Support Information for

Enantioselective Synthesis of Primary 1-(Aryl)alkylamines by Nucleophilic 1,2-Addition of Organolithium Reagents to Hydroxyoxime Ethers and Application to Asymmetric Synthesis of G-Protein-Coupled Receptor Ligands

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Experimental Section

General Methods. ^1H NMR spectra were recorded at 400 MHz or 300 MHz using residual CHCl_3 (7.26 ppm) as reference. ^{13}C NMR spectra were recorded at 100.6 MHz with CDCl_3 (77.05 ppm) as reference. A ^{19}F NMR spectrum was recorded at 376 MHz with benzotrifluoride (0.0 ppm) as reference. IR spectra were taken with an FTIR instrument. Mass spectra were measured at an ionizing voltage of 70 eV (EIMS) or performed on a spectrometer equipped with a positive electrospray ionization mode (ESI). Organic solvents used were dried by standard methods. Silica gel 60 (230–400 mesh, Merck) was used for column chromatography, and precoated silica gel 60F₂₅₄ plates (0.25 mm, Merck) were used for TLC.

(*R*)-Phenylethanediol [(*R*)-6]. To a stirred solution of methyl (*R*)-mandelate (**5**) (27.3 g, 164 mmol) in MeOH (820 mL) was added NaBH_4 (9.3 g, 264 mmol) at 0 °C. After 30 min at rt, the reaction was quenched with water (1000 mL) and the mixture was extracted with CHCl_3 (3 × 1000 mL). The combined extracts were washed with brine (500 mL), dried (MgSO_4), and concentrated in vacuo. Purification of the residue by chromatography (hexane–EtOAc, 2:1) gave (*R*)-**6** (22.0 g, 97%) as a colorless crystalline solid, which was recrystallized from EtOAc–hexane to give colorless needles: mp 67.5–68.5 °C (lit.^{1a} mp 66–67); $[\alpha]_{\text{D}}^{23} -64.0$ (*c* 1.0, CHCl_3) [lit.^{1b} $[\alpha]_{\text{D}} -62.7$ (*c* 0.11, CHCl_3), lit.^{1c} $[\alpha]_{\text{D}}^{20} -68$ (*c* 1, CHCl_3)].

2-[(*S*)-2-Hydroxy-1-phenylethyl]oxy}-1*H*-isoindole-1,3(2*H*)-dione (7**).** To a stirred solution of (*R*)-**6** (22.0 g, 159 mmol) in THF (1.6 L) containing *N*-hydroxyphthalimide (29.1 g, 175 mmol) and triphenylphosphine (45.9 g, 175 mmol) was added diethyl azodicarboxylate (30.0 mL, 191 mmol) at 0 °C. After being stirred for 2 d at rt, the mixture was concentrated in vacuo. To the residue was added water (100 mL) and the mixture was extracted with ether (3 × 500 mL). The combined organic phases were washed with brine (500 mL), dried (MgSO_4), and concentrated in vacuo. The crude material was purified by chromatography (hexane–EtOAc, 3:1) followed by recrystallization, providing **7** (36.5 g, 81%) as colorless needles: mp 114.5–115 °C (CHCl_3 –hexane); $[\alpha]_{\text{D}}^{20} +129$ (*c* 1.0, CHCl_3); IR (KBr) 3453 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 3.41 (1H, dd, *J* = 7.8 Hz), 3.95 (1H, s), 3.95 (1H, ddd *J* = 13.3, 8.1, 3.3 Hz), 4.19 (1H, ddd *J* = 13.1, 6.4, 3.3 Hz), 7.37–7.41 (3H, m), 7.74–7.77 (2H, m), 7.80–7.83 (2H, m); ^{13}C NMR (100.6 MHz, CDCl_3) δ 64.0 (CH_2), 90.8 (CH), 123.6 (CH, 2 carbons), 127.7 (CH, 2 carbons), 128.4 (C, 2 carbons), 129.0 (CH), 134.6 (CH, 2 carbons), 135.3 (C), 164.1 (CH, 2

carbons); EIMS m/z (relative intensity) 285 ($M^+ + 2$, 7), 252 (3), 165 (10), 164 (100), 163 (30), 147 (13), 121 (30), 103 (23), 91 (34), 77 (16). Anal. Calcd for $C_{16}H_{13}NO_4$: C, 67.84; H, 4.63; N, 4.94. Found: C, 67.63; H, 4.61; N, 4.92.

(S)-2-(Aminooxy)-2-phenylethanediol (8). A solution of **7** (36.5 g, 129 mmol) and hydrazine hydrate (26.9 mL, 477 mmol) in EtOH (644 mL) was heated at reflux for 1 h. The solution was concentrated in vacuo, to the residue was added 10% aqueous Na_2CO_3 (200 mL), and the mixture was extracted with $CHCl_3$ (3×500 mL). The combined organic phases were washed with brine (500 mL), dried ($MgSO_4$), and concentrated in vacuo. The oily residue was purified by chromatography ($CHCl_3$ –MeOH–concentrated NH_4OH , 500:9:1) to give **8** (19.6 g, 99%) as a colorless oil: $[\alpha]_D^{19} +87.2$ (c 0.4, $CHCl_3$); IR (neat) 3312, 3061, 3030, 1586 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$) δ 3.71 (1H, A part of ABX, $J = 12.1$, 3.0 Hz), 3.84 (1H, B part of ABX, $J = 12.1$, 8.3 Hz), 4.75 (1H, dd, $J = 8.3$, 3.2 Hz), 7.30–7.40 (5H, m); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 67.0 (CH_2), 87.5 (CH), 126.9 (CH, 2 carbons), 128.4 (CH), 128.8 (CH, 2 carbons), 138.1 (C); EIMS m/z (relative intensity) 307 ($M^+ + 1$, 1), 631 (98), 265 (100). Anal. Calcd for $C_8H_{11}NO_2$: C, 62.73; H, 7.24; N, 9.14. Found: C, 62.64; H, 7.29; N, 9.32.

General Procedure for Preparation of 1-Phenyl Hydroxyoxime Ethers 9a–f. To a stirred solution of **8** (200 mg, 1.31 mmol) in toluene (9 mL) was added appropriate arylaldehyde (1.31 mmol), and the mixture was heated at reflux for 30 min. After evaporation of the solvent in vacuo, the residue was diluted with ether (100 mL), washed with brine (20 mL), dried ($MgSO_4$), and concentrated. Purification of the crude material by chromatography (hexane–EtOAc, 3:1) gave **9a**, **9b**, **9c**, **9d**, **9e**, or **9f** (for yields, see Scheme 1).

Benzaldehyde (E)-O-[(1S)-2-Hydroxy-1-phenylethyl]oxime (9a). Colorless needles: mp 69.5–70.5 $^{\circ}C$ ($CHCl_3$ –hexane); $[\alpha]_D^{28} +16.6$ (c 0.9, $CHCl_3$); IR (KBr) 3388 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$) δ 2.57 (1H, br s), 3.86–3.90 (1H, A part of ABX, $J = 12.1$, 2.1 Hz), 3.98–4.04 (1H, B part of ABX, $J = 12.1$, 7.9 Hz), 5.37 (1H, dd, $J = 7.9$, 3.4 Hz), 7.28–7.35 (8H, m), 7.50–7.53 (2H, m), 8.19 (1H, s); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 66.8 (CH_2), 86.0 (CH), 126.9 (CH, 2 carbons), 127.2 (CH, 2 carbons), 128.1 (CH), 128.5 (CH, 2 carbons), 128.7 (C, 2 carbons), 130.1 (CH), 131.9 (C), 138.3 (C), 150.0 (CH); EIMS m/z (relative intensity) 241 (M^+ , 10), 210 (48), 120 (100), 104 (23), 90 (5), 77 (10). Anal. Calcd for $C_{15}H_{15}NO_2$: C, 74.67; H, 6.27; N, 5.81. Found: C, 74.86; H, 6.21; N, 5.76.

2-Methoxybenzaldehyde (E)-O-[(1S)-2-Hydroxy-1-phenylethyl]oxime (9b).

Colorless oil: $[\alpha]_D^{18} +36.9$ (*c* 1.1, CHCl₃); IR (neat) 3406 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 2.83 (1H, dd, *J* = 9.1, 3.6 Hz), 3.85 (3H, s), 3.90 (1H, ddd, *J* = 12.3, 9.1, 3.2 Hz), 4.05 (1H, ddd, *J* = 12.0, 8.1, 3.5 Hz), 5.39 (1H, dd, *J* = 8.0, 3.2 Hz), 6.88–6.92 (2H, m), 7.26–7.40 (6H, m), 7.68 (1H, dd, *J* = 7.7, 1.7 Hz), 8.60 (1H, s); ¹³C NMR (100.6 MHz, CDCl₃) δ 55.6 (CH₃), 67.4 (CH₂), 85.9 (CH), 111.2 (CH), 120.4 (CH), 120.9 (CH, 2 carbons), 127.0 (CH), 128.1 (CH), 128.5 (CH, 2 carbons), 131.5 (C), 138.3 (CH), 146.4 (C), 157.7 (CH); EIMS *m/z* (relative intensity) 272 (M⁺ + 1, 59), 271 (M⁺, 92), 241 (16), 240 (98), 151 (12), 134 (15), 120 (29), 118 (25), 107 (6). Anal. Calcd for C₁₆H₁₇NO₃: C, 70.83; H, 6.32; N, 5.16. Found: C, 70.67, H, 6.44; N, 5.23.

2-Methoxymethoxybenzaldehyde (E)-O-[(1S)-2-Hydroxy-1-phenylethyl]oxime (9c).

Colorless oil: $[\alpha]_D^{19} -29.0$ (*c* 1.1, CHCl₃); IR (neat) 3421 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 2.82 (1H, dd, *J* = 9.0, 3.7 Hz), 3.48 (3H, s), 3.91 (1H, ddd, *J* = 12.2, 9.0, 3.7 Hz), 4.06 (1H, ddd, *J* = 12.0, 8.0, 3.5 Hz), 5.22 (2H, s), 5.40 (1H, dd, *J* = 8.2, 3.5 Hz), 6.98 (1H, t, *J* = 7.4 Hz), 7.12 (1H, dd, *J* = 8.5, 3.2 Hz), 7.30–7.40 (6H, m) 7.71 (1H, dd, *J* = 7.8, 1.7 Hz), 8.62 (1H, s); ¹³C NMR (100.6 MHz, CDCl₃) δ 56.3 (CH₃), 67.3 (CH₂), 85.9 (CH₃), 114.7 (CH), 121.1 (C), 122.0 (CH), 126.9 (CH), 127.0 (CH, 2 carbons), 128.1 (CH), 128.5 (CH, 2 carbons), 131.4 (CH), 138.2 (C), 146.2 (CH), 155.4 (C); EIMS *m/z* (relative intensity) 302 (M⁺ + 1, 22), 301 (M⁺, 100), 269 (81), 180 (14), 164 (38), 150 (23), 134 (19), 121 (44), 119 (29), 102 (22), 91 (15), 77 (10). Anal. Calcd for C₁₇H₁₉NO₄: C, 67.76; H, 6.36; N, 4.65. Found: C, 67.71; H, 6.36; N, 4.54.

3-Methoxybenzaldehyde (E)-O-[(1S)-2-Hydroxy-1-phenylethyl]oxime (9d).

Colorless oil: $[\alpha]_D^{20} +20.6$ (*c* 1.0, CHCl₃); IR (neat) 3443 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 2.59 (1H, dd, *J* = 8.8, 4.1 Hz), 3.96 (3H, s), 4.06 (1H, ddd, *J* = 12.2, 8.8, 3.4 Hz), 5.54 (1H, dd, *J* = 7.9, 3.5 Hz), 7.07 (1H, ddd, *J* = 8.2, 2.5, 0.8 Hz), 7.25 (1H, d, *J* = 9.7 Hz), 7.40–7.53 (6H, m), 8.34 (1H, s); ¹³C NMR (100.6 MHz, CDCl₃) δ 55.4 (CH₃), 66.9 (CH₂), 86.0 (CH), 111.6 (CH), 116.4 (CH), 120.3 (CH), 127.0 (CH, 2 carbons), 128.2 (CH), 128.6 (CH, 2 carbons), 129.8 (CH), 133.2 (C), 138.3 (C), 150.0 (CH), 159.9 (C); EIMS *m/z* (relative intensity) 273 (M⁺ + 2, 30), 271 (M⁺, 100), 240 (21), 152 (22), 134 (19), 120 (16), 103 (11), 91 (9), 77 (20). Anal. Calcd for C₁₆H₁₇NO₃: C, 70.83; H, 6.32; N, 5.16. Found: C, 70.59; H, 6.34; N, 5.00.

3-(Methylsulfanyl)benzaldehyde (E)-O-[(1S)-2-Hydroxy-1-phenylethyl]oxime (9e).

Colorless oil: $[\alpha]_D^{19} -1.08$ (*c* 1.0, CHCl₃); IR (neat) 3419 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ

2.42 (1H, dd, $J = 8.7, 4.0$ Hz), 2.47 (3H, s), 3.91 (1H, ddd, $J = 12.1, 8.7, 3.5$ Hz), 4.03 (1H, ddd, $J = 12.1, 8.2, 4.1$ Hz), 5.39 (1H, dd, $J = 7.9, 3.6$ Hz), 7.24–7.43 (9H, m), 8.17 (1H, s); ^{13}C NMR (100.6 MHz, CDCl_3) δ 15.8 (CH_3), 66.8 (CH_2), 86.2 (CH), 124.1 (CH), 124.8 (CH), 126.9 (CH, 2 carbons), 128.1 (CH), 128.2 (CH), 128.6 (CH, 2 carbons), 129.1 (C), 132.5 (CH), 138.2 (C), 139.4 (C), 149.5 (CH); EIMS m/z (relative intensity) 287 (M^+ , 100), 256 (5), 149 (51), 133 (9), 91 (2). Anal. Calcd for $\text{C}_{16}\text{H}_{17}\text{NO}_2\text{S}$: C, 66.87; H, 5.96; N, 4.87. Found: C, 66.72; H, 6.03; N, 4.75.

