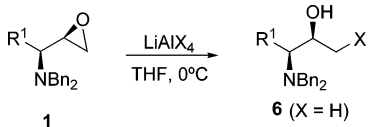
compounds and under the same reaction conditions (Scheme 2, Table 2). In all cases, the corresponding (α*R*,*βS*)-*β*-amino alcohols **5** were obtained in good yields, with high selectivity, and no important differences in the yields were observed from aminoepoxide **1** or **2**.

The selectivity of the reaction was also determined by ^{1}H NMR spectroscopy (300 MHz) of the crude mixture of products, showing a mixture of diastereoisomers **5** in the same relationship as the starting aminoepoxides **2**. The synthesis of *β*-amino alcohols **5** with the same diastereoisomeric excess (de) as the starting aminoepoxides **2**[5] was an indirect support of the total selectivity of the ring opening reaction. After purification of compounds **5** by column chromatography, the major diastereoisomer was isolated as a single stereoisomer. The structure of compounds **5** and, consequently, the absolute configuration was established by single-crystal X-ray analysis of **5i**.[12] The structure of the other *β*-amino alcohols **5** as depicted in Scheme 4 was assigned by analogy. The X-ray analysis of **5i**, confirmed that the ring opening of the oxirane took place at C-3, the absolute configuration of the oxirane being preserved. In turn, the single-crystal X-ray analysis of **5i** confirmed the structure of amino alcohols **4**, as depicted in Scheme 1. Therefore, the epoxides **1** or **2** can be opened with total regioselectivity by the organolithium reagents investigated to give the (*S*,*S*)- or (*R*,*S*)-*β*-amino alcohols **4** or **5**, exclusively.

Preparation of (2*S*,3*S*)- or (2*R*,3*S*)-3-Aminoalkan-2-ols 6 or 7 by Using $LiAlH_4$. The reaction of a solution of aminoepoxides **1** or **2** in THF with $LiAlH_4$ (1.5 equiv.) afforded, after hydrolysis, the corresponding (2*S*,3*S*)- or (2*R*,3*S*)-3-aminoalkan-2-ols **6** or **7** in high yield (>80%) (Schemes 3 and 4 and Tables 3 and 4). Similarly to the synthesis of **4** or **5**, the reaction of **1** or **2** with $LiAlH_4$ was not sensitive to the different stereochemistry and structure of the starting epoxides, and similar yields of **6** or **7** were obtained in all cases.

The reaction of aminoepoxides **1** and **2** with $LiAlH_4$ was also totally regioselective, and no regioisomers or byproducts were observed by ^{1}H and ^{13}C NMR analysis (300 MHz) of crude

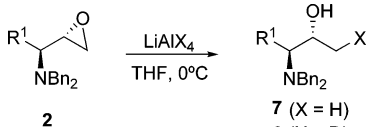
SCHEME 3. Synthesis of (*S*,*S*)-*β*-Amino Alcohols 6 or 8



1

6 (X = H)
8 (X = D)

SCHEME 4. Synthesis of (*R*,*S*)-*β*-Amino Alcohols 7 or 9



2

7 (X = H)
9 (X = D)

TABLE 3. Synthesis of (*S*,*S*)-*β*-Amino Alcohols 6 or 8

entry	compound	R^1	yield (%)[a]
1	**6a**	Me	80
2	**6b**	*i*-Bu	81
3	**6c**	Bn	90
4	**8a**	Me	82
5	**8c**	Bn	84

[a] Isolated yields after column chromatography based on the starting amino epoxide **1**.

TABLE 4. Synthesis of (*R*,*S*)-*β*-Amino Alcohols 7 or 9

entry	compound	R^1	de (%)[a]	yield (%)[b]
6	**7a**	Me	>98 (>98)	82
7	**7b**	*i*-Bu	90 (91)	85
8	**7c**	Bn	91 (92)	91
9	**9a**	Me	>98 (>98)	81
10	**9c**	Bn	90 (92)	83

[a] Diastereoisomeric excess determined by ^{1}H NMR analysis of the crude products **7** or **9**; de of the starting aminoepoxides **2** is given in parentheses. [b] Isolated yields after column chromatography based on the starting aminoepoxide **2**.

reaction products after column chromatography. Synthesis of compounds **6** or **7** previously reported, afforded a mixture of diastereoisomers **6**/**7**[13] or took place in lower yields.[14] The structure of *β*-amino alcohols **6** or **7** (opening of the oxirane at C-3) was established on the basis of their ^{1}H and ^{13}C NMR data and by comparison with the ^{1}H or ^{13}C NMR spectroscopic data of the compounds **6a**−**c** and **7b**,**c**, previously prepared by other methods.[14] The regiochemistry of the reaction was in accordance with the observed results in the ring opening of compounds **1** or **2** when different nucleophiles were used.[6−8]

In view of the utility of isotopically labeled compounds in establishing the mechanisms of organic reactions and of the biosynthesis of many natural compounds,[15] we applied this method to obtain deuterated *β*-amino alcohols **8** or **9** by using $LiAlD_4$ instead of $LiAlH_4$.

No differences were observed during the course of the reaction or on the outcome of the reaction, when aminoepoxides **1** or **2** were treated with $LiAlD_4$ instead of $LiAlH_4$ under the same reaction conditions. In this case, the corresponding (2*S*,3*S*)- or (2*R*,3*S*)-3-amino-1-deuterioalkan-2-ols **8** or **9**, respectively,

(12) CCDC-602397 (5i) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc-.cam.ac.uk/conts/retrieving.html (or the Cambridge Data Centre, 12, Union Road, Cambridge CB21EZ, U.K. Fax: (+44)1223-336.033; or deposit@ccdc.cam.ac.uk).

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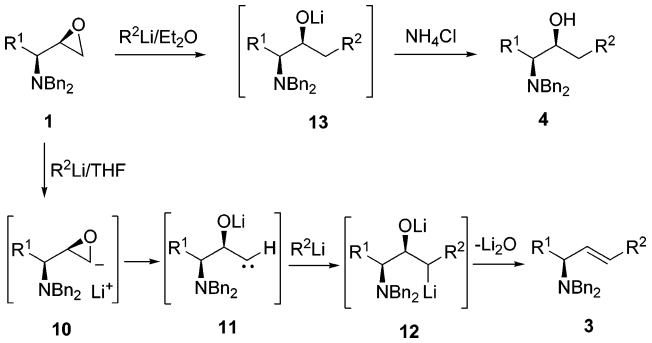
SCHEME 5. Mechanism Proposed



were obtained in high yield as the only product. Therefore, isotopically labeled with deuterium β-amino alcohols **8** or **9** were readily available from aminoepoxides **1** or **2**.

Scheme 5 shows both mechanisms proposed to explain the transformation of aminoepoxides **1** into amino alcohols **4** or into allylamines **3**. Thus, the synthesis of allylamines **3** took place by reaction of compounds **1** with 2 equiv of organolithium compound in THF. The first equivalent of the organolithium compound abstracted a proton at the less hindered side of the oxirane ring. Then, the generated oxiranyl anion intermediate **10** suffered an α-elimination affording an α-alkoxycarbenoid intermediate **11**, which reacted with the second equivalent of the organolithium compounds to give a dianion **12**. Elimination of lithium oxide from the dianion afforded allylamines **3**. An indirect support for this mechanism was provided by the isolation of a 1:1 mixture of the starting aminoepoxide and the corresponding allylamine when the reaction was carried out by using only 1 equiv of organolithium compound.

