A NEW CHIRAL MASKED FORM OF GLYOXAL DIIMINE

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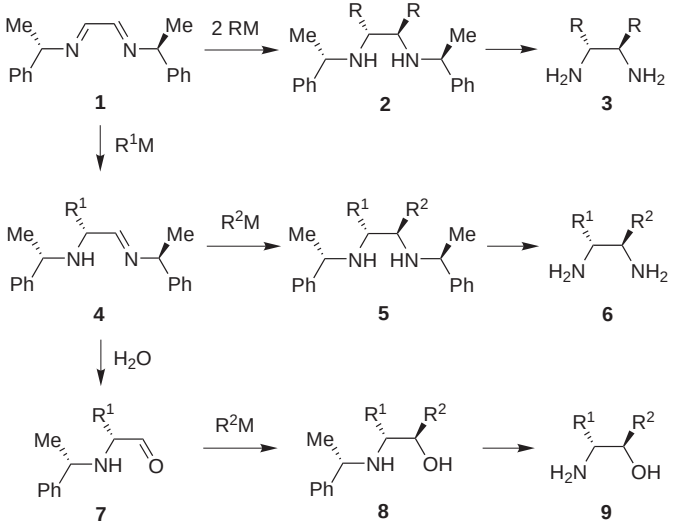
Dedicated to Professor Sergio Roffia on the occasion of his retirement.

(3*R*,4*R*,5*R*,6*R*)-3,6-Diphenyl-*N*,*N*′-bis[(*S*)-1-phenylethyl]octa-1,7-diene-4,5-diamine, by treatment with organolithium reagents RLi in controlled experimental conditions, underwent rearrangement and/or substitution of one or two branched allyl(s) by the R group(s). Hence, this diene behaves as a masked form of the chiral glyoxal diimine from that it is prepared, allowing the preparation of C_1-symmetric 1,2-disubstituted 1,2-diamines, which are generally not available by the direct addition of organometallic reagents to the diimine, and C_2-symmetric 1,2-diamines with good diastereoselectivities.

Keywords: Amines; Imines; Additions; Alkyllithium reagents; Diastereoselective synthesis; Lithium; Retro reactions; Rearrangements; Substitutions.

The chiral glyoxal diimine **1** and its equally available enantiomer are appealing chiral synthons for the preparation of enantiopure C_2-symmetric secondary 1,2-diamines **2** by the double addition of organometallic reagents; the corresponding primary 1,2-diamines **3** are obtained by reductive removal of the auxiliaries (Scheme 1). However, the scope of the reaction is somewhat limited[1–7]. Phenyl-[1], methyl-[1] and allylmagnesium[2] halides had to be added very slowly to **1** to achieve satisfactory to good diastereoselectivities[1], whereas alkylmagnesium halides preferably followed the *N*-alkylation pathway[8]. Using the more reactive organolithium compounds, the stereocontrol was only moderate; moreover, particular attention had to be taken to quench the reaction mixtures with degassed water to prevent formation of a monoimine through homolytic cleavage of the bis(adduct), *i.e.*, the dilithium bis(amide), and oxidation of the so formed α-amido radical[3]. The addition of allylzinc bromide[4] and β- or γ-substituted allylic zinc halides[5,6] led, with excellent diastereoselectivities to the corresponding octa-1,7-diene-4,5-diamine derivatives, which were successively used for

the construction of enantiopure cyclohexane-1,2-diamine[9,10] and cyclohex-4-ene-1,2-diamine[11,12] derivatives and 2,2′-bipyrrolidine[13].