4-Methylbenzaldehyde (*E*)-*O*-[(1*S*)-2-Hydroxy-2-phenylethyl]oxime (9f). Colorless needles: mp 80.5–82 °C (CHCl_3 –hexane); $[\alpha]_{\text{D}}^{19} +17.6$ (c 0.6, CHCl_3); IR (neat) 3314, 2920 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 2.36 (3H, s), 2.56 (1H, dd, $J = 8.8, 3.7$ Hz), 3.93 (1H, ddd, $J = 12.1, 8.9, 3.3$ Hz), 4.03 (1H, ddd, $J = 11.8, 7.9, 3.5$ Hz), 5.38 (1H, dd, $J = 8.0, 3.3$ Hz), 7.16 (1H, d, $J = 8.0$ Hz), 7.25–7.45 (8H, m), 8.19 (1H, s); ^{13}C NMR (100.6 MHz, CDCl_3) δ 21.5 (CH_3), 67.1 (CH_2), 85.9 (CH), 127.0 (CH, 2 carbons), 127.2 (CH, 2 carbons), 128.2 (CH), 128.6 (CH, 2 carbons), 129.1 (C), 129.5 (CH, 2 carbons), 138.3 (C), 140.5 (C), 150.0 (CH); EIMS m/z (relative intensity) 255 (M^+ , 16), 254 ($\text{M} - 1$, 48), 243 (26), 223 (83), 134 (26), 120 (57), 102 (42), 90 (67), 76 (31). Anal. Calcd for $\text{C}_{16}\text{H}_{17}\text{NO}_2$: C, 75.27; H, 6.71; N, 5.49. Found: C, 75.12; H, 6.69; N, 5.28.

(2*R*)-2-(Methoxymethoxy)-2-phenylethanol (10). To a stirred mixture of **5** (1.0 g, 6.02 mmol) and *N,N*-diisopropylethylamine (3.15 mL, 18.1 mmol) in CH_2Cl_2 (60 mL) was added chloromethylmethylether (1.14 mL, 15 mmol) at 0 °C, and the mixture was stirred for 2 days at rt. After addition of water (40 mL), the organic layer was separated, and the aqueous layer was extracted with CHCl_3 (3×100 mL). The combined organic extracts were washed with brine (100 mL), dried (MgSO_4), and concentrated in vacuo. Chromatography (hexane–EtOAc, 3:1) of the oily residue gave methyl (2*R*)-(methoxymethoxy)(phenyl)ethanoate (1.15 g, 91%) as a colorless oil: $[\alpha]_{\text{D}}^{22} -151$ (c 1.3, CHCl_3); ^1H NMR (400 MHz, CDCl_3) δ 3.37 (3H, s), 3.70 (3H, s), 4.68 (1H, 1/2 ABq, $J = 6.9$ Hz), 4.74 (1H, 1/2 ABq, $J = 6.9$ Hz), 5.17 (1H), 7.30–7.36 (3H, m), 7.44–7.45 (2H, m); ^{13}C NMR (100.6 MHz, CDCl_3) δ 52.3 (CH_3), 55.9 (CH_3), 76.7 (CH), 95.0 (CH_2), 127.4 (CH, 2 carbons), 128.6 (CH, 2 carbons), 128.7 (CH), 136.1 (C), 171.2 (C); EIMS m/z (relative intensity) 210 (M^+ , 2), 179 (40), 149 (82), 121 (29), 105 (32), 89 (16), 77 (24). Anal. Calcd for $\text{C}_{11}\text{H}_{14}\text{O}_4$: C, 62.85; H, 6.71. Found: C, 62.75; H, 6.71.

The above product (200 mg, 1.20 mmol) was dissolved in THF (16 mL) and to this solution was added LiAlH_4 (91 mg, 2.40 mmol) at 0 °C with stirring and the resultant suspension was stirred for 0 °C. After 1 h, the reaction was quenched with saturated aqueous NH_4Cl (5 mL), the mixture was filtered through Celite, and the filtrate was concentrated in vacuo. Chromatography (hexane–EtOAc, 3:1) of the residue gave **10** (198 mg, 99%) as a colorless oil: $[\alpha]_{\text{D}}^{22} -204$ (*c* 1.3, CHCl_3); IR (neat) 3443 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 2.48 (1H, dd, $J = 9.1, 4.0$ Hz), 3.41 (3H, s), 3.68 (1H, ddd, $J = 15.6, 9.1, 3.7$ Hz), 3.77 (1H, ddd, $J = 15.6, 9.1, 3.7$ Hz), 4.67 (1H, 1/2 ABq, $J = 6.8$ Hz), 4.72 (1H, 1/2 ABq, $J = 6.8$ Hz), 4.72 (1H, dd, $J = 8.2, 4.4$ Hz), 7.28–7.38 (5H, m); ^{13}C NMR (100.6 MHz, CDCl_3) δ 55.9 (CH_3), 67.4 (CH_2), 80.4 (CH), 95.2 (CH_2), 127.0 (CH, 2 carbons), 128.2 (CH), 128.6 (CH, 2 carbons), 138.5 (C); EIMS m/z (relative intensity) 183 ($\text{M}^+ + 1$, 10), 165 (22), 151 (100), 133 (49), 121 (87), 105 (44), 90 (41), 77 (32); HRMS (EI) calcd for $\text{C}_9\text{H}_{11}\text{O}_2$ ($\text{M}^+ - \text{OMe}$) 151.0759, found 151.0774.

2-[[*(2R)*-2-(Methoxymethoxy)-2-phenylethyl]oxy]-1*H*-isoindole-1,3(*2H*)-dione (11).

To a stirred solution of **10** (10.0 g, 60.2 mmol) in THF (300 mL) containing *N*-hydroxyphthalimide (15.0 g, 90.3 mmol) and triphenylphosphine (24.0 g, 90.3 mmol) was added dropwise diethyl azodicarboxylate (15.2 mL, 96.3 mmol) at 0 °C. After being stirred for 9 h at rt, the mixture was concentrated in vacuo and to the residue was added water (100 mL). The mixture was extracted with ether (3 × 200 mL) and the combined organic phases were washed with brine (50 mL), dried (MgSO_4), and concentrated in vacuo. The residue was purified by chromatography (hexane–EtOAc, 3:1) to give **11** (19.4 g, 99%) as a colorless oil: $[\alpha]_{\text{D}}^{22} -62.5$ (*c* 1.2, CHCl_3); IR (neat) 1731 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 3.35 (3H, s), 4.25–4.28 (1H, A part of ABX, $J = 10.4, 3.2$ Hz), 4.43–4.48 (1H, B part of ABX, $J = 10.4, 8.4$ Hz), 4.64 (1H, 1/2 ABq, $J = 6.7$ Hz), 4.75 (1H, 1/2 ABq, $J = 6.7$ Hz), 5.07 (1H, dd, $J = 8.5, 3.2$ Hz), 7.26–7.40 (5H, m), 7.71–7.73 (2H, m), 7.80–7.83 (2H, m); ^{13}C NMR (100.6 MHz, CDCl_3) δ 55.7 (CH_3), 76.2 (CH), 81.4 (CH_2), 95.0 (CH_2), 123.5 (CH, 2 carbons), 127.1 (CH) (2 carbons), 128.4 (C), 128.6 (CH, 2 carbons), 128.9 (CH, 2 carbons), 134.4 (CH, 2 carbons), 137.6 (CH, 2 carbons), 163.3 (C, 2 carbons); EIMS m/z (relative intensity) 328 ($\text{M}^+ + 1$, 2), 296 (10), 266 (100), 252 (49), 160 (19), 147 (10), 119 (13), 103 (37), 91 (19), 77 (10).

(1*R*)-2-(Aminooxy)-1-phenylethanol (12). To a solution of **11** (19.6 g, 60 mmol) in MeOH (600 mL) was added concentrated HCl (2 mL), and the solution was stirred at rt for 30

min. After concentration in vacuo, the residue was dissolved in EtOH (220 mL) and to this solution was added hydrazine hydrate (4.0 g, 66 mmol). The resulting mixture was heated at reflux for 1 h and concentrated in vacuo. To the residue obtained was added 10% aqueous Na_2CO_3 (200 mL) and the mixture was extracted with CHCl_3 (3 $\times$ 300 mL). The combined organic phases were washed with brine (300 mL), dried (MgSO_4), and concentrated in vacuo to give the oily residue, which was subjected to chromatography (CHCl_3 –MeOH–concentrated NH_4OH , 500:9:1) to give **12** (9.2 g, 99%) as colorless cubics: mp 58–59.5 °C (CHCl_3 –hexane); $[\alpha]_{\text{D}}^{27}$ –45.5 (*c* 1.0, CHCl_3); IR (KBr) 3304, 3221 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 3.71–3.74 (1H, A part of ABX, *J* = 11.8, 8.3 Hz), 3.79–3.83 (1H, B part of ABX, *J* = 11.8, 2.8 Hz), 3.92 (1H, br s), 5.05 (1H, dd, *J* = 8.3, 2.8 Hz), 5.55 (2H, br s), 7.26–7.40 (5H, m); ^{13}C NMR (100.6 MHz, CDCl_3) δ 73.9 (CH), 80.1 (CH_2), 126.2 (CH, 2 carbons), 127.8 (CH), 128.5 (CH, 2 carbons), 140.5 (C); EIMS *m/z* (relative intensity) 154 ($\text{M}^+ + 1$, 6), 151 ($\text{M}^+ - 2$, 46), 135 (37), 121 (32), 105 (65), 91 (61), 77 (68). Anal. Calcd for $\text{C}_{17}\text{H}_{19}\text{NO}_2$: C, 62.73; H, 7.24; N, 9.14. Found: C, 62.71; H, 7.06; N, 8.86.

General Procedure for Preparation of 2-Phenyl Hydroxyoxime Ethers 13a–e. To a stirred solution of **12** (300 mg, 1.95 mmol) in toluene (13 mL) was added appropriate arylaldehyde (1.95 mmol), and the mixture was heated at reflux for 30 min. The reaction mixture was concentrated in vacuo and diluted with ether (150 mL). The ethereal solution was washed with brine (30 mL), dried (MgSO_4), and concentrated to give the crude product, which was purified by chromatography (hexane–EtOAc, 3:1) to give **13a**, **13b**, **13c**, **13d**, or **13e** (for yields, see Scheme 2).

Benzaldehyde *O*–[(1*R*)-2-Hydroxy-1-phenylethyl]oxime (13a). Colorless oil: $[\alpha]_{\text{D}}^{25}$ +11.1 (*c* 0.7, CHCl_3); IR (neat) 3419 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 3.16 (1H, br s), 4.06–4.12 (1H, A part of ABX, *J* = 11.8, 8.5 Hz), 4.16–4.20 (1H, B part of ABX, *J* = 11.8, 3.0 Hz), 4.99 (1H, br d, *J* = 6.3 Hz), 7.17–7.31 (8H, m), 7.44–7.47 (2H, m), 8.02 (1H, s); ^{13}C NMR (100.6 MHz, CDCl_3) δ 73.5 (CH), 79.2 (CH_2), 126.3 (CH, 2 carbons), 127.2 (CH, 2 carbons), 128.8 (CH, 2 carbons), 130.2 (CH), 131.7 (C), 140.2 (C), 149.8 (CH); EIMS *m/z* (relative intensity) 242 ($\text{M}^+ + 1$, 8), 224 (18), 107 (100), 106 (60), 121 (8), 77 (26). Anal. Calcd for $\text{C}_{15}\text{H}_{15}\text{NO}_2$: C, 74.67; H, 6.27; N, 5.81. Found: C, 74.47; H, 6.31; N, 5.88.

2-Methoxybenzaldehyde *O*–[(2*R*)-2-Hydroxy-2-phenylethyl]oxime (13b). Colorless

oil: $[\alpha]_D^{22} +5.95$ (*c* 1.0, CHCl₃); IR (neat) 3421 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 3.52 (1H, br s), 3.86 (3H, s) 4.20–4.25 (1H, A part of ABX, *J* = 12.0, 8.6 Hz), 4.39–4.43 (1H, B part of ABX, *J* = 12.0, 2.8 Hz), 5.16 (1H, dt, *J* = 8.5, 2.3 Hz), 6.92 (1H, d, *J* = 8.3 Hz), 7.05 (1H, m), 6.99 (1H, t, *J* = 7.5 Hz), 7.31–7.47 (6H, m), 7.76 (1H, dd, *J* = 7.7, 1.6 Hz), 8.56 (1H, s); ¹³C NMR (100.6 MHz, CDCl₃) δ 55.6 (CH₃), 73.8 (CH), 79.1 (CH₂), 111.2 (CH), 120.3 (C), 120.8 (CH), 126.3 (CH, 2 carbons), 126.9 (CH), 127.8 (CH), 128.5 (CH, 2 carbons), 131.5 (CH), 140.2 (C), 146.3 (CH), 157.8 (C); EIMS *m/z* (relative intensity) 272 (*M*⁺ + 1, 6), 271 (*M*⁺, 56), 152 (11), 134 (26), 119 (37), 107 (27), 92 (10), 77 (11). Anal. Calcd for C₁₆H₁₇NO₃: C, 70.83; H, 6.32; N, 5.16. Found: C, 70.79; H, 6.33; N, 5.15.

2-(Methoxymethoxy)benzaldehyde *O*-(2*R*)-2-Hydroxy-2-phenylethyl]oxime (13c).