Alternatively, when the reaction of **1** was carried out with organolithium compounds in Et_2O, a ring opening at the less hindered side C-3 by nucleophilic attack of the organolithium compound would occur affording the intermediate **13**, which could be hydrolyzed to give the corresponding (α*S*,β*S*)-β-amino alcohols **3**. The total regioselectivity can be explained on the basis of steric grounds. The nucleophilic attack took place at the less hindered side of the oxirane and, moreover, a bulky substituent (dibenzylamino group) is attached at the α-carbon of the substituent alkyl group of the oxirane ring. This observed regiochemistry was in accordance to that observed in other ring opening processes of compounds **1** by different nucleophiles, such as ketones,[6] nitriles,[7] and carboxylic acids.[8,16]

The different reactivity, of aminoepoxides **1** with organolithium reagents depending on which solvent was used (Et_2O or THF) could be explained taking into account that in THF, which is a more strongly coordinating solvent than Et_2O, an enhancement of the basicity of the organolithium reagent is produced. So, the organolithium reagent in THF produced the lithiation of the oxirane ring[9] instead the ring opening which took place in Et_2O.

Similarly, when the reaction of **1** was carried out with $LiAlH_4$ or $LiAlD_4$ the nucleophilic attack of the hydride or deuteride at C-3 of the oxirane afforded the corresponding alcoholate (Scheme 5, **13**, R^2 = H or D), which (after treatment with an aqueous saturated solution of NH_4Cl) afforded enantiopure 3-aminoalkan-2-ols **6** or 3-amino-1-deuterioalkan-2-ols **8**.

(16) These previous ring openings of the aminoepoxides 1 or 2 with other nucleophiles (ketones, nitriles, and carboxylic acids) were carried out in the presence of $BF_3{\cdot}Et_2O$.

A similar mechanism could explain the reaction of aminoepoxides **2** with the organolithium compounds, hydride or deuteride to afford, after hydrolysis, amino alcohols **5**, **7**, or **9**.

Conclusions

We have reported the reaction conditions to transform the chiral (2*R*,1′*S*)- or (2*S*,1′*S*)-2-(1-aminoalkyl)epoxides **1** or **2** into enantiopure (α*S*,β*S*)- or (α*R*,β*S*)-β-amino alcohols **4** or **5** by a reaction with a variety of organolithium compounds in diethyl ether. The opening of the oxirane ring at C-3 proceeded with total regioselectivity. Moreover, the ring opening by hydride (using $LiAlH_4$) of aminoepoxides **1** or **2** to obtain the corresponding (2*S*,3*S*)- or (2*R*,3*S*)-3-aminoalkan-2-ols **6** and **7** is also reported. The reaction of **1** or **2** with $LiAlD_4$ instead of $LiAlH_4$ afforded the corresponding deuterated β-amino alcohols **8** or **9**.

Experimental Section

General Procedure of Synthesis of 4 and 5. To a stirred solution of the corresponding aminoepoxide **1** or **2** (0.2 mmol) in Et_2O (1 mL), the corresponding organolithium (3 equiv) was added at 0 °C. After the mixture was stirred at this temperature for 6 h, an aqueous saturated solution of NH_4Cl (5 mL) was added, and the mixture was stirred at room temperature for 5 min. Then, the aqueous phase was extracted with diethyl ether (3 × 5 mL), and the combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated in vacuo. Flash column chromatography on silica gel (hexane/EtOAc 20:1) provided pure compounds **4** or **5**.

(2*S*,3*S*)-2-(Dibenzylamino)pentan-3-ol (4a): colorless oil. $[\alpha]^{25}_D$ = +42.5 (*c* 1.77, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.38−7.25 (m, 10 H), 4.47 (br s, 1 H), 3.88 (d, *J* = 13.3 Hz, 2 H), 3.46 (ddd, *J* = 9.4, 8.3, 2.7 Hz, 1 H), 3.35 (d, *J* = 13.3 Hz, 2 H), 2.61 (dq, *J* = 9.4, 6.8 Hz, 1 H), 1.59 (ddq, *J* = 14.0, 7.2, 2.7 Hz, 1 H), 1.24−1.10 (m, 1 H), 1.05 (d, *J* = 6.8 Hz, 3 H), 0.98 (t, *J* = 7.2 Hz, 3 H). ^{13}C NMR (50 MHz, $CDCl_3$): δ 138.8 (2 × C), 128.9 (4 × CH), 128.4 (4 × CH), 127.1 (2 × CH), 71.8 (CH), 57.9 (CH), 53.1 (2 × CH_2), 26.3 (CH_2), 9.9 (CH_3), 7.9 (CH_3). MS (70 eV, EI) *m*/*z* (%): 265 ($M^+ - H_2O$, <1), 224 (70), 91 (100). HRMS (70 eV): calcd for $C_{19}H_{23}N$ ($M^+ - H_2O$), 265.1830; found, 265.1836. IR (neat): 3422, 2925, 2351, 1604, 1496, 1455, 1377 cm^{-1}. R_f = 0.37 (hexane/EtOAc 10:1). Anal. Calcd for $C_{19}H_{25}NO$: C, 80.52; H, 8.89; N, 4.94; O, 5.65. Found: C, 80.65; H, 8.97; N, 4.98; O, 5.60.

(2*S*,3*S*)-2-(Dibenzylamino)hept-6-en-3-ol (4b): colorless oil. $[\alpha]^{25}_D$ = +50.4 (*c* 1.44, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.37−7.27 (m, 10 H), 5.85 (ddt, *J* = 17.0, 10.2, 6.6 Hz, 1 H), 5.07−5.00 (m, 1 H), 4.98−4.94 (m, 1 H), 4.51 (br s, 1 H), 3.86 (d, *J* = 13.3 Hz, 2 H), 3.52 (dt, *J* = 9.4, 2.5 Hz, 1 H), 3.34 (d, *J* = 13.3 Hz, 2 H), 2.60 (dq, *J* = 9.4, 6.7 Hz, 1 H), 2.36−2.24 (m, 1 H), 2.21−2.07 (m, 1 H), 1.64−1.53 (m, 1 H), 1.33−1.22 (m, 1 H), 1.05 (d, *J* = 6.7 Hz, 3 H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 138.8 (2 × C), 138.7 (CH), 128.9 (4 × CH), 128.4 (4 × CH), 127.1 (2 × CH), 114.3 (CH_2), 70.0 (CH), 58.2 (CH), 53.1 (2 × CH_2), 33.0 (CH_2), 29.9 (CH_2), 7.9 (CH_3). MS (70 eV, EI) *m*/*z* (%): 309 (M^+, <1), 225 (100), 91 (13). HRMS (70 eV): calcd for $C_{21}H_{27}NO$ (M^+), 309.2093; found, 309.2080. IR (neat): 3418, 2935, 2398, 1640, 1603, 1495, 1453, 1414, 1380 cm^{-1}. R_f = 0.39 (hexane/EtOAc 10:1). Anal. Calcd for $C_{21}H_{27}NO$: C, 81.51; H, 8.79; N, 4.53; O, 5.17. Found: C, 81.37; H, 8.88; N, 4.58; O, 5.11.

(2*S*,3*S*)-3-(Dibenzylamino)-1-phenylbutan-2-ol (4c): colorless oil. $[\alpha]^{25}_D$ = +1.7 (*c* 1.64, $CHCl_3$). ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.34−7.25 (m, 15 H), 4.62 (br s, 1 H), 3.88 (d, *J* = 13.3 Hz, 2 H), 3.78 (ddd, *J* = 9.0, 8.6, 2.7 Hz, 1 H), 3.36 (d, *J* = 13.3 Hz, 2 H), 2.89 (dd, d, *J* = 13.7, 2.7 Hz, 1 H), 2.68 (dq, *J* = 9.0, 6.6 Hz, 1 H), 2.48 (dd, *J* = 13.7, 9.0 Hz, 1 H), 1.14 (d, *J* = 6.6 Hz, 3 H).