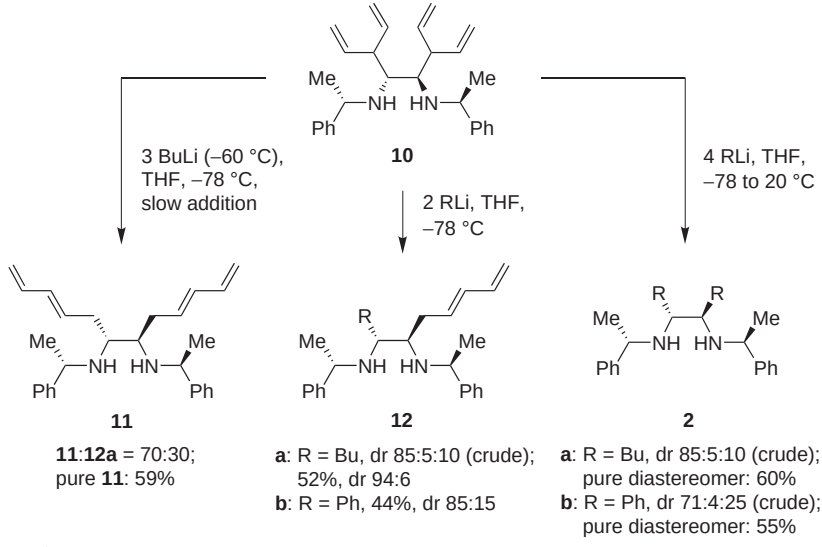


SCHEME 1

On the other hand, the preparation of the unsymmetrically 1,2-disubstituted 1,2-diamines **5** and **6** or the corresponding monosubstituted 1,2-diamines (R^2 = H) is hampered by the difficult control of the organometallic addition, *i.e.*, to avoid the second addition step of the same organometallic reagent R^1M to the intermediate aminoimine **4**. This is, in principle, possible, because imine **4** is less reactive than the starting diimine **1**, but in practice the successful control is achieved only when R^1M is poorly reactive for electronic and/or steric reasons. In fact, Roland described that *tert*-butylmagnesium chloride gave the monoadduct even at room temperature and reacted further at higher temperature to afford the corresponding 1,2-diamine **2** as a single diastereomer through a "kinetic resolution" process[7]. On the other hand, the use of a different organometallic reagent or hydride in the second addition step gave the unsymmetrically di- or monosubstituted 1,2-diamines **5** [14]. Analogously, we have reported the addition of 3-methylbut-2-en-1-ylzinc bromide could be stopped after the first addition, which occurred by allylic rearrangement. The unreacted imine **4** was isolated and then hydrolysed to aldehyde **7**. We also described a one-pot reaction to convert diimine **1** to the 1,2-disubstituted 1,2-amino alcohol **8**, involving a Barbier reaction with 4-bromo-2-methylbut-2-ene and zinc in the presence of cerium trichloride

heptahydrate. The reactive served also for *in situ* hydrolysis of the second imine function after the first organometallic step, forming an aldehyde which further underwent a Barbier-type allylation[15].

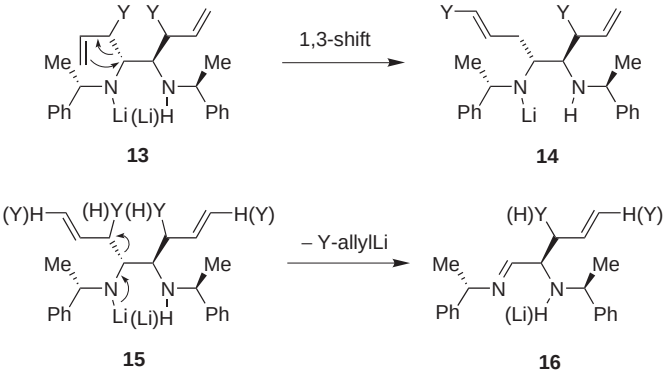
We have recently described an indirect, two-step approach to either C_2- and C_1-symmetric 1,2-disubstituted 1,2-diamines starting from diimine **1** and involving the intermediate (4*R*,5*R*)-*N*,*N*′-bis[(*S*)-1-phenylethyl]-3,6-divinylocta-1,7-diene-4,5-diamine[16] (**10**) (Scheme 2). This diene underwent rearrangement and/or substitution of one/two pentadienyl groups on treatment with 2–4 equivalents of an organolithium reagent in tetrahydrofuran. By a careful choice of the experimental conditions, C_1- or C_2-symmetric 1,2-disubstituted 1,2-diamines were prepared with good stereocontrol. In fact, using butyl- and phenyllithium with different molar ratios RLi/**10** and at different temperatures, either the isomeric straight-chain diamine **11** with complete retention of configuration, or the "mixed" diamines **12**, which bear one pentadienyl and one R substituent, or the C_2-symmetric 1,2-diamines **2a**, **2b** were predominantly formed in moderate yields and diastereoselectivities (ds 71–85%, by GC and/or ^{1}H NMR analysis).