Colorless oil: $[\alpha]_D^{25} -7.15$ (*c* 0.6, CHCl₃); IR (neat) 3445 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 3.35 (1H, br s), 3.36 (3H, s), 4.12 (1H, A part of ABX, *J* = 12.0, 8.6 Hz), 4.20 (1H, 1H, B part of ABX, *J* = 12.0, 2.9 Hz), 5.02 (1H, br d, *J* = 8.5 Hz), 5.10 (2H, s), 6.91 (1H, t, *J* = 7.5 Hz), 7.03 (1H, d, *J* = 8.3 Hz), 7.17–7.34 (6H, m), 7.69 (1H, d, *J* = 7.8 Hz), 8.47 (1H, s); ¹³C NMR (100.6 MHz, CDCl₃) δ 56.3 (CH₃), 73.6 (CH), 79.1 (CH₂), 94.6 (CH₂), 114.7 (CH), 121.0 (C), 122.0 (CH), 126.3 (CH, 2 carbons), 126.7 (CH), 127.8 (CH), 128.4 (CH, 2 carbons), 131.5 (CH), 140.2 (C), 146.0 (CH), 155.4 (C); EIMS *m/z* (relative intensity) 301 (*M*⁺, 8), 165 (17), 162 (71), 148 (21), 120 (26), 107 (100), 79 (44). Anal. Calcd for C₁₇H₁₉NO₄: C, 67.76; H, 6.36; N, 4.65. Found: C, 67.65; H, 6.36; N, 4.65.

3-Methoxybenzaldehyde *O*-[(2*R*)-2-Hydroxy-2-phenylethyl]oxime (13d).

Colorless oil: $[\alpha]_D^{22} +9.89$ (*c* 1.0, CHCl₃); IR (neat) 3443 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 3.18–3.20 (1H, br m), 3.93 (3H, s), 4.29–4.34 (1H, A part of ABX, *J* = 11.9, 8.7 Hz), 4.39–4.43 (1H, B part of ABX, *J* = 11.9, 2.9 Hz), 5.23 (1H, dt, *J* = 8.6, 2.5 Hz), 7.05 (1H, m), 7.22–7.25 (2H, m), 7.34–7.54 (5H, m), 8.22 (1H, s); ¹³C NMR (100.6 MHz, CDCl₃) δ 55.4 (CH₃), 73.5 (CH), 79.3 (CH₂), 111.5 (CH), 116.6 (CH), 120.3 (CH), 126.4 (CH, 2 carbons), 128.0 (CH), 128.6 (CH, 2 carbons), 129.9 (CH), 133.1 (C), 140.2 (C), 149.9 (CH), 160.0 (C); EIMS *m/z* (relative intensity) 272 (*M*⁺ + 1, 100), 271 (*M*⁺, 58), 254 (26), 253 (26), 134 (2), 77 (1); Anal. Calcd for C₁₆H₁₇NO₃: C, 70.83; H, 6.32; N, 5.16. Found: C, 70.78; H, 6.33; N, 5.17.

4-Methylbenzaldehyde *O*-[(2*R*)-2-Hydroxy-2-phenylethyl]oxime (13e).

Colorless needles: mp 37–38 °C (CHCl₃–hexane); $[\alpha]_D^{25} +4.1$ (*c* 0.9, CHCl₃); IR (neat) 3444 cm⁻¹; ¹H

NMR (400 MHz, CDCl_3) δ 2.26 (3H, s), 3.11 (1H, br s), 4.05–4.11 (1H, A part of ABX, $J = 14.3$, 10.3 Hz), 4.16–4.22 (1H, B part of ABX, $J = 14.3$, 3.5 Hz), 5.61 (1H, dt, $J = 10.3$, 2.8 Hz), 7.02–7.40 (9H, m), 8.02 (1H, s); ^{13}C NMR (100.6 MHz, CDCl_3) δ 21.5 (CH_3), 73.6 (CH), 79.1 (CH_2), 126.3 (CH, 2 carbons), 127.2 (2 carbons) (CH), 128.5 (CH, 2 carbons), 128.9 (C), 129.6 (CH, 2 carbons), 140.2 (C), 140.6 (C), 149.9 (CH); EIMS m/z (relative intensity) 256 ($M + 1$, 84), 238 (28), 136 (20), 119 (100), 107 (43), 91 (48), 78 (36), 77 (28). Anal. Calcd for $\text{C}_{16}\text{H}_{17}\text{NO}_2$: C, 75.27; H, 6.71; N, 5.49. Found: C, 75.36; H, 6.78; N, 5.31

General Procedure for Addition of Organolithium Reagents to the (S)-Hydroxyoxime Ethers 9a–f and 13a–e. To a stirred ice-cold solution of **9a–f** or **13a–e** (0.369 mmol) in toluene (3.7 mL) was added a solution of an appropriate organolithium in Et_2O (0.369 mmol) under N_2 atmosphere. After 5 min, the mixture was cooled to the temperature shown in Tables 1 and 2 and a solution of an additional organolithium reagent in Et_2O (0.485 mL, 0.553 mmol) was again added, and then stirring was continued for another 25 min at the temperature described in Tables 1 and 2. The reaction was quenched with water (10 mL) under cooling, the organic layer separated, and the aqueous layer extracted with ether (3×25 mL). The combined extracts were washed with brine (30 mL), dried (MgSO_4), and concentrated in vacuo. The residue was purified by chromatography (hexane– EtOAc , 4:1) to give a diastereomeric mixture of product **14–29** (for yields and ratios, see Tables 1 and 2).

(2S)-2-Phenyl-2-([(1R)-1-phenylethyl]amino)oxyethanol [(R,S)-14]. Colorless needles: mp 57.5–59 °C (EtOAc –hexane); $[\alpha]_{\text{D}}^{18} +70.9$ (c 0.4, CHCl_3); IR (KBr) 3388 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 1.45 (3H, d, $J = 6.6$ Hz), 3.65–3.69 (1H, A part of ABX, $J = 12.0$, 3.0 Hz), 3.84–3.89 (1H, B part of ABX, $J = 12.0$, 8.6 Hz), 4.72 (1H, q, $J = 6.6$ Hz), 4.74 (1H, dd, $J = 8.6$, 2.9 Hz), 7.20–7.32 (10H, m); ^{13}C NMR (100.6 MHz, CDCl_3) δ 19.7 (CH_3), 60.6 (CH), 67.7 (CH_2), 85.3 (CH), 126.9 (CH, 2 carbons), 127.3 (CH, 2 carbons), 127.8 (CH), 128.1 (CH), 128.5 (CH, 2 carbons), 128.6 (CH, 2 carbons), 138.4 (C), 142.0 (C); EIMS m/z (relative intensity) 258 ($M^+ + 1$, 6), 136 (30), 120 (19), 119 (11), 90 (9), 76 (18). Anal. Calcd for $\text{C}_{16}\text{H}_{19}\text{NO}_2$: C, 74.68; H, 7.44; N, 5.44. Found: C, 74.65; H, 7.47; N, 5.22.

(2S)-2-Phenyl-2-([(1S)-1-phenylethyl]amino)oxyethanol [(S,S)-14]. Colorless oil: $[\alpha]_{\text{D}}^{20} +18.2$ (c 0.3, CHCl_3); IR (neat) 3401 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 1.34 (3H, d, $J = 6.8$ Hz), 3.50 (1H, A part of ABX, $J = 12.1$, 8.6 Hz), 3.61 (1H, B part of ABX, $J = 12.1$, 3.1 Hz),

4.24 (1H, q, $J = 6.8$ Hz), 4.69 (1H, dd, $J = 8.6, 3.1$ Hz), 7.28–7.43 (10H, m); ^{13}C NMR (100.6 MHz, CDCl_3) δ 20.0 (CH_3), 60.8 (CH), 67.1 (CH_2), 86.3 (CH), 126.8 (CH, 2 carbons), 127.3 (CH, 2 carbons), 127.8 (CH), 127.9 (CH), 128.6 (CH, 2 carbons), 128.7 (CH, 2 carbons), 138.7 (C), 142.8 (C); ESI m/z $[\text{M} + \text{H}]^+$ 258; HRMS (ESI) calcd for $\text{C}_{16}\text{H}_{20}\text{NO}_2$ $[\text{M} + \text{H}]^+$ 258.1494, found 258.1474.

(2S)-2-([[(1R)-1-(2-Methoxyphenyl)ethyl]amino]oxy)-2-phenylethanol [(R,S)-15].

Colorless oil: $[\alpha]_{\text{D}}^{18} +50.3$ (c 0.1, CHCl_3); IR (neat) 3397 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 1.43 (3H, d, $J = 6.8$ Hz), 3.63–3.66 (1H, A part of ABX, $J = 11.4, 2.8$ Hz), 3.77–3.82 (1H, B part of ABX, $J = 11.4, 8.9$ Hz), 3.80 (3H, s), 4.57 (1H, q, $J = 6.8$ Hz), 4.83 (1H, dd, $J = 8.8, 2.7$ Hz), 6.85 (1H, d, $J = 8.0$ Hz), 6.93 (1H, m), 7.20–7.31 (7H, m); ^{13}C NMR (100.6 MHz, CDCl_3) δ 18.0 (CH_3), 55.4 (CH), 55.7 (CH_3), 68.4 (CH_2), 85.6 (CH), 111.1 (CH), 121.2 (CH), 127.1 (CH, 2 carbons), 127.8 (CH, 2 carbons), 127.8 (CH), 127.9 (CH), 128.4 (CH, 2 carbons), 130.0 (C), 138.8 (C), 157.2 (C); EIMS m/z (relative intensity) 288 ($\text{M}^+ + 1$, 100), 135 (20). Anal. Calcd for $\text{C}_{17}\text{H}_{21}\text{NO}_3$: C, 71.06; H, 7.37; N, 4.87. Found: C, 70.87; H, 7.45; N, 4.81.

(2S)-2-([[(1S)-1-(2-Methoxyphenyl)ethyl]amino]oxy)-2-phenylethanol [(S,S)-15].

Colorless oil: $[\alpha]_{\text{D}}^{20} +30.2$ (c 0.1, CHCl_3); IR (neat) 3398 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 1.39 (3H, d, $J = 6.8$ Hz), 3.61 (1H, A part of ABX, $J = 12.0, 2.2$ Hz), 3.76 (1H, B part of ABX, $J = 12.0, 8.7$ Hz), 3.85 (3H, s), 4.61 (1H, q, $J = 6.8$ Hz), 4.80 (1H, dd, $J = 8.7, 2.2$ Hz), 6.89 (1H, d, $J = 8.2$ Hz), 6.98 (1H, t, $J = 7.4$ Hz), 7.26–7.38 (7H, m); ^{13}C NMR (125 MHz, CDCl_3) δ 18.1 (CH_3), 54.9 (CH), 55.4 (CH_3), 67.9 (CH_2), 85.6 (CH), 110.8 (CH), 120.9 (CH), 126.8 (CH), 126.9 (CH, 2 carbons), 127.8 (CH), 128.1 (CH), 128.5 (CH, 2 carbons), 130.0 (C), 138.8 (C), 157.2 (C); ESI m/z $[\text{M} + \text{H}]^+$ 288; HRMS (ESI) calcd for $\text{C}_{17}\text{H}_{22}\text{NO}_3$ $[\text{M} + \text{H}]^+$ 288.1600, found 288.1599

(2S)-2-([[(1R)-1-(3-Methoxyphenyl)ethyl]amino]oxy)-2-phenylethanol [(R,S)-16].

Colorless needles: mp 55–57.5 °C (CHCl_3 –hexane); $[\alpha]_{\text{D}}^{27} +69.7$ (c 0.5, CHCl_3); IR (KBr) 3406 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 1.42 (3H, d, $J = 8.0$ Hz), 3.41–3.46 (1H, A part of ABX, $J = 14.6, 3.5$ Hz), 3.55 (3H, s), 3.63–3.70 (1H, B part of ABX, $J = 14.6, 3.5$ Hz), 4.04 (1H, q, $J = 8.0$ Hz), 4.74 (1H, dd, $J = 10.4, 3.5$ Hz), 7.24–7.26 (2H, m), 7.32 (1H, d, $J = 9.2$ Hz), 7.71–7.83 (6H, m); ^{13}C NMR (100.6 MHz, CDCl_3) δ 20.1 (CH_3), 55.6 (CH), 61.0 (CH_3), 68.0 (CH_2), 85.7 (CH), 113.3 (CH), 113.4 (CH), 127.2 (CH, 2 carbons), 128.4 (CH), 128.8 (CH, 2 carbons), 129.9 (CH), 138.4 (C), 143.8 (C), 159.8 (C); EIMS m/z (relative intensity) 288 ($\text{M}^+ + 1$, 100), 167 (20), 150

(6), 134 (60), 121 (8), 102 (8), 91 (4), 77 (4). Anal. Calcd for $C_{17}H_{19}NO_2$: C, 71.06; H, 7.37; N, 4.87. Found: C, 70.95; H, 7.21; N, 4.87.

(2S)-2-([(1S)-1-(3-Methoxyphenyl)ethyl]amino)oxy]-2-phenylethanol [(S,S)-16].

Colorless oil: $[\alpha]_D^{20} +36.9$ (*c* 0.4, $CHCl_3$); IR (neat) 3408 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$) δ 1.31 (3H, d, $J = 6.7$ Hz), 3.51 (1H, A part of ABX, $J = 12.0, 2.9$ Hz), 3.63 (1H, B part of ABX, $J = 12.0, 8.8$ Hz), 3.83 (3H, s), 4.20 (1H, q, $J = 6.7$ Hz), 4.71 (1H, dd, $J = 8.8, 2.9$ Hz), 6.85–7.37 (9H, m); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 19.8 (CH_3), 55.3 (CH_3), 60.8 (CH), 67.1 (CH_2), 86.3 (CH), 113.0 (CH), 113.1 (CH), 120.0 (CH), 126.8 (CH, 2 carbons), 128.2 (CH), 128.6 (CH, 2 carbons), 129.7 (CH), 138.7 (C), 144.6 (C), 160.0 (C); ESI m/z $[M + H]^+$ 288; HRMS (ESI) calcd for $C_{17}H_{22}NO_3$ $[M + H]^+$ 288.1600, found 288.1605.