^{13}C NMR (50 MHz, $CDCl_3$): δ 138.6 (3 × C), 129.2 (2 × CH), 128.9 (4 × CH), 128.4 (4 × CH), 128.1 (2 × CH), 127.1 (2 × CH), 125.9 (CH), 71.8 (CH), 57.9 (CH), 53.1 (2 × CH_2), 40.0 (CH_2), 8.2 (CH_3). MS (70 eV, EI) *m*/*z* (%): 345 (M^+, <1), 224 (100), 181 (14), 91 (42), 69 (16). HRMS (70 eV): calcd for $C_{24}H_{27}NO$ (M^+), 345.2093; found, 345.2100. IR (neat): 3397, 3028, 2363, 1603, 1495, 1454, 1378 cm^{-1}. R_f = 0.34 (hexane/EtOAc 10:1). Anal. Calcd for $C_{24}H_{27}NO$: C, 83.44; H, 7.88; N, 4.05; O, 4.63. Found: C, 83.55; H, 7.79; N, 4.00; O, 4.69.

(3*S*,4*S*)-4-(Dibenzylamino)-6-methylheptan-3-ol (4d): colorless oil. $[\alpha]^{25}_D$ = +26.7 (*c* 1.35, $CHCl_3$). ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.31−7.22 (m, 10 H), 3.89 (d, *J* = 13.3 Hz, 2 H), 3.45 (d, *J* = 13.3 Hz, 2 H), 3.44−3.39 (m, 1 H), 2.53 (ddd, *J* = 9.1, 6.4, 4.4 Hz, 1 H), 1.81−1.52 (m, 3 H), 1.30−1.14 (m, 2 H), 0.98 (d, *J* = 6.3 Hz, 6 H), 0.96 (t, *J* = 7.3 Hz, 3 H). ^{13}C NMR (50 MHz, $CDCl_3$): δ 139.1 (2 × C), 130.0 (4 × CH), 128.4 (4 × CH), 127.1 (2 × CH), 72.3 (CH), 60.4 (CH), 53.7 (2 × CH_2), 35.6 (CH_2), 26.6 (CH_2 and CH), 23.3 (CH_3), 22.9 (CH_3), 10.2 (CH_3). MS (70 eV, EI) *m*/*z* (%): 307 ($M^+ - H_2O$, 2), 251 (71), 69 (100), 57 (74). HRMS (70 eV) calcd for $C_{22}H_{29}N$ ($M^+ - H_2O$), 307.2300; found, 307.2327. IR (neat): 3422, 2957, 2350, 1603, 1495, 1454, 1368 cm^{-1}. R_f = 0.32 (hexane/EtOAc 10:1). Anal. Calcd for $C_{22}H_{31}NO$: C, 81.18; H, 9.60; N, 4.30; O, 4.92. Found: C, 81.03; H, 9.71; N, 4.34; O, 4.86.

(5*S*,6*S*)-6-(Dibenzylamino)-8-methylnon-1-en-5-ol (4e): colorless oil. $[\alpha]^{25}_D$ = +10.2 (*c* 2.14, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.36−7.24 (m, 10 H), 5.86 (ddt, *J* = 17.1, 10.2, 6.7 Hz, 1 H), 5.04 (dd, *J* = 17.1, 2.0 Hz, 1 H), 4.76 (dd, *J* = 10.2, 2.0 Hz, 1 H), 4.54 (br s, 1 H), 3.89 (d, *J* = 13.4 Hz, 2 H), 3.51 (dt, *J* = 9.0, 2.3 Hz, 1 H), 3.46 (d, *J* = 13.4 Hz, 2 H), 2.54 (ddd, *J* = 9.0, 6.2, 4.7 Hz, 1 H), 2.35−2.23 (m, 1 H), 2.20−2.08 (m, 1 H), 1.81−1.72 (m, 1 H), 1.65−1.55 (m, 2 H), 1.37−1.22 (m, 2 H), 1.00 (d, *J* = 6.5 Hz, 6 H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 139.0 (2 × C), 138.7 (CH), 128.9 (4 × CH), 128.3 (4 × CH), 127.1 (2 × CH), 114.3 (CH_2), 70.5 (CH), 60.7 (CH), 53.7 (2 × CH_2), 35.5 (CH_2), 33.3 (CH_2), 30.2 (CH_2), 26.6 (CH), 23.3 (CH_3), 22.9 (CH_3). MS (70 eV, EI) *m*/*z* (%): 351 (M^+, <1), 266 (100). HRMS (70 eV): calcd for $C_{24}H_{33}NO$ (M^+), 351.2562; found, 351.2558. IR (neat): 3426, 2955, 2360, 1603, 1495, 1454, 1368 cm^{-1}. R_f = 0.42 (hexane/EtOAc 10:1). Anal. Calcd for $C_{24}H_{33}NO$: C, 82.00; H, 9.46; N, 3.98; O, 4.55. Found: C, 81.85; H, 9.38; N, 4.03; O, 4.61.

(2*S*,3*S*)-3-(Dibenzylamino)-5-methyl-1-phenylhexan-2-ol (4f): colorless oil. $[\alpha]^{25}_D$ = −25.1 (*c* 0.98, $CHCl_3$). ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.33−7.20 (m, 15 H), 4.42 (br s, 1 H), 3.92 (d, *J* = 13.3 Hz, 2 H), 3.75 (dt, *J* = 9.0, 2.3 Hz, 1 H), 3.49 (d, *J* = 13.3 Hz, 2 H), 2.85 (dd, *J* = 13.9, 2.3 Hz, 1 H), 2.69−2.59 (m, 1 H), 2.50 (dd, *J* = 13.9, 9.0 Hz, 1 H), 1.90−1.61 (m, 2 H), 1.41−1.29 (m, 1 H), 0.99 (d, *J* = 6.3 Hz, 3 H), 0.98 (d, *J* = 6.3 Hz, 3 H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 139.2 (C), 139.1 (2 × C), 129.2 (2 × CH), 129.0 (4 × CH), 128.4 (4 × CH), 128.2 (2 × CH), 127.1 (2 × CH), 126.0 (CH), 72.3 (CH), 60.4 (CH), 54.0 (2 × CH_2), 40.6 (CH_2), 35.9 (CH_2), 26.6 (CH), 23.1 (2 × CH_3). MS (70 eV, EI) *m*/*z* (%): 443 (42), 387 (M^+, 2), 181 (100), 175 (33), 169 (33). HRMS (70 eV): calcd for $C_{27}H_{33}NO$ (M^+), 387.2562; found, 387.2581. IR (neat): 3422, 2955, 2357, 1602, 1495, 1454, 1367 cm^{-1}. R_f = 0.40 (hexane/EtOAc 20:1). Anal. Calcd for $C_{27}H_{33}NO$: C, 83.68; H, 8.58; N, 3.61; O, 4.13. Found: C, 83.81; H, 8.69; N, 3.57; O, 4.19.