SCHEME 2

We propose that the products **11**, **12** and **2** were formed through competitive pathways which are described in Scheme 3 for the model lithium amides **13** and **15** bearing branched or straight-chain Y-substituted allyl groups. The branched to straight-chain isomerisation of the allylic substituent should take place at low temperatures by 1,3-shift of the branched

homoallylic lithium amide with retention of configuration of the involved stereocentre and complete *E*-selectivity, affording **14**. On the other hand, the substitution of the group R coming from the organometallic reagent for the branched or straight-chain allylic substituent should involve the

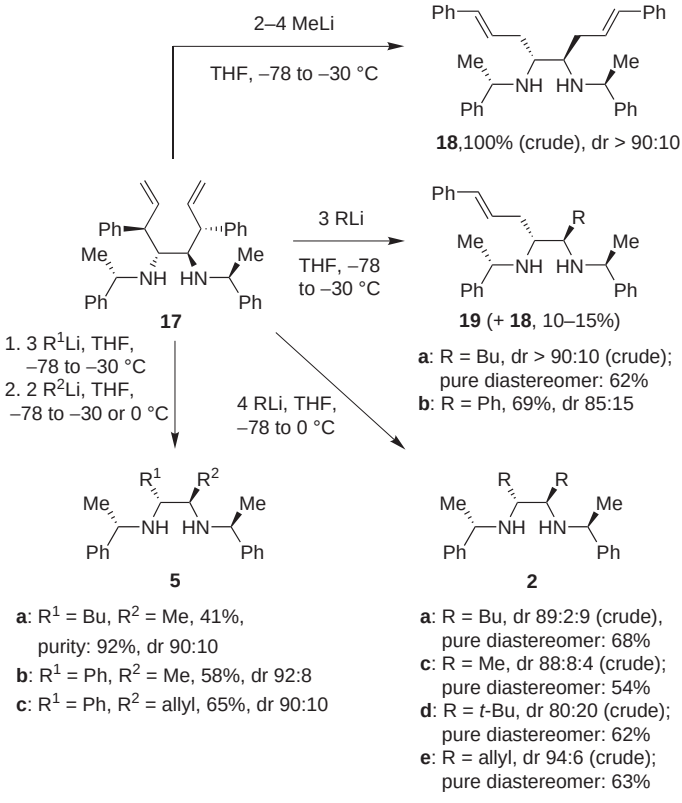


SCHEME 3

retro-pentadienyllithiation of **15** to form intermediate imine **16**. We attributed the occurrence of these processes even at low temperature to the steric and electronic properties of the pentadienyl substituent and envisioned that other branched bishomoallylic 1,2-diamines would behave similarly and provide even a better selectivity and/or stereocontrol. In other words, the nature of the substituent Y in **13** and **15** would affect the overall selectivity. Hence we investigated the behaviour of the branched diamine **17** [6], also available from the diimine **1**, towards organolithium reagents and also extended our efforts to the one-pot preparation of unsymmetrically 1,2-disubstituted 1,2-diamines **5** by the previously unexplored stepwise addition of two different organolithium reagents R^1Li and R^2Li (Scheme 4).

We first studied the reaction with methyllithium and observed that treatment of **17** with 2 equivalents of this reagent at –78 °C almost exclusively produced the isomeric straight-chain diamine[3] **18** with a small loss of configurational purity. Similar result was obtained when 4 equivalents of MeLi was used and the temperature was allowed to reach 0 °C, as no substitution process was observed. Conversely, the reactions with butyl- and phenyllithium (3 equivalents) at –78 to –30 °C afforded the "mixed" diamines **19a** and **19b**, respectively, resulting from sequential isomerisation and substitution processes. A mixture of products, including small amounts of the double substitution products, *e.g.* **2b**, and diastereomers (dr > 85:15) were formed in both cases; column chromatography gave pure **19a** with 62%

yield, whereas the diastereomers of **19b** were not separated in this way (69% yield, dr 85:15).