(2S)-2-([(1R)-1-[3-(Methylsulfanyl)phenyl]ethyl]amino)oxy]-2-phenylethanol [(R,S)-17]. Colorless oil: $[\alpha]_D^{19} +63.0$ (*c* 0.82, $CHCl_3$); IR (neat) 3397 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$) δ 1.43 (3H, d, $J = 6.6$ Hz), 2.46 (3H, s), 3.67–3.70 (1H, A part of ABX, $J = 11.9, 1.7$ Hz), 3.84–3.89 (1H, B part of ABX, $J = 11.9, 8.6$ Hz), 4.18 (1H, q, $J = 6.6$ Hz), 4.75 (1H, dd, $J = 8.6, 3.0$ Hz), 7.08 (1H, m), 7.17–7.31 (8H, m); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 15.9 (CH_3), 19.7 (CH_3), 60.6 (CH), 67.6 (CH_2), 85.4 (CH), 124.0 (CH), 125.6 (CH), 125.9 (CH), 126.9 (CH, 2 carbons), 128.2 (CH), 128.5 (CH, 2 carbons), 129.1 (CH), 138.4 (C), 138.8 (C), 142.9 (C); EIMS m/z (relative intensity) 304 ($M^+ + 1$, 9), 303 (M^+ , 100), 279 (26), 182 (61), 165 (10), 150 (95), 135 (12), 120 (20), 102 (30), 91 (35), 76 (12). Anal. Calcd for $C_{17}H_{21}NO_2S$: C, 67.29; H, 6.98; N, 4.62. Found: C, 66.97; H, 6.94; N, 4.80.

(2S)-2-([(1S)-1-[3-(Methylsulfanyl)phenyl]ethyl]amino)oxy]-2-phenylethanol [(S,S)-17]. Colorless oil: $[\alpha]_D^{19} -25.7$ (*c* 0.6, $CHCl_3$); IR (neat) 3402 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$) δ 1.31 (3H, d, $J = 6.8$ Hz), 2.50 (3H, s), 3.51 (1H, A part of ABX, $J = 12.0, 3.0$ Hz), 3.62 (1H, A part of ABX, $J = 12.0, 8.6$ Hz), 4.19 (1H, q, $J = 6.8$ Hz), 4.68 (1H, dd, $J = 8.6, 3.0$ Hz), 7.18–7.38 (9H, m); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 15.9 (CH_3), 19.7 (CH_3), 60.7 (CH), 67.1 (CH_2), 86.3 (CH), 123.9 (CH), 125.6 (CH), 125.9 (CH), 126.8 (CH, 2 carbons), 128.2 (CH), 128.6 (CH, 2 carbons), 129.1 (CH), 138.7 (C), 143.6 (C), 146.7 (C); ESI m/z $[M + H]^+$ 304; HRMS (ESI) calcd for $C_{17}H_{22}NO_2S$ $[M + H]^+$ 304.1372, found 304.1384.

(2S)-2-([(1R)-1-(4-Methylphenyl)ethyl]amino)oxy]-2-phenylethanol [(R,S)-18].

Colorless needles: mp 69–70.5 °C ($CHCl_3$ –hexane); $[\alpha]_D^{18} +80.0$ (*c* 0.2, $CHCl_3$); IR (KBr) 3407

cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 1.44 (3H, d, $J = 6.6$ Hz), 2.32 (3H, s), 3.65–3.69 (1H, A part of ABX, $J = 12.0, 2.8$ Hz), 3.84–3.89 (1H, B part of ABX, $J = 12.0, 8.7$ Hz), 4.17 (1H, q, $J = 6.6$ Hz), 4.75 (1H, dd, $J = 8.6, 2.8$ Hz), 5.48 (1H, br s), 7.11–7.32 (9H, m); ^{13}C NMR (100.6 MHz, CDCl_3) δ 19.7 (CH_3), 21.2 (CH_3), 60.3 (CH), 67.8 (CH_2), 85.3 (CH), 126.9 (CH, 2 carbons), 127.2 (CH, 2 carbons), 128.1 (CH), 128.5 (CH, 2 carbons), 129.3 (CH, 2 carbons), 137.5 (C), 138.4 (C), 139.0 (C); EIMS m/z (relative intensity) 273 ($\text{M}^+ + 2$, 5), 271 (M^+ , 35), 150 (15), 134 (7), 102 (15), 91 (14), 76 (10). Anal. Calcd for $\text{C}_{17}\text{H}_{21}\text{NO}_2$: C, 75.25; H, 7.80; N, 5.16. Found: C, 75.24; H, 7.76; N, 4.93.

(2S)-2-([[(1S)-1-(4-Methylphenyl)ethyl]amino]oxy)-2-phenylethanol [(S,S)-18].

Colorless oil: $[\alpha]_{\text{D}}^{20} +40.4$ (c 1.1, CHCl_3); IR (neat) 3396 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 1.32 (3H, d, $J = 6.7$ Hz), 2.36 (3H, s), 3.50 (1H, A part of ABX, $J = 12.0, 3.0$ Hz), 3.61 (1H, B part of ABX, $J = 12.0, 8.6$ Hz), 4.19 (1H, q, $J = 6.7$ Hz), 4.69 (1H, dd, $J = 8.6, 3.0$ Hz), 7.17–7.38 (9H, m); ^{13}C NMR (100.6 MHz, CDCl_3) δ 19.7 (CH_3), 21.2 (CH_3), 60.5 (CH), 67.3 (CH_3), 86.2 (CH), 126.8 (CH, 2 carbons), 127.2 (CH, 2 carbons), 128.1 (CH), 128.6 (CH, 2 carbons), 129.4 (CH, 2 carbons), 137.5 (C), 138.7 (C), 139.7 (C); ESI m/z $[\text{M} + \text{H}]^+$ 272; HRMS (ESI) calcd for $\text{C}_{17}\text{H}_{22}\text{NO}_2$ $[\text{M} + \text{H}]^+$ 272.1651, found 272.1642.

(2S)-2-([[(1R)-1-[2-(Methoxymethoxy)phenyl]-2-propenyl]amino]oxy)-2-

phenylethanol [(R,S)-19]. Colorless oil: $[\alpha]_{\text{D}}^{20} +25.3$ (c 1.0, CHCl_3); IR (neat) 3387 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 3.45 (3H, s), 3.65 (1H, A part of ABX, $J = 12.0, 2.9$ Hz), 3.80 (1H, B part of ABX, $J = 12.0, 8.9$ Hz), 4.86 (1H, dd, $J = 8.9, 2.9$ Hz), 5.08 (1H, d, $J = 7.2$ Hz), 5.19 (1H, 1/2 ABq, $J = 6.7$ Hz), 5.20 (1H, 1/2 ABq, $J = 6.7$ Hz), 5.31 (2H, dd, $J = 17.2, 10.3$ Hz), 6.17 (1H, ddd, $J = 17.2, 10.3, 7.2$ Hz), 6.97 (1H, td, $J = 7.5, 1.0$ Hz), 7.09 (1H, m), 7.20–7.31 (7H, m); ^{13}C NMR (100.6 MHz, CDCl_3) δ 56.3 (CH_3), 62.7 (CH), 67.5 (CH_2), 86.0 (CH), 94.7 (CH_2), 114.5 (CH), 117.7 (CH_2), 122.1 (CH), 126.8 (CH, 2 carbons), 128.1 (CH), 128.5 (CH, 2 carbons), 128.5 (C), 129.0 (CH), 129.1 (CH), 137.6 (CH), 138.8 (C), 154.9 (C); ESI m/z $[\text{M} + \text{H}]^+$ 330; HRMS (ESI) calcd for $\text{C}_{19}\text{H}_{24}\text{NO}_4$ $[\text{M} + \text{H}]^+$ 330.1705, found 330.1701. Anal. Calcd for $\text{C}_{19}\text{H}_{23}\text{NO}_4$: C, 69.28; H, 7.04; N, 4.25. Found: C, 69.51; H, 7.04; N, 4.23.

(2S)-2-([[(1S)-1-[2-(Methoxymethoxy)phenyl]-2-propenyl]amino]oxy)-2-phenylethanol

[(S,S)-19]. Colorless oil: IR (neat) 3396 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 3.44 (3H, s), 3.58 (1H, A part of ABX, $J = 12.0, 2.9$ Hz), 3.73 (1H, B part of ABX, $J = 12.0, 8.7$ Hz), 4.79 (1H,

dd, $J = 8.7, 2.9$ Hz), 5.04 (1H, d, $J = 7.2$ Hz), 5.18 (2H, s), 5.26 (2H, dd, $J = 17.4, 10.2$ Hz), 6.06 (1H, ddd, $J = 17.4, 10.2, 7.2$ Hz), 7.01 (1H, td, $J = 7.5, 1.0$ Hz), 7.10 (1H, m), 7.20–7.29 (7H, m); ^{13}C NMR (100.6 MHz, CDCl_3) δ 56.2 (CH_3), 62.7 (CH), 67.3 (CH_2), 85.6 (CH), 94.7 (CH_2), 114.6 (CH), 117.7 (CH), 122.1 (CH), 126.9 (CH, 2 carbons), 128.0 (CH), 128.5 (CH, 2 carbons), 128.8 (C), 128.8 (C), 128.9 (CH), 136.9 (CH), 138.6 (C), 154.9 (C); ESI m/z $[\text{M} + \text{H}]^+$ 330; HRMS (ESI) calcd for $\text{C}_{19}\text{H}_{24}\text{NO}_4$ $[\text{M} + \text{H}]^+$ 330.1705, found 330.1701.

(2S)-2-({[(R)-[2-(Methoxymethoxy)phenyl](phenyl)methyl]amino}oxy)-2-phenylethanol [(R,S)-20]. Colorless oil: $[\alpha]_{\text{D}}^{19} +66.9$ (c 1.0, CHCl_3); IR (neat) 3433 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 2.16 (1H, br s), 3.30 (3H, s), 3.45 (1H, A part of ABX, $J = 11.9, 3.1$ Hz), 3.57 (1H, B part of ABX, $J = 11.9, 9.0$ Hz), 4.78 (1H, dd, $J = 9.0, 3.1$ Hz), 5.11 (1H, 1/2 ABq, $J = 6.7$ Hz), 5.14 (1H, 1/2 ABq, $J = 6.7$ Hz), 5.70 (1H, s), 6.14 (1H, br s), 6.87 (1H, t, $J = 7.6$ Hz), 7.01 (1H, d, $J = 8.1$ Hz), 7.14–7.33 (12H, m), 7.46 (2H, d, $J = 7.4$ Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 56.1 (CH_3), 62.9 (CH), 66.7 (CH_2), 86.2 (CH), 94.3 (CH_2), 114.0 (CH), 121.9 (CH), 126.7 (CH, 2 carbons), 127.4 (CH), 128.0 (CH), 128.2 (CH, 2 carbons), 128.4 (CH, 2 carbons), 128.5 (CH, 2 carbons), 128.7 (CH), 128.8 (CH), 129.0 (CH), 138.8 (C), 140.8 (C), 154.5 (C), 171.1 (C); ESI m/z $[\text{M} + \text{H}]^+$ 380; HRMS (ESI) calcd for $\text{C}_{23}\text{H}_{26}\text{NO}_4$ $[\text{M} + \text{H}]^+$ 380.1862, found 380.1826. Anal. Calcd for $\text{C}_{23}\text{H}_{25}\text{NO}_4$: C, 72.80; H, 6.64; N, 3.69. Found: C, 72.82; H, 6.67; N, 3.66.

(2S)-2-({[(S)-[2-(Methoxymethoxy)phenyl](phenyl)methyl]amino}oxy)-2-phenylethanol [(S,S)-20]. Colorless oil: $[\alpha]_{\text{D}}^{19} +29.9$ (c 0.3, CHCl_3); IR (neat) 3419 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 2.42 (1H, br s), 3.31 (3H, s), 3.58 (1H, A part of ABX, $J = 12.1, 3.1$ Hz), 3.71 (1H, B part of ABX, $J = 12.1, 8.8$ Hz), 4.84 (1H, dd, $J = 8.8, 3.1$ Hz), 5.12 (1H, 1/2 ABq, $J = 6.7$ Hz), 5.17 (1H, 1/2 ABq, $J = 6.7$ Hz), 5.70 (1H, s), 6.14 (br s), 7.05–7.37 (12H, m), 7.55 (1H, d, $J = 7.7$ Hz), 7.66 (1H, d, $J = 7.7$ Hz); ^{13}C NMR (100.6 MHz, CDCl_3) δ 56.2 (CH_3), 63.1 (CH), 66.9 (CH_2), 85.9 (CH), 94.6 (CH_2), 114.3 (CH), 121.9 (CH), 126.9 (CH, 2 carbons), 127.5 (CH), 128.0 (CH), 128.2 (CH, 2 carbons), 128.4 (CH, 2 carbons), 128.55 (CH, 2 carbons), 128.6 (CH), 129.8 (C), 138.6 (C), 140.0 (C), 154.7 (C); ESI m/z $[\text{M} + \text{H}]^+$ 380; HRMS (ESI) calcd for $\text{C}_{23}\text{H}_{26}\text{NO}_4$ $[\text{M} + \text{H}]^+$ 380.1862, found 380.1876.

(1R)-1-Phenyl-2-({[(1S)-1-phenylethyl]amino}oxo)ethanol [(S,R)-21]. Colorless oil: $[\alpha]_{\text{D}}^{23} -18.0$ (c 0.2, CHCl_3); IR (neat) 3396 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 1.42 (3H, d, $J =$

6.7 Hz), 3.69 (1H, A part of ABX, $J = 12.0, 8.5$ Hz), 3.76 (1H, B part of ABX, $J = 12.0, 2.4$ Hz), 4.21 (1H, q, $J = 6.7$ Hz), 4.92 (1H, dd, $J = 8.5, 2.4$ Hz), 7.26–7.37 (10H, m); ^{13}C NMR (125 MHz, CDCl_3) δ 19.6 (CH_3), 60.7 (CH), 74.0 and 74.3 (CH), 78.9 and 79.0 (CH_2), 126.2 (CH, 2 carbons), 127.3 (CH, 2 carbons), 127.7 (CH), 127.9 (CH), 128.4 (CH, 2 carbons), 128.7 (CH, 2 carbons), 140.4 (C), 142.1 (C); EIMS m/z (relative intensity) 259 ($\text{M}^+ + 2$, 14), 258 ($\text{M}^+ + 1$, 100), 77 (6). Anal. Calcd for $\text{C}_{16}\text{H}_{19}\text{NO}_2$: C, 74.68; H, 7.44; N, 5.44. Found: C, 74.41; H, 7.50; N, 5.42.