(2*S*,3*S*)-2-(Dibenzylamino)-1-phenylpentan-3-ol (4g): colorless oil. $[\alpha]^{25}_D$ = +23.4 (*c* 1.49, $CHCl_3$). ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.36−7.27 (m, 15 H), 4.51 (br s, 1 H), 3.96 (d, *J* = 13.3 Hz, 2 H), 3.59 (dt, *J* = 8.3, 2.4 Hz, 1 H), 3.42 (d, *J* = 13.3 Hz, 2 H), 3.14 (dd, *J* = 14.1, 6.3 Hz, 1 H), 2.99−2.89 (m, 1 H), 2.72 (dd, *J* = 14.1, 5.9 Hz, 1 H), 1.58−1.43 (m, 1 H), 1.20−1.09 (m, 1 H), 0.86 (t, *J* = 7.2 Hz, 3 H). ^{13}C NMR (50 MHz, $CDCl_3$): δ 140.5 (C), 138.7 (2 × C), 129.1 (2 × CH), 129.0 (4 × CH), 128.5 (2 × CH), 128.4 (4 × CH), 127.2 (2 × CH), 126.2 (CH), 71.4 (CH), 63.5 (CH), 53.8 (2 × CH_2), 32.4 (CH_2), 26.9 (CH_2), 9.7 (CH_3). MS (70 eV, EI) *m*/*z* (%): 359 (M^+, 1), 300 (100), 268 (15), 181 (16). HRMS (70 eV): calcd for $C_{25}H_{29}NO$ (M^+), 359.2249; found, 359.2228. IR (neat): 3421, 3027, 2350, 1602, 1495, 1454, 1374 cm^{-1}. R_f = 0.32 (hexane/EtOAc 10:1). Anal. Calcd for $C_{25}H_{29}NO$: C, 83.52; H, 8.13; N, 3.90; O, 4.45. Found: C, 83.66; H, 8.21; N, 3.95; O, 4.40.

(2*S*,3*S*)-2-(Dibenzylamino)-1-phenylhept-6-en-3-ol (4h): colorless oil. $[\alpha]^{25}_D$ = +25.4 (*c* 1.39, $CHCl_3$). ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.41−7.29 (m, 15 H), 5.78 (dt, *J* = 16.9, 11.0, 6.8 Hz, 1 H), 5.02−4.92 (m, 2 H), 4.51 (br s, 1 H), 3.98 (d, *J* = 13.3 Hz, 2 H), 3.68 (dt, *J* = 8.8, 2.7 Hz, 1 H), 3.48 (d, *J* = 13.3 Hz, 2 H), 3.17 (dd, *J* = 13.9, 6.3 Hz, 1 H), 2.96 (ddd, *J* = 8.8, 6.3, 6.1 Hz, 1 H), 2.74 (dd, *J* = 13.9, 6.1 Hz, 1 H), 2.19−2.07 (m, 2 H), 1.65−1.48 (m, 1 H), 1.35−1.14 (m, 1 H). ^{13}C NMR (50 MHz, $CDCl_3$): δ 140.3 (C), 138.7 (2 × C), 138.6 (CH), 129.1 (2 × CH), 128.9 (4 × CH), 128.5 (2 × CH), 128.3 (4 × CH), 127.1 (2 × CH), 126.1 (CH), 114.3 (CH_2), 69.7 (CH), 63.7 (CH), 53.7 (2 × CH_2), 33.5 (CH_2), 32.3 (CH_2), 29.7 (CH_2). MS (70 eV, EI) *m*/*z* (%): 385 (M^+, 2), 301 (100), 295 (19), 250 (33), 181 (17). HRMS (70 eV): calcd for $C_{27}H_{31}NO$ (M^+), 385.2406; found, 385.2395. IR (neat): 3417, 3027, 2924, 2349, 1602, 1495, 1454, 1375 cm^{-1}. R_f = 0.35 (hexane/EtOAc 10:1). Anal. Calcd for $C_{27}H_{31}NO$: C, 84.11; H, 8.10; N, 3.63; O, 4.15. Found: C, 84.23; H, 8.03; N, 3.59; O, 4.21.

(2*S*,3*S*)-3-(Dibenzylamino)-1,4-diphenylbutan-2-ol (4i): colorless oil. $[\alpha]^{25}_D$ = +4.7 (*c* 1.43, $CHCl_3$). ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.42−7.12 (m, 20 H), 3.99 (d, *J* = 13.3 Hz, 2 H), 3.87 (dt, *J* = 8.2, 2.7 Hz, 1 H), 3.43 (d, *J* = 13.3 Hz, 2 H), 3.19 (dd, *J* = 13.1, 5.9 Hz, 1 H), 2.99 (dt, *J* = 8.2, 6.3 Hz, 1 H), 2.86 (dd, *J* = 13.1, 6.5 Hz, 1 H), 2.74 (dd, *J* = 14.1, 2.4 Hz, 1 H), 2.48 (dd, *J* = 14.1, 8.6 Hz, 1 H). ^{13}C NMR (50 MHz, $CDCl_3$): δ 140.3 (C), 139.1 (C), 138.8 (2 × C), 129.2 (2 × CH), 129.0 (5 × CH), 128.6 (2 × CH), 128.3 (5 × CH), 128.1 (2 × CH), 127.1 (2 × CH), 126.2 (CH), 126.0 (CH), 71.5 (CH), 63.4 (CH), 54.0 (2 × CH_2), 40.8 (CH_2), 32.3 (CH_2). MS (70 eV, EI) *m*/*z* (%): 421 (M^+, <1), 330 (100), 300 (78), 210 (24). HRMS (70 eV): calcd for $C_{30}H_{31}NO$ (M^+), 421.2406; found, 421.2412. IR (neat): 3421, 3026, 2343, 1602, 1495, 1454, 1367 cm^{-1}. R_f = 0.32 (hexane/EtOAc 10:1). Anal. Calcd for $C_{30}H_{31}NO$: C, 85.47; H, 7.41; N, 3.32; O, 3.80. Found: C, 85.58; H, 7.50; N, 3.26; O, 3.75.

(2*S*,3*R*)-2-(Dibenzylamino)pentan-3-ol (5a): colorless oil. $[\alpha]^{25}_D$ = +33.0 (*c* 1.26, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.25−7.12 (m, 10 H), 3.66 (d, *J* = 13.8 Hz, 2 H), 3.45−3.40 (m, 1 H), 3.37 (d, *J* = 13.8 Hz, 2 H), 2.61 (apparent qt, *J* = 6.6 Hz, 1 H), 1.70−1.63 (m, 1 H), 1.25−1.18 (m, 1 H), 1.01 (d, *J* = 6.8 Hz, 3 H), 0.76 (d, *J* = 7.4 Hz, 3 H). ^{13}C NMR (50 MHz, $CDCl_3$): δ 140.1 (2 × C), 128.7 (4 × CH), 128.2 (4 × CH), 126.8 (2 × CH), 75.2 (CH), 57.0 (CH), 54.7 (2 × CH_2), 27.1 (CH_2), 10.3 (CH_3), 8.6 (CH_3). HRMS (70 eV): calcd for $C_{18}H_{22}NO$ ($M^+ - CH_3$), 268.1701; found, 268.1689. IR (neat): 3396, 2963, 2359, 102, 1494, 1453, 1377 cm^{-1}. R_f = 0.40 (hexane/EtOAc 5:1). Anal. Calcd for $C_{19}H_{25}NO$: C, 80.52; H, 8.89; N, 4.94; O, 5.65. Found: C, 80.39; H, 8.80; N, 5.00; O, 5.71.

(2*S*,3*R*)-2-(Dibenzylamino)hept-6-en-3-ol (5b): colorless oil. $[\alpha]^{25}_D$ = +40.8 (*c* 1.05, $CHCl_3$). ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.42−7.25 (m, 10 H), 5.87 (ddt, *J* = 17.1, 10.3, 6.7 Hz, 1 H), 5.11−4.97 (m, 2 H), 3.82 (d, *J* = 14.0 Hz, 2 H), 3.65 (apparent t, *J* = 6.7 Hz, 1 H), 3.50 (d, *J* = 14.0 Hz, 2 H), 2.77 (apparent qt, *J* = 6.7 Hz, 1 H), 2.24−1.82 (m, 2 H), 1.52−1.31 (m, 2 H), 1.17 (d, *J* = 6.7 Hz, 3 H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 139.9 (2 × C), 138.7 (CH), 128.8 (4 × CH), 128.3 (4 × CH), 126.8 (2 × CH), 114.6 (CH_2), 73.0 (CH), 57.1 (CH), 54.6 (2 × CH_2), 33.3 (CH_2), 30.1 (CH_2), 8.5 (CH_3). MS (70 eV, EI) *m*/*z* (%): 309 (M^+, 1), 224 (100). HRMS (70 eV): calcd for $C_{21}H_{27}NO$ (M^+), 309.2093; found, 309.2094. IR (neat): 3386, 2923, 2347, 1602, 1494, 1453, 1376 cm^{-1}. R_f = 0.20 (hexane/EtOAc 10:1). Anal. Calcd for $C_{21}H_{27}NO$: C, 81.51; H, 8.79; N, 4.53; O, 5.17. Found: C, 81.62; H, 8.71; N, 4.57; O, 5.22.