SCHEME 4

The preparation of C_2-symmetric 1,2-disubstituted 1,2-diamines by the double substitution process was then addressed. The reactions with 4 equivalents of several RLi reagents (R = methyl, butyl, *tert*-butyl, allyl) were carried out at –78 to 0 °C until completion of the substitution steps, generally with good stereocontrol. The best diastereomeric ratio was obtained with allyllithium (dr 94:6 for **2d**, in this case, the reaction was complete even at –30 °C), whereas *tert*-butyllithium gave the worst dr for **2c** (80:20). All the main diastereomers of **2a**–**2d** were isolated pure with satisfactory yields after chromatography on a silica gel column. No care was taken to isolate the minor diastereomers, but their C_2-symmetry was evident in the ^{1}H NMR spectra of the crude reaction products. The stereoisomers with *R*,*S* configuration of the NC*C*N carbons were not observed. This observation implies

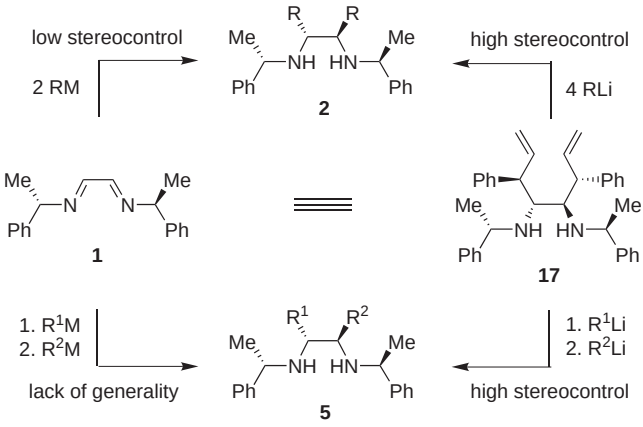
that the stereocontrol is lost only in the first substitution step, but the second step proceeds with (almost) complete diastereoselectivity.

Finally, we devoted some efforts to the preparation of 1,2-diamines **5** by controlled, sequential addition of two different organolithium reagents, R^1Li and R^2Li. In order to prepare diamines **5a** and **5b** (with one methyl substituent), considering the results of the previously described reactions with methyllithium, by which the monosubstitution did not take place at low temperatures, we performed the first addition with butyl- or phenyllithium (R^1Li) at –78 °C, and then used methyllithium as the second reagent (R^2Li). Both compounds **5a** and **5b** were obtained with an overall good efficiency and selectivity, but column chromatography did not allow to separate the diastereomers (dr 90:10 for **5a**, dr 92:8 for **5b**). On the other hand, diamine **5c**, with phenyl and allyl substituents, was obtained by the stepwise addition of the corresponding lithium reagents in a good yield (65% after column chromatography) and stereocontrol (dr > 85:15), although we did not succeed in separation of the diastereomers.

It should be underlined that the stereochemistry of the minor diastereomers of the C_1-symmetric diamines **19a**, **19b** and **5a**–**5c** was not determined with certainty, but the relative configuration of the NC*C*N stereocenters involved in the rearrangement and substitution steps is probably *syn*, in analogy with the configuration of the minor diastereomers of the C_2-symmetric diamines **2a**–**2d**. Minor amounts (<2%) of other diastereomers could have not been detected in the GC-MS and/or ^1H NMR analyses of the crude reaction mixtures. Separation of the diastereomers of **19b** and **5a**–**5c** was not accomplished by column chromatography, but the chance to purify the primary diamines or their suitable *N*,*N*′-derivatives after removal of the chiral auxiliaries is left.