(1R)-1-Phenyl-2-([(1R)-1-phenylethyl]amino)oxoethanol [(R,R)-21]. Colorless oil: $[\alpha]_{\text{D}}^{23} -41.2$ (c 1.4, CHCl_3); IR (neat) 3407 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 1.38 (3H, d, $J = 6.7$ Hz), 3.59 (1H, A part of ABX, $J = 12.0, 8.7$ Hz), 3.67 (1H, B part of ABX, $J = 12.0, 2.4$ Hz), 4.15 (1H, q, $J = 6.7$ Hz), 4.88 (1H, dd, $J = 8.7, 2.4$ Hz), 7.19–7.33 (10H, m); ^{13}C NMR (125 MHz, CDCl_3) δ 19.6 (CH_3), 60.7 (CH), 74.1 (CH), 78.9 (CH_2), 126.2 (CH, 2 carbons), 127.3 (CH, 2 carbons), 127.6 (CH), 127.9 (CH), 128.4 (CH, 2 carbons), 128.7 (CH, 2 carbons), 140.5 (C), 142.1 (C); ESI m/z $[\text{M} + \text{H}]^+$ 258; HRMS (ESI) calcd for $\text{C}_{16}\text{H}_{20}\text{NO}_2$ $[\text{M} + \text{H}]^+$ 258.1494, found 258.1499.

(1R)-2-([(1S)-1-(2-Methoxyphenyl)ethyl]amino)oxy-1-phenylethanol [(S,R)-22]. Colorless oil: IR (neat) 3418 cm^{-1} ; $[\alpha]_{\text{D}}^{20} -13.1$ (c 0.2, CHCl_3); ^1H NMR (500 MHz, CDCl_3) δ 1.47 (3H, d, $J = 6.8$ Hz), 3.72 (1H, A part of ABX, $J = 12.2, 8.6$ Hz), 3.78 (1H, B part of ABX, $J = 12.2, 2.6$ Hz), 4.55 (1H, q, $J = 6.8$ Hz), 5.03 (1H, dd, $J = 8.6, 2.6$ Hz), 6.90 (1H, d, $J = 8.2$ Hz), 6.96 (1H, t, $J = 7.9$ Hz), 7.24–7.46 (7H, m); ^{13}C NMR (125 MHz, CDCl_3) δ 17.9 (CH_3), 55.5 (CH_3), 55.5 (CH), 74.9 (CH), 78.6 (CH_2), 110.8 (CH), 120.9 (CH), 126.2 (CH, 2 carbons), 127.6 (CH), 128.0 (CH), 128.4 (CH, 2 carbons), 128.8 (CH, 2 carbons), 129.5 (C), 140.7 (C), 157.3 (C); EIMS m/z (relative intensity) 288 ($\text{M}^+ + 1$, 12), 287 (M^+ , 100), 91 (2). Anal. Calcd for $\text{C}_{17}\text{H}_{21}\text{NO}_3$: C, 71.06; H, 7.37; N, 4.87. Found: C, 71.03; H, 7.27; N, 4.94.

(1R)-2-([(1R)-1-(2-Methoxyphenyl)ethyl]amino)oxy-1-phenylethanol [(R,R)-22]. Colorless oil: $[\alpha]_{\text{D}}^{20} -21.0$ (c 0.6, CHCl_3); IR (neat) 3388 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 1.31 (3H, d, $J = 6.8$ Hz), 3.72 (1H, A part of ABX, $J = 12.2, 8.7$ Hz), 3.79 (1H, B part of ABX, $J = 12.2, 2.2$ Hz), 4.56 (1H, q, $J = 6.8$ Hz), 5.02 (1H, dd, $J = 8.7, 2.2$ Hz), 6.90 (1H, d, $J = 8.2$ Hz), 6.97 (1H, t, $J = 7.4$ Hz), 7.25–7.40 (7H, m); ^{13}C NMR (125 MHz, CDCl_3) δ 17.8 (CH_3), 55.3 (CH_3), 74.6 (CH), 78.5 (CH_2), 110.8 (CH), 120.8 (CH), 126.2 (CH, 2 carbons), 127.5 (CH), 127.9 (CH), 128.3 (CH, 2 carbons), 128.6 (CH, 2 carbons), 129.7 (C), 140.7 (C), 157.2 (C); ESI m/z $[\text{M}$

+ H]⁺ 288; HRMS (ESI) calcd for C₁₇H₂₂NO₃ [M + H]⁺ 288.1600, found 288.1613.

(1R)-2-([(1S)-1-(3-Methoxyphenyl)ethyl]amino)oxy)-1-phenylethanol [(S,R)-23].

Colorless oil: IR (neat) 3419 cm⁻¹; [α]_D²⁰ -19.7 (*c* 0.2, CHCl₃); ¹H NMR (500 MHz, CDCl₃) δ 1.43 (3H, d, *J* = 6.7 Hz), 3.63 (1H, A part of ABX, *J* = 12.2, 8.8 Hz), 3.74 (1H, B part of ABX, *J* = 12.2, 2.4 Hz), 3.82 (3H, s), 4.18 (1H, q, *J* = 6.7 Hz), 4.96 (1H, dd, *J* = 8.8, 2.4 Hz), 6.84–7.34 (9H, m); ¹³C NMR (125 MHz, CDCl₃) δ 20.0 (CH₃), 55.6 (CH₃), 61.1 (CH), 74.4 (CH), 79.2 (CH₂), 113.4 (CH), 113.5 (CH), 119.9 (CH), 126.5 (CH, 2 carbons), 128.0 (CH), 128.7 (CH, 2 carbons), 130.1 (CH), 140.5 (C), 143.9 (C), 159.9 (C); EIMS *m/z* (relative intensity) 284 (M⁺ + 1, 48), 134 (100), 91 (12). Anal. Calcd for C₁₇H₂₁NO₃: C, 71.06; H, 7.37; N, 4.87. Found: C, 71.11; H, 7.34; N, 4.82.

(1R)-2-([(1S)-1-(3-Methoxyphenyl)ethyl]amino)oxy)-1-phenylethanol [(R,R)-23].

Colorless oil: [α]_D²⁰ -25.6 (*c* 0.3, CHCl₃); IR (neat) 3418 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 1.41 (3H, d, *J* = 6.7 Hz), 3.70 (1H, A part of ABX, *J* = 12.0, 8.4 Hz), 3.78 (1H, B part of ABX, *J* = 12.0, 2.7 Hz), 4.94 (1H, dd, *J* = 8.4, 2.7 Hz), 6.82–7.34 (9H, m); ¹³C NMR (125 MHz, CDCl₃) δ 20.0 (CH₃), 55.6 (CH₃), 61.0 (CH), 74.5 (CH), 79.3 (CH₂), 113.3 (CH), 113.4 (CH), 120.0 (CH), 126.5 (CH, 2 carbons), 128.0 (CH), 128.7 (CH, 2 carbons), 130.0 (CH), 140.6 (C), 143.9 (C), 159.9 (C); ESI *m/z* [M + H]⁺ 288; HRMS (ESI) calcd for C₁₇H₂₂NO₃ [M + H]⁺ 288.1600, found 288.1581.

(1R)-2-([(1S)-1-(4-Methylphenyl)ethyl]amino)oxo)-1-phenylethanol [(S,R)-24].

Colorless oil: [α]_D²⁰ -22.2 (*c* 0.1, CHCl₃); IR (neat) 3734 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 1.41 (3H, d, *J* = 6.7 Hz), 2.28 (3H, s), 3.70 (1H, A part of ABX, *J* = 12.0, 8.4 Hz), 3.76 (1H, B part of ABX, *J* = 12.0, 2.6 Hz), 4.17 (1H, q, *J* = 6.7 Hz), 4.93 (1H, dd, *J* = 8.4, 2.6 Hz), 7.15–7.33 (9H, m); ¹³C NMR (125 MHz, CDCl₃) δ 19.6 (CH₃), 21.2 (CH₃), 60.4 (CH), 74.4 (CH), 78.9 (CH₂), 126.2 (CH, 2 carbons), 127.1 (CH, 2 carbons), 127.7 (CH), 128.4 (CH, 2 carbons), 129.4 (CH, 2 carbons), 137.5 (C), 139.3 (C), 140.6 (C); EIMS *m/z* (relative intensity) 272 (M⁺ + 1, 13), 271 (M⁺, 100), 272 (13), 91 (7), 77 (4). Anal. Calcd for C₁₇H₂₁NO₂: C, 75.25; H, 7.80; N, 5.16. Found: C, 75.20; H, 7.88; N, 5.18.

(1R)-2-([(1R)-1-(4-Methylphenyl)ethyl]amino)oxo)-1-phenylethanol [(R,R)-24].

Colorless oil: [α]_D²⁰ -40.4 (*c* 0.9, CHCl₃); IR (neat) 3397 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 1.43 (3H, d, *J* = 6.7 Hz), 2.35 (3H, s), 3.64 (1H, A part of ABX, *J* = 12.1, 8.8 Hz), 3.73 (1H, B

part of ABX, $J = 12.1, 2.4$ Hz), 4.17 (1H, q, $J = 6.7$ Hz), 4.96 (1H, dd, $J = 8.7, 2.4$ Hz), 7.16–7.34 (9H, m); ^{13}C NMR (125 MHz, CDCl_3) δ 19.9 (CH_3), 21.5 (CH_3), 60.7 (CH), 74.5 (CH), 79.2 (CH_2), 126.5 (CH, 2 carbons), 127.5 (CH, 2 carbons), 128.0 (CH), 128.7 (CH, 2 carbons), 129.7 (CH, 2 carbons); ESI m/z $[\text{M} + \text{H}]^+$ 272; HRMS (ESI) calcd for $\text{C}_{17}\text{H}_{22}\text{NO}_2$ $[\text{M} + \text{H}]^+$ 272.1651, found 272.1664.

(1R)-1-Phenyl-2-([(1S)-1-phenylpentyl]amino)oxy)ethanol [(S,R)-25]. Colorless oil: $[\alpha]_{\text{D}}^{21} -20.4$ (c 0.2, CHCl_3); IR (neat) 3409, 2955, 2930 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 0.86 (3H, t, $J = 7.3$ Hz), 1.17 (2H, m), 1.68 (2H, m), 1.84 (2H, m), 3.57 (1H, A part of ABX, $J = 12.2, 8.8$ Hz), 3.67 (1H, B part of ABX, $J = 12.2, 2.4$ Hz), 4.00 (1H, dd, $J = 8.7, 5.7$ Hz), 4.90 (1H, dd, $J = 8.8, 2.4$ Hz), 7.24–7.38 (10H, m); ^{13}C NMR (100.6 MHz, CDCl_3) δ 14.0 (CH_3), 22.8 (CH_2), 28.5 (CH_2), 33.2 (CH_2), 66.1 (CH), 74.0 (CH), 78.8 (CH_2), 126.1 (CH, 2 carbons), 127.6 (CH), 127.8 (CH, 2 carbons), 127.9 (CH), 128.4 (CH, 2 carbons), 128.7 (CH, 2 carbons), 140.4 (C), 141.4 (C); ESI m/z $[\text{M} + \text{H}]^+$ 300; HRMS (ESI) calcd for $\text{C}_{19}\text{H}_{26}\text{NO}_2$ $[\text{M} + \text{H}]^+$ 300.1964, found 300.1956. Anal. Calcd for $\text{C}_{19}\text{H}_{25}\text{NO}_2$: C, 76.22; H, 8.42; N, 4.68. Found: C, 76.37; H, 8.24; N, 4.69.

(1R)-1-Phenyl-2-([(1R)-1-phenylpentyl]amino)oxy)ethanol [(R,R)-25]. Colorless oil: $[\alpha]_{\text{D}}^{21} -21.3$ (c 0.7, CHCl_3); IR (neat) 3402, 2950, 2930 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 0.86 (3H, t, $J = 7.2$ Hz), 1.17 (2H, m), 1.66 (2H, m), 1.84 (2H, m), 3.65 (1H, A part of ABX, $J = 12.0, 8.5$ Hz), 3.72 (1H, B part of ABX, $J = 12.0, 2.4$ Hz), 3.99 (1H, dd, $J = 8.5, 5.8$ Hz), 4.90 (1H, dd, $J = 8.5, 2.4$ Hz), 7.26–7.46 (10H, m); ^{13}C NMR (125 MHz, CDCl_3) δ 14.0 (CH_3), 22.8 (CH_2), 28.4 (CH_2), 33.2 (CH_2), 66.0 (CH), 74.2 (CH), 78.9 (CH_2), 126.2 (CH, 2 carbons), 127.6 (CH), 127.7 (CH, 2 carbons), 127.8 (CH), 128.4 (CH, 2 carbons), 128.6 (CH, 2 carbons), 140.6 (C), 141.4 (C); ESI m/z $[\text{M} + \text{H}]^+$ 300; HRMS (ESI) calcd for $\text{C}_{19}\text{H}_{26}\text{NO}_2$ $[\text{M} + \text{H}]^+$ 300.1964, found 300.1972.

(1R)-2-([(1S)-2,2-Dimethyl-1-phenylpropyl]amino)oxy)ethanol [(S,R)-26]. Colorless oil: $[\alpha]_{\text{D}}^{20} -27.3$ (c 0.3, CHCl_3); IR (neat) 3407, 2952, 2925 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 0.95 (9H, s), 3.56 (1H, A part of ABX, $J = 12.1, 8.9$ Hz), 3.66 (1H, B part of ABX, $J = 12.1, 2.4$ Hz), 3.84 (1H, s), 4.89 (1H, dd, $J = 8.9, 2.4$ Hz), 7.24–7.37 (10H, m); ^{13}C NMR (100.6 MHz, CDCl_3) δ 27.5 (CH_3 , 3 carbons), 34.0 (C), 73.6 (CH), 74.7 (CH), 78.6 (CH_2), 126.2 (CH, 2 carbons), 127.5 (CH), 127.6 (CH), 127.9 (CH, 2 carbons), 128.4 (CH, 2 carbons), 128.8 (CH), 140.1 (C), 140.4 (C); ESI m/z $[\text{M} + \text{H}]^+$ 300; HRMS (ESI) calcd for $\text{C}_{19}\text{H}_{26}\text{NO}_2$ $[\text{M} + \text{H}]^+$

300.1964, found 300.1972. Anal. Calcd for $C_{19}H_{25}NO_2$: C, 76.22; H, 8.42; N, 4.68. Found: C, 76.36; H, 8.42; N, 4.48.