(2*R*,3*S*)-3-(Dibenzylamino)-1-phenylbutan-2-ol (5c): colorless oil. $[\alpha]^{25}_D$ = +11.1 (*c* 0.90, $CHCl_3$). ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.51−7.20 (m, 15 H), 3.92 (d, *J* = 13.7 Hz, 2 H), 3.85−3.73 (m,

1 H), 3.60 (d, J = 13.7 Hz, 2 H), 3.42 (dd, J = 13.7, 3.1 Hz, 1 H), 2.87 (apparent qt, J = 6.8 Hz, 1 H), 2.43 (dd, J = 13.7, 9.8 Hz, 1 H), 1.84, (br s, 1 H), 1.28 (d, J = 6.7 Hz, 3 H). ^{13}C NMR (50 MHz, $CDCl_3$): δ 140.0 (2 × C), 139.3 (C), 129.2 (2 × CH), 128.7 (4 × CH), 128.4 (2 × CH), 128.2 (4 × CH), 126.8 (2 × CH), 126.2 (CH), 74.6 (CH), 51.2 (CH), 54.5 (2 × CH_2), 41.4 (CH_2), 8.5 (CH_3). MS (70 eV, EI) *m*/*z* (%): 345 (M^+, 1), 224 (100). HRMS (70 eV): calcd for $C_{24}H_{27}NO$ (M^+), 345.2093; found, 345.2102. IR (neat): 3419, 3027, 2352, 1602, 1494, 1453, 1377 cm^{-1}. R_f = 0.26 (hexane/EtOAc 10:1). Anal. Calcd for $C_{24}H_{27}NO$: C, 83.44; H, 7.88; N, 4.05; O, 4.63. Found: C, 83.58; H, 7.79; N, 4.10; O, 4.58.

(3*R*,4*S*)-4-(Dibenzylamino)-6-methylheptan-3-ol (5d): colorless oil. $[\alpha]^{25}_D$ = +1.5 (*c* 1.13, $CHCl_3$). ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.37−7.26 (m, 10 H), 3.70 (AB syst., J = 14.5 Hz, 4 H), 3.66−3.61 (m, 1 H), 2.80−2.72 (m, 1 H), 1.87−1.04 (m, 5 H), 0.97 (t, J = 7.8 Hz, 3 H), 0.95 (d, J = 6.7 Hz, 3 H), 0.76 (d, J = 6.7 Hz, 3 H). ^{13}C NMR (50 MHz, $CDCl_3$): δ 140.2 (2 × C), 128.9 (4 × CH), 128.2 (4 × CH), 126.9 (2 × CH), 72.2 (CH), 58.1 (CH), 55.0 (2 × CH_2), 34.4 (CH_2), 27.5 (CH_2), 24.7 (CH), 23.1 (CH_3), 22.7 (CH_3), 11.1 (CH_3). MS (70 eV, EI) *m*/*z* (%): 310 ($M^+ - CH_3$, <1), 266 (46), 91 (100). HRMS (70 eV): calcd for $C_{21}H_{28}NO$ ($M^+ - CH_3$), 310.2171; found, 31.2169. IR (neat): 3448, 2956, 2350, 1494, 1455, 1364 cm^{-1}. R_f = 0.26 (hexane/EtOAc 10:1). Anal. Calcd for $C_{22}H_{31}NO$: C, 81.18; H, 9.60; N, 4.30; O, 4.92. Found: C, 81.36; H, 9.51; N, 4.34; O, 4.87.

(5*R*,6*S*)-6-(Dibenzylamino)-8-methylnon-1-en-5-ol (5e): white solid. $[\alpha]^{25}_D$ = +0.2 (*c* 2.31, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.25−7.15 (m, 10 H), 5.74 (ddt, J = 17.2, 10.2, 6.6 Hz, 1 H), 5.00−4.93 (m, 1 H), 4.92−4.87 (m, 1 H), 3.70−3.63 (m, 1 H), 3.58 (s, 4 H), 2.66 (dt, J = 7.0, 4.0 Hz, 1 H), 2.25−2.08 (m, 2 H), 2.05−1.92 (m, 1 H), 1.75−1.62 (m, 1 H), 1.57−1.33 (m, 3 H), 1.25−1.13 (m, 1 H), 0.84 (d, J = 6.7 Hz, 3 H), 0.68 (d, J = 6.5, 3 H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 140.0 (2 × C), 138.4 (CH), 128.9 (4 × CH), 128.2 (4 × CH), 127.0 (2 × CH), 114.8 (CH_2), 70.0 (CH), 58.5 (CH), 55.1 (2 × CH_2), 34.4 (CH_2), 33.6 (CH_2), 30.8 (CH_2), 24.7 (CH), 22.9 (CH_3), 22.8 (CH_3). MS (70 eV, EI) *m*/*z* (%): 351 (M^+, <1), 266 (55), 91 (36), 69 (100). HRMS (70 eV): calcd for $C_{24}H_{33}NO$ (M^+), 351.2562; found, 351.2553. IR (neat): 3387, 2952, 2361, 1603, 1494, 1453, 1367 cm^{-1}. R_f = 0.29 (hexane/EtOAc 10:1). Anal. Calcd for $C_{24}H_{33}NO$: C, 82.00; H, 9.46; N, 3.98; O, 4.55. Found: C, 81.86; H, 9.58; N, 3.93; O, 4.61.

(2*R*,3*S*)-3-(Dibenzylamino)-5-methyl-1-phenylhexan-2-ol (5f): white solid. $[\alpha]^{25}_D$ = −38.0 (*c* 1.25, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.41−7.20 (m, 15 H), 4.19−4.14 (m, 1 H), 3.82 (d, J = 13.7 Hz, 2 H), 3.57 (d, J = 13.7 Hz, 2 H), 2.87 (dd, J = 13.5, 5.0 Hz, 1 H), 2.77−2.72 (m, 1 H), 2.66 (dd, J = 13.5, 8.9 Hz, 1 H), 1.99−1.87 (m, 1 H), 1.73 (ddd, J = 13.9, 8.5, 5.2 Hz, 1 H), 1.34−1.29 (m, 1 H), 0.98 (d, J = 6.6 Hz, 3 H), 0.67 (d, J = 6.6 Hz, 3 H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 140.2 (2 × C), 138.9 (C), 129.3 (2 × CH), 129.0 (4 × CH), 128.6 (2 × CH), 128.1 (4 × CH), 126.7 (2 × CH), 126.3 (CH), 71.7 (CH), 57.7 (CH), 54.6 (2 × CH_2), 42.1 (CH_2), 34.6 (CH_2), 24.6 (CH), 23.5 (CH_3), 22.2 (CH_3). MS (70 eV, EI) *m*/*z* (%): 387 (M^+, 1), 278 (26), 266 (100). HRMS (70 eV): calcd for $C_{27}H_{33}NO$ (M^+), 387.2562; found, 387.2571. IR (neat): 3389, 2948, 2357, 1601, 1493, 1454, 1362 cm^{-1}. R_f = 0.22 (hexane/EtOAc 10:1). Anal. Calcd for $C_{27}H_{33}NO$: C, 83.68; H, 8.58; N, 3.61; O, 4.13. Found: C, 83.56; H, 8.65; N, 3.65; O, 4.09.