The results described in this paper indicate that C_2-symmetric 1,2-diamines **2** and C_1-symmetric 1,2-diamines **19** and, in particular, **5** are available by the controlled (stepwise) addition of one or two different organolithium reagents to 1,2-diamine **17**. Hence, **17**, similarly to **10**, can be used as a novel "chiral masked form of glyoxal diimine"[17] (Scheme 5), although the diimine is probably not an intermediate in the reaction pathway. This two-step route (from **1** through **17**) is obviously plagued by the need to use an excess of organometallic reagents in the two steps; hence the preparation of symmetrically 1,2-disubstituted 1,2-diamines **2** is not attractive, although it generally provides a better diastereoselectivity than the direct double addition of organolithium reagents to **1**. Instead, the branched diamine **17** can be more advantageously exploited for the preparation of

C_1-symmetric 1,2-disubstituted 1,2-diamines **5** from **1**, as no general method has been so far described for this purpose.



SCHEME 5

EXPERIMENTAL

Melting points are uncorrected. Tetrahydrofuran and diethyl ether were before use distilled over sodium benzophenone ketyl and lithium aluminium hydride. Optical rotations were measured on a digital polarimeter in a 1-dm cell and $[\alpha]_D$-values are given in 10^{-1} deg cm^3 g^{-1}. ^{1}H NMR spectra were recorded on a Varian Gemini instrument at 300 or 200 MHz for samples in $CDCl_3$ which was stored over magnesium: ^{1}H chemical shifts are reported in ppm (δ-scale) relative to $CDCl_3$ (δ_H 7.27) and *J* values are given in Hz. MS spectra were taken at an ionizing voltage of 70 eV on a Hewlett–Packard 5970 or 5890 spectrometer with GLC injection. Chromatographic purifications were performed on columns of silica gel (Merck, 230–400 mesh) at medium pressure. The following organolithium reagents and organic compounds were purchased from Aldrich: butyl-, *tert*-butyl-, methyl- and phenyllithium, glyoxal trimeric dihydrate, (*S*)-1-phenylethylamine, allyltributyltin, allylbenzene. Diimine **1** [11,12] and diamine **17** [6] were prepared according to the already described procedures. Allyllithium was prepared from allyltributyltin and butylithium in ether. All the organometallic reactions were performed in a flame-dried apparatus under a static atmosphere of dry nitrogen. GC-MS and ^{1}H NMR were used to follow the progress of the reactions and the composition of the crude mixtures, including the diastereomeric ratios. However, products **18** and **19** decomposed during GC-MS analysis. Generally, only two diastereomers were observed in the reaction mixtures. The configuration of the known compounds **18** [3], **2a** [3], **2c** [1,3], **2d** [3,7] and **2e** [4] was assigned by their order of elution (GC) and spectral properties. For the new compounds **19a**, **19b** and **5a**–**5c**, minor amounts of other diastereomers (<2%) could have not been detected.

Synthesis of (*E*,*E*)-(4*R*,5*R*)- and (*E*,*E*)-(4*S*,5*S*)-1,8-Diphenyl-*N*,*N*′-bis[(*S*)-1-phenylethyl]octa-1,7-diene-4,5-diamine (**18**)

A solution of the 1,2-diamine **17** (2.50 g, 5 mmol) in anhydrous tetrahydrofuran (20 ml) was cooled to –78 °C. After 10 min MeLi (1.6 M in ether, 6.3 ml, 10 mmol) was added under

stirring during 15 min. The mixture was stirred for another 2 h, while allowing the temperature to rise slowly to –30 °C, then quenched with degassed water (10 ml). The organic phase was extracted with ether (3 × 20 ml), the collected ethereal layers were dried with anhydrous sodium sulfate and concentrated to give **18** [3] as an oil: 2.49 g (100%), (*E*,*E*)-(4*R*,5*R*)/(*E*,*E*)-(4*S*,5*S*) = 90:10 (GC-MS and ^{1}H NMR). Separation of the diastereomers was previously described[3].