(1R)-2-([(1R)-2,2-Dimethyl-1-phenylpropyl]amino)oxy)ethanol [(R,R)-26]. Colorless oil: $[\alpha]_D^{20} +10.6$ (*c* 0.3, $CHCl_3$); IR (neat) 3397, 2953 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$) δ 0.95 (9H, s), 3.61 (1H, A part of ABX, *J* = 11.9, 8.7 Hz), 3.72 (1H, B part of ABX, *J* = 11.9, 2.5 Hz), 3.83 (1H, s), 4.89 (1H, dd, *J* = 8.7, 2.5 Hz), 7.24–7.35 (10H, m); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 27.5 (CH_3 , 3 carbons), 34.0 (C), 74.0 (CH), 74.4 (CH), 78.6 (CH_2), 126.1 (CH, 2 carbons), 127.4 (CH), 127.7 (CH), 127.9 (CH, 2 carbons), 128.4 (CH, 2 carbons), 128.7 (CH, 2 carbons), 139.9 (C), 140.5 (C); ESI *m/z* $[M + H]^+$ 300; HRMS (ESI) calcd for $C_{19}H_{26}NO_2$ $[M + H]^+$ 300.1964, found 300.1972.

(1R)-2-([(1S)-1-[2-(Methoxymethoxy)phenyl]-2,2-dimethylpropyl]amino)oxy]-1-phenylethanol [(S,R)-27]. Colorless oil: $[\alpha]_D^{20} -29.7$ (*c* 0.7, $CHCl_3$); IR (neat) 3414, 2956, 2930 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$) δ 1.31 (9H, s), 3.52 (3H, s), 3.76 (1H, A part of ABX, *J* = 12.3, 8.1 Hz), 3.83 (1H, B part of ABX, *J* = 12.3, 2.6 Hz), 4.13 (1H, s), 5.05 (1H, dd, *J* = 8.1, 2.6 Hz), 5.25 (2H, s), 7.03 (1H, m), 7.14–7.39 (8H, m); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 31.4 (3 carbons, CH_3), 34.9 (C), 51.5 (CH), 56.4 (CH_3), 75.0 (CH), 78.6 (CH_2), 94.9 (CH_2), 111.8 (CH), 119.0 (CH), 122.1 (CH), 126.2 (2 carbons, CH), 127.6 (CH), 127.5 (C), 128.4 (2 carbons, CH), 130.8 (CH), 140.8 (C), 153.1 (C); HRMS (ESI) calcd for $C_{21}H_{30}NO_4$ $[M + H]^+$ 360.2175, found 360.2158; Anal. Calcd for $C_{21}H_{29}NO_4$: C, 70.17; H, 8.13; N, 3.90. Found: C, 70.26; H, 8.12; N, 3.50.

(1R)-2-([(1R)-1-[2-(Methoxymethoxy)phenyl]-2,2-dimethylpropyl]amino)oxy]-1-phenylethanol [(R,R)-27]. Colorless oil: IR (neat) 3410, 2953 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$) δ 1.25 (9H, s), 3.52 (3H, s), 3.76 (1H, A part of ABX, *J* = 12.3, 8.1 Hz), 3.82 (1H, B part of ABX, *J* = 12.3, 2.7 Hz), 5.06 (1H, dd, *J* = 8.1, 2.7 Hz), 5.25 (2H, s), 7.01 (1H, m), 7.14–7.39 (8H, m); HRMS (ESI) calcd for $C_{21}H_{30}NO_4$ $[M + H]^+$ 360.2175, found 360.2191.

(1R)-1-Phenyl-2-([(1S)-1-phenyl-2-propenyl]amino)oxy)ethanol [(S,R)-28]. Colorless oil: $[\alpha]_D^{21} -17.0$ (*c* 0.6, $CHCl_3$); IR (neat) 3407 cm^{-1} ; 1H NMR (500 MHz, $CDCl_3$) δ 3.55 (1H, A part of ABX, *J* = 11.8, 8.9 Hz), 3.65 (1H, B part of ABX, *J* = 11.8, 2.5 Hz), 4.54 (1H, d, *J* = 7.2 Hz), 4.82 (1H, dd, *J* = 8.9, 2.5 Hz), 5.18 (1H, dd, *J* = 17.5, 10.3 Hz), 5.67 (1H, br s), 5.98 (1H, ddd, *J* = 17.5, 10.3, 7.2 Hz), 7.15–7.32 (10H, m); ^{13}C NMR (125 MHz, $CDCl_3$) δ 68.5 (CH), 73.7

(CH), 79.2 (CH₂), 118.0 (CH₂), 126.2 (CH, 2 carbons), 127.7 (CH), 128.0 (CH, 2 carbons), 128.2 (CH), 128.4 (CH, 2 carbons), 128.8 (CH, 2 carbons), 139.6 (C), 140.4 (C); ESI m/z [M + H]⁺ 270; HRMS (ESI) calcd for C₁₇H₂₀NO₂ [M + H]⁺ 270.1494, found 270.1516; Anal. Calcd for C₁₇H₁₉NO₂: C, 75.81; H, 7.11; N, 5.20. Found: C, 76.00; H, 7.17; N, 4.88.

(1R)-1-Phenyl-2-([(1R)-1-phenyl-2-propenyl]amino)oxy)ethanol [(R,R)-28].

Colorless oil: $[\alpha]_D^{21}$ -31.6 (*c* 0.3, CHCl₃); IR (neat) 3397 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 3.68 (1H, A part of ABX, *J* = 12.0, 8.6 Hz), 3.78 (1H, B part of ABX, *J* = 12.0, 2.6 Hz), 4.63 (1H, d, *J* = 8.7 Hz), 4.96 (1H, dd, *J* = 8.6, 2.6 Hz), 5.31 (2H, dd, *J* = 17.3, 10.2 Hz), 6.09 (1H, ddd, *J* = 17.3, 10.2, 8.7 Hz), 7.25–7.38 (10H, m); ¹³C NMR (100.6 MHz, CDCl₃) δ 68.6 (CH), 73.5 (CH), 79.2 (CH₂), 118.1 (CH₂), 126.2 (CH, 2 carbons), 127.7 (CH), 127.9 (CH, 2 carbons), 128.1 (CH), 128.4 (CH, 2 carbons), 128.7 (CH, 2 carbons), 137.6 (C), 139.6 (C); ESI m/z [M + H]⁺ 270; HRMS (ESI) calcd for C₁₇H₂₀NO₂ [M + H]⁺ 270.1494, found 270.1498.

(1R)-2-([(S)-[(2-(Methoxymethoxy)phenyl)(phenyl)methyl]amino]oxy)-1-

phenylethanol [(S,R)-29]. Colorless oil: $[\alpha]_D^{20}$ -14.8 (*c* 0.2, CHCl₃); IR (neat) 3420 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 3.35 (3H, s), 3.69 (1H, A part of ABX, *J* = 12.0, 8.7 Hz), 3.78 (1H, B part of ABX, *J* = 12.0, 2.6 Hz), 4.96 (1H, dd, *J* = 8.7, 2.6 Hz), 5.16 (1H, 1/2 ABq, *J* = 6.7 Hz), 5.20 (1H, 1/2, ABq, *J* = 6.7 Hz), 5.68 (1H, s), 7.01 (1H, t, *J* = 7.5 Hz), 7.09 (1H, d, *J* = 8.3 Hz), 7.24–7.44 (12H, m); ¹³C NMR (100.6 MHz, CDCl₃) δ 56.1 (CH₃), 63.4 (CH), 73.5 (CH), 78.9 (CH₂), 94.3 (CH₂), 126.1 (CH, 2 carbons), 127.5 (CH), 127.6 (CH, 2 carbons), 127.9 (CH, 2 carbons), 128.0 (CH, 2 carbons), 128.3 (CH, 3 carbons), 128.4 (CH, 2 carbons), 128.8 (C), 140.3 (C), 140.4 (C), 154.6 (C); ESI m/z [M + H]⁺ 380; HRMS (ESI) calcd for C₂₃H₂₆NO₄ [M + H]⁺ 380.1862, found 380.1834. Anal. Calcd for C₂₃H₂₅NO₄: C, 72.80; H, 6.64; N, 3.69. Found: C, 72.91; H, 6.64; N, 3.81.

(1R)-2-([(R)-[(2-(Methoxymethoxy)phenyl)(phenyl)methyl]amino]oxy)-1-

phenylethanol [(R,R)-29]. Colorless oil: $[\alpha]_D^{20}$ -35.7 (*c* 0.1, CHCl₃); IR (neat) 3406 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 3.41 (3H, s), 3.65 (1H, A part of ABx, *J* = 11.9, 8.7 Hz), 3.77 (1H, B part of ABX, *J* = 11.9, 2.5 Hz), 4.88 (1H, dd, *J* = 8.7, 2.5 Hz), 5.10 (1H, 1/2 ABq, *J* = 6.7 Hz), 5.14 (1H, 1/2 ABq, *J* = 6.7 Hz), 5.63 (1H, s), 6.95 (1H, t, *J* = 7.5 Hz), 7.03 (1H, d, *J* = 8.4 Hz), 7.15–7.37 (12H, m); ¹³C NMR (100.6 MHz, CDCl₃) δ 56.1 (CH₃), 63.4 (CH), 73.6 (CH), 78.9 (CH₂), 94.3 (CH₂), 126.1 (CH, 2 carbons), 127.47 (CH), 127.6 (CH, 2 carbons), 127.9 (CH, 2

carbons), 128.0 (CH, 2 carbons), 128.3 (CH, 3 carbons), 128.4 (CH, 2 carbons), 128.8 (C), 140.2 (C), 140.5 (C), 154.7 (C); ESI m/z $[M + H]^+$ 380; HRMS (ESI) calcd for $C_{23}H_{26}NO_4$ $[M + H]^+$ 380.1862, found 380.1872.

3-Methoxybenzaldehyde *O*-[(1*S*)-2-Methoxy-1-phenylethyl]oxime (31). To a stirred ice-cold solution of **9d** (300 mg, 1.11 mmol) in THF (12 mL) was added a 60% suspension of NaH (66.0 mg, 1.66 mmol) in mineral oil, and the mixture was stirred for 30 min at 0 °C. To this mixture was added iodomethane (0.104 mL, 1.66 mmol), the mixture was stirred for additional 1 h at rt, and then the reaction was quenched with saturated aqueous NH_4Cl (10 mL). The mixture was extracted with ether (3 × 30 mL) and the combined organic phases were washed with brine (30 mL), dried ($MgSO_4$), and concentrated in vacuo. The residue was purified by chromatography (hexane–EtOAc, 6:1) to give **31** (277 mg, 87%) as a colorless oil: $[\alpha]_D^{25} -2.09$ (c 0.74, $CHCl_3$); 1H NMR (400 MHz, $CDCl_3$) δ 3.42 (3H, s), 3.63–3.67 (1H, A part of ABX, $J = 11.0, 4.1$ Hz), 3.79 (3H, s), 3.79–3.83 (1H, B part of ABX, $J = 11.0, 7.9$ Hz), 5.40 (1H, dd, $J = 7.7, 4.2$ Hz), 6.82–6.85 (2H, m), 7.22–7.39 (6H, m), 7.68 (1H, dd, $J = 7.7, 1.7$ Hz), 8.61 (1H, s); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 55.6 (CH_3), 59.4 (CH_3), 75.5 (CH_2), 84.1 (CH), 111.1 (CH), 120.7 (C), 120.8 (CH), 126.6 (CH), 127.1 (CH, 2 carbons), 128.3 (CH, 2 carbons), 131.1 (CH), 139.5 (C), 145.6 (CH), 157.6 (C); EIMS m/z (relative intensity) 286 ($M^+ + 1$, 100), 285 (M^+ , 25), 239 (12), 152 (5), 133 (16), 106 (7). Anal. Calcd for $C_{17}H_{19}NO_3$: C, 71.56; H, 6.71; N, 4.91. Found: C, 71.60; H, 6.72; N, 4.85.

***N*-[(1*R*)-1-(2-Methoxyphenyl)ethyl]-*O*-[(1*S*)-2-methoxy-1-phenylethyl]hydroxylamine [(*R,S*)-32]. and *N*-[(1*S*)-1-(2-Methoxyphenyl)ethyl]-*O*-[(1*S*)-2-methoxy-1-phenylethyl]hydroxylamine [(*S,S*)-32].** To a stirred ice-cold solution of **31** (0.200 g, 0.701 mmol) in toluene (17 mL) was added a 1.14 M solution of methyllithium in Et_2O (0.614 mL, 0.701 mmol) under N_2 atmosphere. After being stirred for 5 min at 0 °C, the mixture was cooled to –20 °C, and then an additional 1.14 M solution of methyllithium in Et_2O (0.921 mL, 1.05 mmol) was added. After stirring for an additional 25 min at –20 °C, the reaction was quenched with water (10 mL) under cooling, the organic layer was separated, and the aqueous layer was extracted with ether (3 × 30 mL). The combined organic phases were washed with brine (30 mL), dried ($MgSO_4$), and concentrated in vacuo. The residue was purified by chromatography (hexane–EtOAc, 4:1) to give as the first fraction (*S,S*)-**32** (15.3 mg, 7%) as a colorless oil:

$[\alpha]_D^{20} +43.5$ (*c* 0.2, CHCl_3); ^1H NMR (400 MHz, CDCl_3) δ 1.26 (3H, d, $J = 6.6$ Hz), 3.21 (3H, s), 3.38 (1H, A part of ABX, $J = 10.9, 4.2$ Hz), 3.54 (1H, B part of ABX, $J = 10.9, 7.3$ Hz), 3.81 (3H, s), 4.15 (1H, q, $J = 6.6$ Hz), 4.72 (1H, dd, $J = 7.3, 4.2$ Hz), 5.57 (1H, br s), 6.81 (1H, m), 6.93 (2H, m), 7.22–7.35 (6H, m); ^{13}C NMR (100.6 MHz, CDCl_3) δ 20.0 (CH_3), 55.3 (CH), 59.0 (CH_3), 60.6 (CH_3), 75.4 (CH_2), 84.3 (CH), 112.8 (CH), 112.9 (CH), 119.7 (CH), 127.1 (CH, 2 carbons), 127.9 (CH), 128.4 (CH, 2 carbons), 129.3 (CH), 139.9 (C), 145.0 (C), 159.7 (C); HRMS (ESI) calcd for $\text{C}_{18}\text{H}_{24}\text{NO}_3$ $[\text{M} + \text{H}]^+$ 302.1729, found 302.1754.