(2*S*,3*R*)-2-(Dibenzylamino)-1-phenylpentan-3-ol (5g): colorless oil. $[\alpha]^{25}_D$ = −9.6 (*c* 1.11, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.28−7.05 (m, 15 H), 3.88−3.82 (m, 1 H), 3.67 (d, J = 13.8 Hz, 2 H), 3.56 (d, J = 13.8 Hz, 2 H), 3.43−3.32 (m, 1 H), 3.02−2.89 (m, 2 H), 2.70 (dd, J = 12.6, 5.6 Hz, 1 H), 1.62−1.54 (m, 1 H), 1.35−1.23 (m, 1 H), 0.79 (t, J = 7.4 Hz, 3 H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 140.6 (C), 139.7 (2 × C), 129.3 (2 × CH), 128.7 (4 × CH), 128.3 (2 × CH), 128.2 (4 × CH), 126.9 (2 × CH), 125.9 (CH), 73.3 (CH), 63.0 (CH), 55.0 (2 × CH_2), 31.8 (CH_2), 27.6 (CH_2), 10.8 (CH_3). MS (70 eV, EI) *m*/*z* (%): 359 (M^+, <1), 300 (28), 91 (24), 69 (100). HRMS (70 eV): calcd for $C_{25}H_{29}NO$ (M^+), 359.2249; found, 359.2244. IR (neat): 3427, 2929, 2360, 1602, 1494, 1454, 1365 cm^{-1}. R_f = 0.13 (hexane/EtOAc 10:1). Anal. Calcd for $C_{25}H_{29}NO$: C, 83.52; H, 8.13; N, 3.90; O, 4.45. Found: C, 83.67; H, 8.21; N, 3.95; O, 4.51.

(2*S*,3*R*)-2-(Dibenzylamino)-1-phenylhept-6-en-3-ol (5h): colorless oil. $[\alpha]^{25}_D$ = +17.4 (*c* 1.34, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.26−7.10 (m, 15 H), 5.73 (ddt, J = 17.1, 10.2, 6.7 Hz, 1 H), 4.98−4.88 (m, 2 H), 3.75−3.63 (m, 4 H), 3.57 (d, J = 13.7 Hz, 2 H), 3.07−2.94 (m, 2 H), 2.74 (dd, J = 12.4, 5.7 Hz, 1 H), 2.19−2.09 (m, 1 H), 2.01−1.89 (m, 1 H), 1.74−1.64 (m, 1 H), 1.44−1.35 (m, 1 H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 140.4 (C), 139.6 (2 × C), 138.3 (CH), 129.2 (2 × CH), 128.7 (4 × CH), 128.2 (2 × CH), 128.1 (4 × CH), 126.9 (2 × CH), 125.9 (CH), 114.8 (CH_2), 71.0 (CH), 63.2 (CH), 55.0 (2 × CH_2), 33.7 (CH_2), 31.9 (CH_2), 30.5 (CH_2). MS (70 eV, EI) *m*/*z* (%): 385 (M^+, <1), 300 (100), 301 (27). HRMS (70 eV): calcd for $C_{27}H_{31}NO$ (M^+), 385.2406; found, 385.2381. IR (neat): 3432, 3026, 2360, 1602, 1494, 1453, 1364 cm^{-1}. R_f = 0.16 (hexane/EtOAc 10:1). Anal. Calcd for $C_{27}H_{31}NO$: C, 84.11; H, 8.10; N, 3.63; O, 4.15. Found: C, 84.28; H, 8.01; N, 3.58; O, 4.21.

(2*S*,3*R*)-3-(Dibenzylamino)-1,4-diphenylbutan-2-ol (5i): white solid. $[\alpha]^{25}_D$ = −5.7 (*c* 1.71, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.29−7.02 (m, 20 H), 4.01−3.97 (m, 1 H), 3.65 (AB syst., J = 15.0 Hz, 4 H), 3.09−3.00 (m, 2 H), 2.96 (dd, J = 13.6, 3.8 Hz, 1 H), 2.89 (dd, J = 10.9, 3.1 Hz, 1 H), 2.44 (dd, J = 13.6, 9.8 Hz, 1 H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 140.7 (C), 139.7 (C), 138.9 (2 × C), 129.4 (2 × CH), 129.2 (2 × CH), 128.7 (4 × CH), 128.5 (2 × CH), 128.2 (2 × CH), 128.1 (4 × CH), 126.8 (2 × CH), 126.3 (CH), 125.8 (CH), 72.9 (CH), 62.8 (CH), 54.8 (2 × CH_2), 41.9 (CH_2), 32.0 (CH_2). HRMS (70 eV): calcd for $C_{30}H_{31}NO$ (M^+) 421.2406; found, 421.2413. IR (neat): 3420, 3028, 2359, 1602, 1494, 1454, 1364 cm^{-1}. R_f = 0.17 (hexane/EtOAc 10:1). Anal. Calcd for $C_{30}H_{31}NO$: C, 85.47; H, 7.41; N, 3.32; O, 3.80. Found: C, 85.33; H, 7.50; N, 3.26; O, 3.86.

General Procedure of Synthesis of Compounds 6, 7, 8, and 9. To a stirred solution of the corresponding aminoepoxide **1** or **2** (0.2 mmol) in THF (1 mL), $LiAlH_4$ or $LiAlD_4$ (0.3 mmol, 0.3 mL, 1 M in THF) was added at 0 °C. After stirring at this temperature for 2 h, an aqueous saturated solution of NH_4Cl (5 mL) was added, and the mixture was stirred at room temperature for 5 min. Then, the aqueous phase was extracted with diethyl ether (3 × 5 mL), and the combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated in vacuo. Flash column chromatography on silica gel (hexane/EtOAc 10:1) provided pure compounds **6**, **7**, **8**, or **9**.

(2*S*,3*S*)-3-(Dibenzylamino)butan-2-ol (6a): colorless oil. $[\alpha]^{25}_D$ = +75.0 (*c* 2.30, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.38−7.26 (m, 10 H), 4.41 (br s, 1 H), 3.87 (d, J = 13.3 Hz, 2 H), 3.67 (dq, J = 9.3, 6.1 Hz, 1 H), 3.36 (d, J = 13.3 Hz, 2 H), 2.53 (dq, J = 9.3, 6.6 Hz, 1 H), 1.12 (d, J = 6.1 Hz, 3 H), 1.05 (d, J = 6.6 Hz, 3 H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 138.8 (2 × C), 128.9 (4 × CH), 128.5 (4 × CH), 127.1 (2 × CH), 66.9 (CH), 60.2 (CH), 53.3 (2 × CH_2), 19.3 (CH_3), 7.8 (CH_3). MS (70 eV, EI) *m*/*z* (%): 251 ($M^+ - H_2O$, <1), 224 (92), 91 (100). HRMS (70 eV): calcd for $C_{18}H_{21}N$ (M^+-H_2O), 251.1674; found, 251.1702. IR (neat): 3426, 2971, 2351, 1603, 1495, 1453, 1383 cm^{-1}. R_f = 0.17 (hexane/EtOAc 10:1). Anal. Calcd for $C_{18}H_{23}NO$: C, 80.26; H, 8.61; N, 5.20; O, 5.94. Found: C, 80.40; H, 8.52; N, 5.15; O, 5.99.