Synthesis of Unsymmetrically 1,2-Disubstituted Diamines **19**

A solution of the 1,2-diamine **17** (2.50 g, 5 mmol) in anhydrous tetrahydrofuran (20 ml) was cooled to –78 °C. After 10 min the organolithium reagent (15 mmol) was added under stirring during 15 min. The mixture was stirred for another 2 h, while allowing the temperature to rise slowly to –30 °C, then quenched with degassed water (10 ml). The organic phase was extracted with ether (3 × 20 ml), the collected ethereal layers were dried with anhydrous sodium sulfate and concentrated to give an oily residue. Chromatography on an silica gel column, eluting with cyclohexane–ethyl acetate mixtures, allowed to separate the 1,2-diamines.

(E)-(4R,5R)- and (E)-(4S,5S)-1-Phenyl-N,N′-bis[(S)-1-phenylethyl]non-4-ene-4,5-diamine (**19a**): yellowish thick oil, 1.36 g (62%), (*E*)-(4*R*,5*R*)/(*E*)-(4*S*,5*S*) > 90:10 by ^{1}H NMR. $[\alpha]^{20}_D$ –144.0 (*c* 0.5, chloroform). For $C_{31}H_{40}N_2$ (440.7) calculated: 84.49% C, 9.15% H, 6.36% N; found: 84.52% C, 9.10% H, 6.33% N. ^{1}H NMR (200 MHz): 0.75 t, 3 H, *J* = 7.4 (CH_3); 0.83 m, 2 H (CH_2CH_3); 1.09 m, 2 H ($CH_2CH_2CH_3$); 1.31 d, 3 H, *J* = 6.6 ($CHCH_3$); 1.33 d, 3 H, *J* = 6.6 ($CHCH_3$); 1.43 m, 4 H ($CHCH_2$ and NH); 2.09 m, 1 H (CHN); 2.24 m, 3 H (CHN and CH_2CH=CH); 3.78 q, 1 H, *J* = 6.6 ($CHCH_3$); 3.85 q, 1 H, *J* = 6.6 ($CHCH_3$); 5.71 dt, 1 H, *J* = 16 and 7.2 (CH_2CH=CHPh); 5.88 d, 1 H, *J* = 16 (CH=CHPh); 7.05–7.20 m, 10 H (Ph).

(E)-(4R,5R)- and (E)-(4S,5S)-1,5-Diphenyl-N,N′-bis[(S)-1-phenylethyl]pent-1-ene-4,5-diamine (**19b**): yellowish thick oil, 1.59 g (69%), (*E*)-(4*R*,5*R*)/(*E*)-(4*S*,5*S*) = 85:15 by ^{1}H NMR. $[\alpha]^{20}_D$ –55.1 (*c* 0.48, chloroform). For $C_{33}H_{36}N_2$ (460.6) calculated: 86.04% C, 7.88% H, 6.08% N; found: 86.10% C, 7.89% H, 6.05% N. ^{1}H NMR (200 MHz): 1.25 d, 3 H, *J* = 6.6 ($CHCH_3$); 1.33 d, 3 H, *J* = 6.6 ($CHCH_3$); 1.92 broad, 2 H (NH); 2.09 m, 1 H (CHN); 2.04 m, 1 H (CH_2CH=CH); 2.24 m, 1 H (CH_2CH=CH); 2.51 dt, 1 H, *J* = 7.0 and 6.1 ($CHCH_2$); 3.20 d, 1 H, *J* = 7.0 (NCHPh); 3.42 q, 1 H, *J* = 6.6 ($CHCH_3$); 3.88 q, 1 H, *J* = 6.6 ($CHCH_3$); 5.82 dt, 1 H, *J* = 16.0 and 7.2 (CH_2CH=CHPh); 6.10 d, 1 H, *J* = 16.0 (CH=CHPh); 7.05–7.40 m, 20 H (Ph).