The second fraction afforded (*R,S*)-**32** (46.5 mg, 22%) as a colorless oil: $[\alpha]_D^{18} +53.6$ (*c* 1.6, CHCl_3); ^1H NMR (400 MHz, CDCl_3) δ 1.40 (3H, d, $J = 6.7$ Hz), 3.34 (3H, s), 3.45–3.48 (1H, A part of ABX, $J = 10.8, 4.1$ Hz), 3.57–3.61 (1H, B part of ABX, $J = 10.8, 7.6$ Hz), 3.71 (3H, s), 4.53 (1H, q, $J = 6.5$ Hz), 4.84 (1H, dd, $J = 7.6, 4.0$ Hz), 5.90 (1H, br s), 6.76 (1H, d, $J = 8.2$ Hz), 6.81 (1H, m), 7.12–7.31 (7H, m); ^{13}C NMR (100.6 MHz, CDCl_3) δ 18.2 (CH_3), 54.8 (CH), 55.2 (CH_3), 75.8 (CH_2), 84.4 (CH_3), 110.4 (CH), 120.5 (CH), 126.9 (CH, 2 carbons), 127.6 (CH), 127.9 (CH), 128.1 (CH), 128.2 (CH, 2 carbons), 130.1 (C), 140.0 (C), 157.0 (C); EIMS m/z (relative intensity) 302 ($\text{M}^+ + 1$, 100), 167 (25), 135 (87), 103 (24), 91 (17), 77 (12). Anal. Calcd for $\text{C}_{18}\text{H}_{23}\text{NO}_3$: C, 71.73; H, 7.69; N, 4.65. Found: C, 71.59; H, 7.71; N, 4.68.

Preparation of Phenylethanamines (*R*)-34–44, (*S*)-34, (*S*)-35, (*S*)-37, (*S*)-38, and (*S*)-40–44 by Reductive Cleavage of the N–O-bond. Method A. To a stirred solution of (*R,S*)-**14–18** or (*R,S*)-**20** (1.31 mmol) in a mixture of AcOH–THF–water (3:1:1) (19 mL) was added zinc powder (1.20 g, 18.4 mmol) in several portions at rt. After being stirred for 4 h at 60 °C, the mixture was basified with concentrated NH_4OH (20 mL) and stirred for 1 h at rt. The mixture was filtered to remove inorganic material, the filtrate was extracted with CHCl_3 (3 $\times$ 70 mL), and the combined extracts were dried (MgSO_4). Evaporation of the solvent in vacuo and chromatography (CHCl_3 –MeOH–concentrated NH_4OH , 200:9:1) gave as the first fraction (*S*)-phenylethanediol [(*S*)-**6**]. The second fraction gave (*R*)-**34–38** or (*R*)-**40** (for yields, see Scheme 4).

Method B. To a solution of (*R,S*)-**14**, (*R,S*)-**15**, (*R,S*)-**18–20**, or (*S,R*)-**21–29** (1.31 mmol) in a mixture of MeCN–water (15:1) (13 mL) was added $\text{Mo}(\text{CO})_6$ (692 mg, 2.62 mmol) at rt, and the mixture was stirred at 85 °C. After 1 h, concentrated NH_4OH (10 mL) was added and the resultant mixture was stirred for 1 h at rt and extracted with CHCl_3 (3 $\times$ 50 mL). The combined

extracts were dried (MgSO_4) and concentrated in vacuo to give the crude product, which was subjected to chromatography (CHCl_3 –MeOH–concentrated NH_4OH , 200:9:1) to give as the first fraction (*S*)-phenylethanediol [(*S*)-**6**] for (*R,S*)-**15** or (*R,S*)-**18–20** or (*R*)-phenylethanediol [(*R*)-**6**] for (*S,R*)-**21–29**. The second fraction afforded (*R*)-**34**, (*R*)-**35**, (*R*)-**38–40**, (*S*)-**34**, (*S*)-**35**, (*S*)-**37**, (*S*)-**38**, or (*S*)-**40–44** (for yields, see Scheme 4).

(1*R*)-1-(3-Methoxyphenyl)ethanamine [(*R*)-34**].** Pale yellow oil: $[\alpha]_{\text{D}}^{21} +34.5$ (*c* 1.0, CHCl_3) [lit.² $[\alpha]_{\text{D}}^{20} +32.0$ (*c* 0.3, CHCl_3)]. ^1H and ^{13}C NMR spectra were identical with those of the reported material.²

(1*R*)-1-(2-Methoxyphenyl)ethanamine [(*R*)-35**].** Pale yellow oil: $[\alpha]_{\text{D}}^{21} +18.9$ (*c* 1.0, MeOH) [lit.^{3a} $[\alpha]_{\text{D}}^{20} +18$ (*c* 1.0, MeOH)]; A ^1H NMR spectrum was identical with those of the reported^{3b} material.

(1*R*)-1-(3-Methoxyphenyl)ethanamine [(*R*)-36**].** Pale yellow oil: $[\alpha]_{\text{D}}^{25} +27.7$ (*c* 0.4, MeOH) [lit.⁴ $[\alpha]_{\text{D}}^{20} +17.6$ (*c* 2, MeOH)]; IR (neat) 3362, 3291, 2926 cm^{-1} ; ^1H and ^{13}C NMR spectra were identical with those of the reported⁵ material.

(1*R*)-1-[3-(Methylsulfanyl)phenyl]ethanamine [(*R*)-37**].** Pale yellow oil: $[\alpha]_{\text{D}}^{25} +34.0$ (*c* 1.2, CHCl_3); IR (neat) 3286 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 1.38 (3H, d, *J* = 6.4 Hz), 2.49 (3H, s), 4.09 (1H, q, *J* = 6.6 Hz), 7.10–7.14 (2H, m), 7.23–7.27 (2H, m); ^{13}C NMR (100.6 MHz, CDCl_3) δ 15.9 (CH_3), 25.7 (CH_3), 122.7 (CH), 124.2 (CH), 125.0 (CH), 129.1 (CH), 138.6 (C), 148.6 (C); EIMS *m/z* (relative intensity) 169 ($\text{M}^+ + 2$, 11), 168 ($\text{M}^+ + 1$, 11), 167 (M^+ , 66), 166 (100), 153 (10), 151 (6). Anal.Calcd for $\text{C}_9\text{H}_{13}\text{NS}$: C, 64.62; H, 7.83; N, 8.37. Found: C, 64.59; H, 7.92; N, 8.16.

(1*R*)-1-(4-Methylphenyl)ethanamine [(*R*)-38**].** Pale yellow oil: $[\alpha]_{\text{D}}^{21} +31.2$ (*c* 1.2, CHCl_3) [lit.⁵ $[\alpha]_{\text{D}} +28.1$ (*c* 1.0, CHCl_3)]. ^1H and ^{13}C NMR spectra were identical with those of the reported material.⁵

(1*R*)-1-[2-(Methoxymethoxy)phenyl]-2-propen-1-amine [(*R*)-39**].** Pale yellow oil: $[\alpha]_{\text{D}}^{21} +26.1$ (*c* 0.7, CHCl_3); IR (neat) 3376 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 3.48 (3H, s), 3.91 (2H, br s), 4.86 (1H, d, *J* = 5.6 Hz), 5.19 (2H, dd, *J* = 16.0, 10.4 Hz), 5.21 (1H, 1/2 ABq, *J* = 6.7 Hz), 5.22 (1H, 1/2 ABq, *J* = 6.7 Hz), 6.10 (1H, ddd, *J* = 16.0, 10.4, 5.6 Hz), 6.98–7.10 (2H, m), 7.18–7.29 (2H, m); ^{13}C NMR (100.6 MHz, CDCl_3) δ 52.9 (CH_3), 56.3 (CH), 94.5 (CH_2), 114.1 (CH_2), 114.3 (CH), 122.1 (CH), 127.5 (CH), 128.4 (CH), 132.5 (C), 140.4 (CH), 154.5 (C); ESI

m/z $[M + H]^+$ 194; HRMS (ESI) calcd for $C_{11}H_{16}NO_2$ $[M + H]^+$ 194.1181, found 194.1181. Anal. Calcd for $C_{11}H_{15}NO_2$: C, 68.37; H, 7.82; N, 7.25. Found: C, 68.60; H, 7.62; N, 7.12.

(1R)-1-[2-(Methoxymethoxy)phenyl](phenyl)methanamine [(R)-40]. Pale yellow oil: $[\alpha]_D^{21} +37.7$ (c 0.5, $CHCl_3$); IR (neat) 3375 cm^{-1} ; 1H NMR (400 MHz, $CDCl_3$) δ 1.88 (2H, br s), 3.30 (3H, s), 5.10 (1H, 1/2 ABq, $J = 6.7$ Hz), 5.14 (1H, 1/2 ABq, $J = 6.7$ Hz), 5.50 (1H, s), 6.98–7.06 (2H, m), 7.06–7.39 (7H, m); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 54.2 (CH_3), 56.0 (CH), 94.2 (CH_2), 114.0 (CH), 121.8 (CH), 126.6 (CH), 127.0 (CH, 2 carbons), 127.6 (CH), 128.0 (CH), 128.2 (CH, 2 carbons), 134.7 (C), 145.2 (C), 154.3 (C); ESI m/z $[M + H]^+$ 244; HRMS (ESI) calcd for $C_{15}H_{18}NO$ $[M + H]^+$ 244.1338, found 244.1344. Anal. Calcd for $C_{15}H_{17}NO$: C, 74.05; H, 7.04; N, 5.76. Found: C, 73.90; H, 7.09; N, 5.71.

(1S)-1-Phenylethanamine [(S)-34]. Pale yellow oil: $[\alpha]_D^{20} -35.1$ (c 1.0, $CHCl_3$); for 1H and ^{13}C NMR, see (R)-34.

(1S)-1-(2-Methoxyphenyl)ethanamine [(S)-35]. Pale yellow oil: $[\alpha]_D^{20} -18.8$ (c 0.8, $CHCl_3$); for 1H and ^{13}C NMR, see (R)-35.

(1S)-1-(3-Methoxyphenyl)ethanamine [(S)-36]. Pale yellow oil: $[\alpha]_D^{20} -26.0$ (c 0.8, $CHCl_3$); for 1H and ^{13}C NMR, see (R)-36.

(1S)-1-(4-Methylphenyl)ethanamine [(S)-38]. Pale yellow oil: $[\alpha]_D^{20} -30.9$ (c 1.0, $CHCl_3$); for 1H and ^{13}C NMR, see (R)-38.

(1S)-1-[2-(Methoxymethoxy)phenyl](phenyl)methanamine [(S)-40]. Pale yellow oil: $[\alpha]_D^{21} -35.4$ (c 0.5, $CHCl_3$); for 1H and ^{13}C NMR, see (R)-40.

(1S)-1-Phenyl-1-pentanamine [(S)-41]. Pale yellow oil: $[\alpha]_D^{20} -12.1$ (c 0.9, $CHCl_3$) [lit.⁶ $[\alpha]_D -12.7$ (c 2.50, $CHCl_3$)]; 1H , ^{13}C and IR spectra were identical with those of the reported⁶ material.

(1S)-2,2-Dimethyl-1-phenyl-1-propanamine [(S)-42]. Pale yellow oil: $[\alpha]_D^{20} -4.7$ (c 1, $CHCl_3$) [lit.⁷ $[\alpha]_D -5.5$ (c 1.00, $CHCl_3$)]; 1H , ^{13}C and IR spectra were identical with those of the reported⁷ material.

(1S)-1-[2-(Methoxymethoxy)phenyl]-2,2-dimethyl-1-propanamine [(S)-43]. Pale yellow oil: $[\alpha]_D^{20} -20.6$ (c 0.4, $CHCl_3$)⁸; 1H NMR (400 MHz, $CDCl_3$) δ 1.06 (9H, s), 2.01 (2H, br s), 3.25 (3H, s), 3.90 (1H, s), 6.04 (2H, s), 6.72–6.77 (2H, m), 6.97–7.01 (2H, m); ^{13}C NMR (100.6 MHz, $CDCl_3$) δ 30.5 (3 carbons, CH_3), 35.9 (C), 53.1 (CH_3), 66.3 (CH), 94.7 (CH_2), 122.3

(CH), 127.7 (CH), 128.6 (CH), 132.7 (C), 140.4 (CH), 154.7 (C); HRMS (ESI) calcd for $C_{13}H_{22}NO_2$ $[M + H]^+$ 224.1651, found 224.1636. Anal. Calcd for $C_{13}H_{21}NO_2$: C, 69.92; H, 9.487; N, 6.27. Found: C, 69.78; H, 9.30; N, 5.96.

(1S)-1-Phenyl-2-propen-1-amine [(S)-44]. Pale yellow oil: $[\alpha]_D^{20} -9.7$ (c 0.9, $CHCl_3$); for 1H and ^{13}C NMR, see (*R*)-**44** in the journal page.