(2*S*,3*S*)-3-(Dibenzylamino)-5-methylhexan-2-ol (6b): colorless oil. $[\alpha]^{25}_D$ = +60.9 (*c* 2.18, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.38−7.26 (m, 10 H), 4.48 (br s, 1 H), 3.90 (d, J = 13.4 Hz, 2 H), 3.67 (dq, J = 9.0, 6.1 Hz, 1 H), 3.47 (d, J = 13.4 Hz, 2 H), 2.48 (ddd, J = 9.0, 6.6, 4.4 Hz, 1 H), 1.84−1.77 (m, 1 H), 1.63 (ddd, J = 14.2, 8.2, 4.4 Hz, 1 H), 1.16 (d, J = 6.1 Hz, 3 H), 1.08−1.04 (m, 1 H), 1.02 (d, J = 6.6 Hz, 6 H). ^{13}C NMR (50 MHz, $CDCl_3$): δ 139.1 (2 × C), 128.8 (4 × CH), 128.3 (4 × CH), 127.0 (2 × CH), 67.2 (CH), 62.6 (CH), 53.7 (2 × CH_2), 35.3 (CH_2),

26.4 (CH), 23.3 (CH_3), 22.8 (CH_3), 19.7 (CH_3). MS (70 eV, EI) *m*/*z* (%): 311 (M^+, <1), 266 (100), 181 (11). HRMS (70 eV): calcd for $C_{21}H_{29}NO$ (M^+), 311.2249; found, 311.2266. IR (neat): 3441, 2956, 2360, 1603, 1495, 1454, 1377 cm^{-1}. R_f = 0.24 (hexane/EtOAc 10:1). Anal. Calcd for $C_{21}H_{29}NO$: C, 80.98; H, 9.38; N, 4.50; O, 5.14. Found: C, 81.15; H, 9.30; N, 4.56; O, 5.08.

(2*S*,3*S*)-3-(Dibenzylamino)-4-phenylbutan-2-ol (6c): white solid. $[\alpha]^{25}_D$ = +49.2 (*c* 1.60, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$): δ 7.39−7.26 (m, 15 H), 4.41 (br s, 1 H), 3.96 (d, *J* = 13.3 Hz, 2 H), 3.81 (dq, *J* = 8.7, 6.0 Hz, 1 H), 3.44 (d, *J* = 13.3 Hz, 2 H), 3.14 (dd, *J* = 14.2, 6.4 Hz, 1 H), 2.87 (ddd, *J* = 8.7, 6.4, 6.3 Hz, 1 H), 2.72 (dd, *J* = 14.2, 6.3 Hz, 1 H), 1.06 (d, *J* = 6.0 Hz, 3 H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 140.4 (C), 138.8 (2 × C), 129.1 (2 × CH), 128.9 (4 × CH), 128.4 (2 × CH), 128.3 (4 × CH), 127.1 (2 × CH), 126.1 (CH), 66.5 (CH), 65.9 (CH), 53.7 (2 × CH_2), 32.1 (CH_2), 20.2 (CH_3). MS (70 eV, EI) *m*/*z* (%): 345 (M^+, <1), 300 (100). HRMS (70 eV): calcd for $C_{24}H_{27}NO$ (M^+), 345.2093; found, 345.2086. IR (neat): 3361, 3025, 2370, 1602, 1495, 1454, 1378 cm^{-1}. R_f = 0.20 (hexane/EtOAc 10:1). Anal. Calcd for $C_{24}H_{27}NO$: C, 83.44; H, 7.88; N, 4.05; O, 4.63. Found: C, 83.30; H, 7.79; N, 4.10; O, 4.69.

(2*R*,3*S*)-3-(Dibenzylamino)butan-2-ol (7a): white solid. $[\alpha]^{25}_D$ = +78.8 (*c* 1.46, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$): δ 7.42−7.27 (m, 10 H),), 3.82 (d, *J* = 13.7 Hz, 2 H), 3.50 (d, *J* = 13.7 Hz, 2 H), 3.43−3.35 (m, 1 H), 2.68 (apparent qt, *J* = 6.7 Hz, 1 H), 2.14 (br s, 1 H), 1.26 (d, *J* = 6.4 Hz, 3 H), 1.16 (d, *J* = 6.7 Hz, 3 H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 140.0 (2 × C), 128.6 (4 × CH), 128.1 (4 × CH), 126.8 (2 × CH), 70.0 (CH), 58.4 (CH), 54.6 (2 × CH_2), 21.0 (CH_3), 8.5 (CH_3). MS (70 eV, EI) *m*/*z* (%): 254 ($M^+ - CH_3$), <1), 224 (100), 181 (20), 119 (28). HRMS (70 eV): calcd for $C_{17}H_{20}NO$ ($M^+ - CH_3$), 254.1545; found, 254.1545. IR (neat): 3262, 3022, 2970, 2356, 1602, 1494, 1453, 1364 cm^{-1}. R_f = 0.44 (hexane/EtOAc 3:1). Anal. Calcd for $C_{18}H_{23}NO$: C, 80.26; H, 8.61; N, 5.20; O, 5.94. Found: C, 80.42; H, 8.70; N, 5.24; O, 5.89.

(2*R*,3*S*)-3-(Dibenzylamino)-5-methylhexan-2-ol (7b): colorless oil. $[\alpha]^{25}_D$ = +34.2 (*c* 1.34, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$): δ 7.29−7.13 (m, 10 H), 3.87 (dq, *J* = 6.6, 4.2 Hz, 1 H), 3.60 (AB syst., *J* = 13.6 Hz, 4 H), 3.63 (ddd, *J* = 6.9, 6.8, 4.2 Hz, 1 H), 2.33 (br s, 1 H), 1.76−1.63 (m, 1 H), 1.55−1.46 (m, 1 H), 1.25−1.16 (m, 1 H), 1.11 (d, *J* = 6.6 Hz, 3 H), 0.85 (d, *J* = 6.5 Hz, 3 H), 0.71 (d, *J* = 6.5 Hz, 3 H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 140.0 (2 × C), 128.9 (4 × CH), 128.2 (4 × CH), 126.9 (2 × CH), 66.5 (CH), 59.1 (CH), 55.1 (2 × CH_2), 34.5 (CH_2), 24.8 (CH), 22.9 (CH_3), 22.8 (CH_3), 20.7 (CH_3). MS (70 eV, EI) *m*/*z* (%): 311 (M^+, <1), 266 (100), 91 (31). HRMS (70 eV): calcd for $C_{21}H_{29}NO$ (M^+), 311.2249; found, 311.2247. IR (neat): 3419, 2955, 2350, 1603, 1494, 1454, 1367 cm^{-1}. R_f = 0.21 (hexane/EtOAc 10:1). Anal. Calcd for $C_{21}H_{29}NO$: C, 80.98; H, 9.38; N, 4.50; O, 5.14. Found: C, 80.80; H, 9.46; N, 4.44; O, 5.19.

(2*R*,3*S*)-3-(Dibenzylamino)-4-phenylbutan-2-ol (7c): colorless oil. $[\alpha]^{25}_D$ = +13.7 (*c* 1.52, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$): δ 7.25−7.10 (m, 15 H), 3.84−3.80 (m, 1 H), 3.74 (d, *J* = 13.8 Hz, 2 H), 3.55 (d, *J* = 13.8 Hz, 2 H), 3.02 (dd, *J* = 13.3, 6.7 Hz, 1 H), 2.85 (dt, *J* = 6.7, 4.8 Hz, 1 H), 2.74 (dd, *J* = 13.3, 6.6 Hz, 1 H), 2.18 (br s, 1 H), 1.17 (d, *J* = 6.5 Hz, 3 H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 140.4 (C), 139.5 (2 × C), 129.2 (2 × CH), 128.7 (4 × CH), 128.3 (2 × CH), 128.2 (4 × CH), 126.9 (2 × CH), 125.9 (CH), 67.6 (CH), 64.0 (CH), 55.1 (2 × CH_2), 32.0 (CH_2), 20.9 (CH_3). MS (70 eV, EI) *m*/*z* (%): 345 (M^+, <1), 300 (97), 91 (100). HRMS (70 eV): calcd for $C_{24}H_{27}NO$ (M^+), 345.2093; found, 345.2103. IR (neat): 3418, 3027, 2360, 1602, 1494, 1454, 1372 cm^{-1}. R_f = 0.37 (hexane/EtOAc 5:1). Anal. Calcd for $C_{24}H_{27}NO$: C, 83.44; H, 7.88; N, 4.05; O, 4.63. Found: C, 83.58; H, 7.97; N, 4.00; O, 4.69.