Synthesis of C_2-Symmetric 1,2-Disubstituted 1,2-Diamines **2**

An organolithium reagent RLi (10 mmol) was added during 15 min to the stirred solution of 1,2-diamine **17** (2.50 g, 5 mmol) in anhydrous tetrahydrofuran (20 ml) at –78 °C. After 1.5 h another 10 mmol of RLi was slowly added. The mixture was further stirred while allowing the temperature to rise slowly to –30 °C (R = allyl) or 0 °C during 1 h. Usual work-up gave the products **2a**, **2c**–**2e** as mixture of diastereomers (see Scheme 4), then chromatographic separation afforded the pure main diastereomers, which were identified by comparison of their physical and spectral properties with those reported for the authentic materials.

(5R,6R)-N,N′-Bis[(S)-1-phenylethyl]decane-5,6-diamine (**2a**): yellowish oil, 1.30 g (68%). $[\alpha]^{20}_D$ –138.8 (*c* 1.0, chloroform); ref.[3]: $[\alpha]^{20}_D$ –122.2 (*c* 1.3, chloroform).

(2R,3R)-N,N′-Bis[(S)-1-phenylethyl]butane-2,3-diamine (**2c**): yellowish oil, 0.80 g (54%). $[\alpha]^{20}_D$ –106.2 (*c* 0.41, chloroform); ref.[1] for the enantiomer of **2c**: $[\alpha]^{20}_D$ +128 (*c* 0.84, chloroform).

(3R,4R)-2,2,5,5-Tetramethyl-N,N′-bis[(S)-1-phenylethyl]hexane-2,3-diamine (**2d**): yellowish oil, 1.18 g (62%). $[\alpha]^{20}_D$ +55.6 (*c* 0.68, chloroform); ref.[3]: $[\alpha]^{20}_D$ +73.4 (*c* 1.8, chloroform); ref.[7]: $[\alpha]^{20}_D$ +31.6 (*c* 0.364, chloroform).

(4R,5R)-N,N′-Bis[(S)-1-phenylethyl]octa-1,7-diene-4,5-diamine (**2e**): white solid, 1.10 g (63%) after chromatography (silica gel column) and crystallization (methanol); m.p. 67–68 °C. $[\alpha]^{20}_D$ –120 (*c* 1.1, chloroform); ref.[4]: $[\alpha]^{20}_D$ –126.8 (*c* 2.04, chloroform).

Synthesis of Unsymmetrically 1,2-Disubstituted Diamines **5**

A solution of 1,2-diamine **17** (2.50 g, 5 mmol) in anhydrous tetrahydrofuran (20 ml) was cooled to –78 °C. After 10 min an R^1Li (15 mmol) was added under stirring during 15 min. The mixture was stirred for another 2 h, while allowing the temperature to rise slowly to –30 °C. Then the mixture was cooled again to –78 °C, an R^2Li (10 mmol) was slowly added and the stirred mixture was allowed to reach –30 °C (R^2 = allyl) or 0 °C during 1–2 h. After quenching with degassed water and usual work-up, the products were isolated by column chromatography, eluting with cyclohexane–ethyl acetate mixtures.

(2R,3R)- and (2S,3S)-N,N′-Bis[(S)-1-phenylethyl]heptane-2,3-diamine (**5a**): yellowish oil, 2.05 g (41%) (*ca* 8% of an impurity was detected by GC-MS and ^{1}H NMR), (2*R*,3*R*)/(2*S*,3*S*) = 90:10 by GC-MS. $[\alpha]^{20}_D$ –121.8 (*c* 0.42, chloroform). ^{1}H NMR (300 MHz): 0.83 t, 3 H, *J* = 7.2 ($CH_2C\mathbf{H}_3$); 0.88 d, 3 H, *J* = 6.3 ($CHC\mathbf{H}_3$); 0.98 m, 2 H ($C\mathbf{H}_2CH_3$); 1.20 m, 2 H ($C\mathbf{H}_2C_2H_5$); 1.27 s, 2 H (NH); 1.31 d, 3 H, *J* = 6.6 ($CHC\mathbf{H}_3$); 1.32 d, 3 H, *J* = 6.6 ($CHC\mathbf{H}_3$); 1.45 m, 2 H ($CHC\mathbf{H}_2$); 2.0 q, 1 H, *J* = 4.8 ($CHC\mathbf{H}CH_3$); 2.19 m, 1 H ($C\mathbf{H}CH_2$); 3.79 q, 1 H, *J* = 6.6 ($PhC\mathbf{H}CH_3$); 3.81 q, 1 H, *J* = 6.6 ($PhC\mathbf{H}CH_3$); 7.18–7.42 m, 10 H (Ph). EI-MS, *m/z* (rel.%): 190 (100) [M^+ – 148], 105 (81), 86 (48), 191 (16), 77 (15), 148 (14) [M^+ – 190].