(2*S*,3*S*)-1-Deuterium-3-(dibenzylamino)butan-2-ol (8a): colorless oil. $[\alpha]^{25}_D$ = +107.3 (*c* 1.45, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$): δ 7.26−7.14 (m, 10 H), 4.27 (br s, 1 H), 3.75 (d, *J* = 13.3 Hz, 2 H), 3.54 (dt, *J* = 9.4, 5.9 Hz, 1 H), 3.23 (d, *J* = 13.3 Hz, 2 H), 2.40 (dq, *J* = 9.4, 6.6 Hz, 1 H), 0.98 (d, *J* = 5.9 Hz, 2 H), 0.93 (d, *J* = 6.6 Hz, 3 H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 138.9 (2 × C), 128.9 (4 × CH), 128.3 (4 × CH), 127.1 (2 × CH), 66.8 (CH), 60.1 (CH), 53.2 (2 × CH_2), 19.0 (t, *J* = 76.4 Hz, CH_2D), 7.7 (CH3). MS (70 eV, EI) *m*/*z* (%): 270 (M^+, <1), 224 (84), 91 (100), 69 (27), 58 (43). HRMS (70 eV): calcd for $C_{18}H_{22}DNO$ (M^+), 270.1842; found, 270.1858. IR (neat): 3431, 2969, 2361, 1603, 1495, 1453, 1377 cm^{-1}. R_f = 0.21 (hexane/EtOAc 10:1). Anal. Calcd for $C_{18}H_{22}DNO$: C, 79.96; H, 8.95; N, 5.18; O, 5.92. Found: C, 79.79; H, 9.03; N, 5.13; O, 5.98.

(2*S*,3*S*)-1-Deuterium-3-(dibenzylamino)-4-phenylbutan-2-ol (8c): white solid. $[\alpha]^{25}_D$ = +35.8 (*c* 1.97, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$): δ 7.28−7.11 (m, 15 H), 4.27 (br s, 1 H), 3.82 (d, *J* = 13.3 Hz, 2 H), 3.67 (dt, *J* = 8.9, 6.0 Hz, 1 H), 3.30 (d, *J* = 13.3 Hz, 2 H), 3.01 (dd, *J* = 14.3, 6.3 Hz, 1 H), 2.73 (dt, *J* = 8.9, 6.3 Hz, 1 H), 2.58 (dd, *J* = 14.3, 6.3 Hz, 1 H), 0.90 (d, *J* = 6.0 Hz, 2 H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 140.4 (C), 138.8 (2 × C), 129.1 (2 × CH), 129.0 (4 × CH), 128.5 (2 × CH), 128.4 (4 × CH), 127.2 (2 × CH), 126.1 (CH), 66.5 (CH), 66.0 (CH), 53.8 (2 × CH_2), 32.1 (CH_2), 20.0 (t, *J* = 76.4 Hz, CH_2D). MS (70 eV, EI) *m*/*z* (%): 346 (M^+, <1), 300 (80), 210 (90), 91 (100). HRMS (70 eV): calcd for $C_{24}H_{26}DNO$ (M^+), 346.2155; found, 346.2158. IR (neat): 3407, 3054, 2360, 1603, 1496, 1454, 1374 cm^{-1}. R_f = 0.22 (hexane/EtOAc 10:1). Anal. Calcd for $C_{24}H_{26}DNO$: C, 83.19; H, 8.14; N, 4.04; O, 4.62. Found: C, 83.06; H, 8.23; N, 4.08; O, 4.67.

(2*R*,3*S*)-1-Deuterium-3-(dibenzylamino)butan-2-ol (9a): white solid. $[\alpha]^{25}_D$ = +86.5 (*c* 0.99, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$): δ 7.29−7.14 (m, 10 H), 3.69 (d, *J* = 14.0 Hz, 2 H), 3.36 (d, *J* = 14.0 Hz, 2 H), 3.29−3.22 (m, 1 H), 2.55 (apparent qt, *J* = 6.7 Hz, 1 H), 1.84 (br s, 1 H), 1.12 (d, *J* = 6.2 Hz, 2 H), 1.03 (d, *J* = 6.8 Hz, 3 H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 140.0 (2 × C), 128.7 (4 × CH), 128.2 (4 × CH), 126.8 (2 × CH), 69.6 (CH), 58.4 (CH), 54.6 (2 × CH_2), 20.7 (t, *J* = 76.4 Hz, CDH_2), 8.5 (CH_3). MS (70 eV, EI) *m*/*z* (%): 255 ($M^+ - CH_3$, <1), 224 (88), 91 (100). HRMS (70 eV): calcd for $C_{17}H_{19}DNO$ ($M^+ - CH_3$), 255.1608; found, 255.1615. IR (neat): 3316, 2933, 2360, 1602, 1494, 1452, 1326 cm^{-1}. R_f = 0.31 (hexane/EtOAc 5:1). Anal. Calcd for $C_{18}H_{22}DNO$: C, 79.96; H, 8.95; N, 5.18; O, 5.92. Found: C, 79.81; H, 89.03; N, 5.14; O, 5.97.

(2*R*,3*S*)-1-Deuterium-3-(dibenzylamino)-4-phenylbutan-2-ol (9c): colorless oil. $[\alpha]^{25}_D$ = +22.5 (*c* 1.99, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$): δ 7.28−7.13 (m, 15 H), 3.83 (apparent q, *J* = 5.9 Hz, 1 H), 3.75 (d, *J* = 13.8 Hz, 2 H), 3.58 (d, *J* = 13.8 Hz, 2 H), 3.04 (dd, *J* = 13.3, 6.8 Hz, 1 H), 2.94 (dt, *J* = 6.7, 4.9 Hz, 1 H), 2.75 (dd, *J* = 13.3, 6.6 Hz, 1 H), 2.10 (br s, 1 H), 1.18 (d, *J* = 6.5 Hz, 2 H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 140.5 (C), 139.6 (2 × C), 129.2 (2 × CH), 128.7 (4 × CH), 128.3 (2 × CH), 128.2 (4 × CH), 126.9 (2 × CH), 125.9 (CH), 67.5 (CH), 64.0 (CH), 55.1 (2 × CH_2), 32.0 (CH_2), 20.6 (t, *J* = 76.4 Hz, CH_2D). MS (70 eV, EI) *m*/*z* (%): 346 (M^+, <1), 300 (32), 208 (27), 91 (100), 69 (41). HRMS (70 eV): calcd for $C_{24}H_{26}DNO$ (M^+), 346.2155; found, 346.2147. IR (neat): 3412, 3027, 2360, 1602, 1494, 1453, 1364 cm^{-1}. R_f = 0.36 (hexane/EtOAc 5:1). Anal. Calcd for $C_{24}H_{26}DNO$: C, 83.19; H, 8.14; N, 4.04; O, 4.62. Found: C, 83.25; H, 8.07; N, 4.09; O, 4.56.

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Supporting Information Available: ^{13}C NMR spectra of **4**, **5**, **6**, **7**, **8**, and **9**. This material is available free of charge via the Internet at http://pubs.acs.org.

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