(1R,2R)- and (1S,2S)-N,N′-Bis[(S)-1-phenylethyl]propane-1,2-diamine (**5b**): yellowish oil, 1.04 g (58%) (2–3% of an impurity was detected by GC-MS and ^{1}H NMR), (1*R*,2*R*)/(1*S*,2*S*) = 92:8 by ^{1}H NMR. $[\alpha]^{20}_D$ –219.7 (*c* 0.34, chloroform). For $C_{25}H_{30}N_2$ (358.5) calculated: 83.75% C, 8.43% H, 7.81% N; found: 83.85% C, 8.48% H, 7.78% N. ^{1}H NMR (300 MHz): 0.67 d, 3 H, *J* = 6.6 ($CHC\mathbf{H}_3$); 1.24 d, 3 H, *J* = 6.6 ($NCHC\mathbf{H}_3$); 1.38 d, 3 H, *J* = 6.9 ($NCHC\mathbf{H}_3$); 1.82 br, 2 H (NH); 2.43 dq, 1 H, J_1 = 9.0, J_2 = 6.6 ($CHC\mathbf{H}CH_3$); 3.0 d, 1 H, *J* = 9.0 (CHPh); 3.41 q, 1 H, *J* = 6.6 ($PhC\mathbf{H}CH_3$); 3.86 q, 1 H, *J* = 6.9 ($PhC\mathbf{H}CH_3$); 7.02 m, 2 H (Ph); 7.18–7.42 m, 13 H (Ph).

(1R,2R)- and (1S,2S)-1-Phenyl-N,N′-bis[1(S)-phenylethyl]pent-4-ene-1,2-diamine (**5c**): oil, 1.25 g (65%), (1*R*,2*R*)/(1*S*,2*S*) = 90:10 by ^{1}H NMR. $[\alpha]^{20}_D$ –185.4 (*c* 0.6, chloroform). For $C_{27}H_{32}N_2$ (384.5) calculated: 84.33% C, 8.39% H, 7.29% N; found: 84.35% C, 8.40% H, 7.28% N. ^{1}H NMR (300 MHz): 1.24 d, 3 H, *J* = 6.6 ($CHC\mathbf{H}_3$); 1.35 d, 3 H, *J* = 6.6 ($CHC\mathbf{H}_3$); 1.81 m, 1 H ($CHC\mathbf{H}_2$); 2.0 bs, 2 H (NH); 2.16 m, 1 H ($CHC\mathbf{H}_2$); 2.49 m, 1 H ($C\mathbf{H}CH_2$); 3.16 d, 1 H, *J* = 7.8 ($NC\mathbf{H}CHCH_2$); 3.41 q, 1 H, *J* = 6.6 ($C\mathbf{H}CH_3$); 3.86 q, 1 H, *J* = 6.6 ($C\mathbf{H}CH_3$); 4.85 d, 1 H, J_{trans} = 15.6 ($CH=C\mathbf{H}_2$); 4.93 d, 1 H, J_{cis} = 9.0 ($CH=C\mathbf{H}_2$); 5.58 ddt, 1 H, *J* = 9.0, 15.6 and 6.3 ($C\mathbf{H}=CH_2$); 7.10 m, 2 H (Ph); 7.20–7.40 m, 13 H (Ph). GC-MS (partial decomposition) EI-MS, *m/z* (rel.%): 105 (100), 174 (90), 70 (34), 106 (22), 79 (12), 77 (10), 210 (6), 135 (4), 343 (3).

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