Figure 1. ORTEP drawing of the hydroxy oxime ether **9f**.

Crystal Data of **9f**

The study of the titled structure was undertaken to establish its three dimensional structure. Geometries are tabulated below. All diagrams and calculations were performed using maXus (Bruker Nonius, Delft & MacScience, Japan).

Crystal data

$M_r = 255.31$

monoclinic

 $P2_1$

$a = 5.5250 (2) \text{ \AA}$

$b = 30.9290 (13) \text{ \AA}$

$c = 8.1140 (4) \text{ \AA}$

$\alpha = 90.00^\circ$

$\beta = 95.724 (2)^\circ$

$\gamma = 90.00^\circ$

$V = 1379.63 (10) \text{ \AA}^3$

$Z = 4$

$D_x = 1.229 \text{ Mg m}^{-3}$

Density measured by: not measured

fine-focus sealed tube

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 2845 reflections

$\theta = 2.61\text{---}27.21^\circ$

$\mu = 0.081 \text{ mm}^{-1}$

$T = 100 (2) \text{ K}$

Needle

$0.50 \times 0.25 \times 0.23 \text{ mm}$

colourless

Data collection ω

Absorption correction: none

$T_{\min} = 0.9606, T_{\max} = 0.9816$

2845 measured reflections

2845 independent reflections

2200 observed reflections

Criterion: $>2\sigma(I)$

$R_{\text{int}} = 0.047$

$\theta_{\max} = 27.21^\circ$

$h = 0 \rightarrow 6$

$k = 0 \rightarrow 39$

$l = -10 \rightarrow 10$

RefinementRefinement on F^2

fullmatrix least squares refinement

$R(\text{all}) = 0.0763$

$R(\text{gt}) = 0.0607$

$wR(\text{ref}) = 0.1708$

$wR(\text{gt}) = 0.1550$

$S(\text{ref}) = 0.997$

2845 reflections

348 parameters

1 restraints

mixed

Calculated weights calc

$\Delta/\sigma_{\max} = 0.001$

$\Delta\rho_{\max} = 0.374 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.272 \text{ e \AA}^{-3}$

Extinction correction: SHELXL

$F_c^* = kFc [1 + 0.001 \times Fc^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

$\text{Extinction coefficient} = 0.059 (9)$

Atomic scattering factors from International Tables Vol C Tables 4.2.6.8 and 6.1.1.4 Flack parameter = 1 (2)
Flack H D (1983), Acta Cryst. A39, 876-881

Data collection: DIP2000 software Xpress (MAC Science, 1989)

Cell refinement: DIP2000 software 'DENZO' (Otwinowski & Minor, 1996)

Data reduction: DIP2000 software 'DENZO' (Otwinowski & Minor, 1996)

Program(s) used to solve structure: SIR97 (Altomare, et al, 1999)

Program(s) used to refine structure: SHELXL-97 (Sheldrick, 1997)

Molecular graphics: maXus (Mackay et al., 1999)

Software used to prepare material for publication: maXus (Mackay et al., 1999)

Current Data Parameters
 NAME: alpha-o-OMOM-H
 EXPNO: 1
 PROCNO: 1

F2 - Acquisition Parameters
 Date_: 20040111
 Time: 4:50
 INSTRUM: spect
 PROBRD: 5 mm Maltin
 PULPROG: zgpg30
 TD: 32768
 SOLVENT: CDCl3
 NS: 64
 DS: 4
 SWH: 7507.507 Hz
 FIDRES: 0.229111 Hz
 AQ: 2.1823988 sec
 RG: 322.5
 DW: 66.600 usec
 DE: 6.00 usec
 TE: 300.0 K
 D1: 1.0000000 sec

===== CHANNEL f1 =====
 NUC1: 1H
 P0: 5.80 usec
 PL1: -4.00 dB
 SFO1: 500.035002 MHz

F2 - Processing parameters
 SI: 16384
 SF: 500.0300107 MHz
 WDW: EM
 SSB: 0
 LB: 0.30 Hz
 GB: 0
 PC: 1.00

1D NMR plot parameters
 CX: 32.00 cm
 FIP: 10.000 ppm
 FL: 5000.30 Hz
 F2P: 0.000 ppm
 F2: 0.00 Hz
 PPMCM: 0.31250 ppm/cm
 HZCM: 156.25937 Hz/cm

Current Data Parameters
NAME arsyne
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040224
Time_ 18.17
INSTRUM dpx400
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 64
DS 0
SWH 6793.478 Hz
FIDRES 0.207320 Hz
AQ 2.4117749 sec
RG 512
DM 73.600 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.40 usec
PL1 -6.00 dB
SF01 400.1326354 MHz

F2 - Processing parameters
SI 16384
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.31250 ppm/cm
HZCM 125.04063 Hz/cm

Current Data Parameters
NAME atobe
EXNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20011121
Time 18:50
INSTRUM dpx400
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 64
DS 0
SWH 6793.478 Hz
FIDRES 0.207320 Hz
AQ 2.4117749 sec
RG 256
DM 73.600 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
P1 7.40 usec
SF01 400.1326354 MHz
NUC1 1H
PL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 400.1300091 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PRMCH 0.31250 ppm/cm
HZCM 125.04063 Hz/cm

Current Data Parameters
 NAME atobe
 EXPNO 12
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20011207
 Time 21.17
 INSTRUM dpx400
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 64
 DS 0
 SWH 6793.478 Hz
 FIDRES 0.207320 Hz
 AQ 2.4117749 sec
 RG 256
 DW 73.600 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 7.40 usec
 SFO1 400.1326354 MHz
 NUC1 1H
 P11 -6.00 dB

F2 - Processing parameters
 SI 16384
 SF 400.1300091 MHz
 WDW EN
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 32.00 cm
 F1P 9.000 ppm
 F1 3601.17 Hz
 F2P -1.000 ppm
 F2 -400.13 Hz
 PPMCM 0.31250 ppm/cm
 HZCM 125.04063 Hz/cm

(2S,3S)-63

Current Data Parameters
 NAME atobe
 EXNO 40
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20011222
 Time 3.47
 INSTRUM dp4100
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 64
 DS 0
 SWH 6793.478 Hz
 FIDRES 0.207320 Hz
 AQ 2.4117749 sec
 RG 203.2
 DW 73.600 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 7.40 usec
 SFO1 400.1326354 MHz
 NUC1 1H
 PL1 -6.00 dB

F2 - Processing parameters
 SI 16384
 SF 400.1300091 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 32.00 cm
 FIP 10.000 ppm
 F1 4001.30 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.31250 ppm/cm
 HZCM 125.04063 Hz/cm

(2S,3R)-63

Current Data Parameters
 NAME atobe
 EXNO 45
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20011222
 Time_ 5.05
 INSTRUM dpx400
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 64
 DS 0
 SWH 6793.478 Hz
 FIDRES 0.207320 Hz
 AQ 2.4117749 sec
 RG 256
 DW 73.600 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 7.40 usec
 SFO1 400.1326354 MHz
 NUC1 1H
 PL1 -6.00 dB

F2 - Processing parameters
 SI 16384
 SF 400.1300091 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 32.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.31250 ppm/cm
 HZCM 125.04063 Hz/cm

Current Data Parameters
NAME atobe
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20011018
Time_ 17.48
INSTRUM dpx400
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 64
DS 0
SWH 6793.478 Hz
FIDRES 0.207320 Hz
AQ 2.4117149 sec
RG 256
DM 73.600 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
F1 7.40 usec
SFO1 400.1326354 MHz
NUC1 1H
FL1 -6.00 dB

F2 - Processing parameters
SI 16384
SF 400.1300091 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
F1P 9.700 ppm
F1 3881.26 Hz
F2P -0.300 ppm
F2 -120.04 Hz
PPMCM 0.31250 ppm/cm
HZCM 125.04063 Hz/cm

Current Data Parameters
NAME atobe
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20011225
Time 11.09
INSTRUM dp-500
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 64
DS 4
SWH 7507.507 Hz
FIDRES 0.223111 Hz
AQ 2.182388 sec
RG 161.3
DM 66.600 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
P0 5.80 usec
SF01 500.0335002 MHz
NUC1 1H
PL1 -4.00 dB

F2 - Processing parameters
SI 16384
SF 500.0300107 MHz
WDW EN
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
F1P 10.000 ppm
F1 9000.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPM0H 0.31250 ppm/cm
HZCM 156.28937 Hz/cm

Current Data Parameters
NAME atope
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040131
Time 7.00
INSTRUM dpx400
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 64
DS 0
SWH 6793.478 Hz
FIDRES 0.207320 Hz
AQ 2.4117749 sec
RG 724.1
DM 73.600 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 7.40 usec
PL1 -6.00 dB
SFO1 400.1326354 MHz

F2 - Processing parameters
SI 16384
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.31250 ppm/cm
HZCM 125.04063 Hz/cm

Current Data Parameters
 NAME atobe
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040203
 Time 11.08
 INSTRUM dp400
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 64
 DS 0
 SWH 6793.478 Hz
 FIDRES 0.207320 Hz
 AQ 2.4117749 sec
 RG 362
 DW 73.600 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.40 usec
 PL1 -6.00 dB
 SFO1 400.1326354 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300091 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 32.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.31250 ppm/cm
 HZCM 125.04063 Hz/cm

Current Data Parameters
 NAME atobe
 EXNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20020306
 Time 13.01
 INSTRUM QNP400
 PROBD 5 mm QNP 1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 64
 DS 0
 SWH 6793.478 Hz
 FIDRES 0.207320 Hz
 AQ 2.4117749 sec
 RG 256
 DW 73.600 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 7.40 usec
 SFO1 400.1326354 MHz
 NUC1 1H
 PL1 -6.00 dB

F2 - Processing parameters
 SI 16384
 SF 400.1300087 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CY 32.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCH 0.31250 ppm/cm
 HZCM 125.04063 Hz/cm

Current Data Parameters
 NAME a
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20010113
 Time 5.28
 INSTRUM QNP400
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 64
 DS 0
 SWH 6793.478 Hz
 FIDRES 0.207320 Hz
 AQ 2.4117749 sec
 RG 256
 DW 73.600 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec
 P1 7.40 usec
 SFO1 400.1326354 MHz
 NUC1 1H
 PL1 -6.00 dB

F2 - Processing parameters
 SI 16384
 SF 400.1300087 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 32.00 cm
 FIP 10.000 ppm
 F1 4001.30 Hz
 F2 0.000 ppm
 F2 0.00 Hz
 PPMCN 0.31250 ppm/cm
 HZCN 125.04063 Hz/cm

Current Data Parameters
 NAME Alkylasbi
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20010105
 Time 8.22
 INSTRUM 400400
 PRCHRD 5 mm QNP -H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl₃
 NS 64
 DS 0
 SWH 6793.478 Hz
 FIDRES 0.207320 Hz
 AQ 2.4117749 s
 RG 256
 DE 73.600 us
 TE 300.0 K
 DI 1.00000000 s
 PL 7.00 C
 SFO1 400.1126354 MHz
 NUC1 ¹H
 PL1 -6.00 dB

F2 - Processing parameters
 SI 16384
 SF 400.1300051 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 32.00 cm
 F1 10.000 pp
 F1 4301.30 Hz
 F2 0.000 pp
 F2 0.00 Hz
 PPMCM 0.3125 pp /cm
 HZCM 125.0403 Hz cm

11

(S,S)-14

Current Data Parameters
 NAME atobe
 EXNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20030508
 Time_ 1.56
 INSTRUM dxt400
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 64
 DS 0
 SWH 6793.478 Hz
 FIDRES 0.200320 Hz
 AQ 2.4117749 sec
 RG 255
 DW 73.500 usec
 DE 5.00 usec
 TE 300.0 K
 TS 1.0000000 sec
 D1 7.00
 P1 1.0000000 sec
 SFO1 400.1326354 MHz
 NUC1 1H
 P11 -6.00 dB

F2 - Processing parameters
 SI 16384
 SF 400.1300951 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 32.00 cm
 F1P 10.000 ppm
 F1 400.130 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 FPCOM 0.31250 ppm/cm
 HZCM 125.04065 Hz/cm

(S,S)-17

Current Data Parameters
 NAME atcbe
 EXPNO 40
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040218
 Time_ 19:11
 INSTRUM dpx400
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 64
 DS 0
 SWH 6793.478 Hz
 FIDRES 0.207320 Hz
 AQ 2.411749 sec
 RG 256
 DW 73.600 usec
 DE 6.000 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.40 usec
 PL1 -6.00 dB
 SFO1 400.1326354 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 32.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPM0H 0.31250 ppm/cm
 HZCM 125.04063 Hz/cm

Current Data Parameters
 NAME atobe
 EXNO 18
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040214
 Time_ 20.02
 INSTRUM dx400
 PROBD 5 mm QNP 1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 64
 DS 0
 SWH 6793.478 Hz
 FIDRES 0.207320 Hz
 AQ 2.4117749 sec
 RG 645.1
 DW 73.600 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.40 usec
 PL1 -6.00 dB
 SFO1 400.1326354 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300346 MHz
 MDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 32.00 cm
 F1P 10.000 ppm
 F1 4001.30 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.31250 ppm/cm
 RECM 125.04063 Hz/cm

Current Data Parameters
NAME Be-ta-H-1
EXTNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040119
Time 11.10
INSTRUM drx500
PROBHD 5 mm Multinu
PULPROG zg0
TD 32768
SOLVENT CDCl3
NS 64
DS 4
SWH 7507.507 Hz
FIDRES 0.229111 Hz
AQ 2.1821988 sec
RG 64
DM 66.600 usec
DE 6.00 usec
TE 300.0 K
DL 1.00000000 sec

===== CHANNEL f1 =====
NUC1 H
P0 5.80 usec
PL1 -4.00 dB
SFO1 500.0335002 MHz

F2 - Processing parameters
SI 16384
SF 500.0300427 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
FLP 10.000 ppm
F1 5000.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.31250 ppm/cm
HZCM 156.25938 Hz/cm

(R,R)-21

Current Data Parameters
NAME beta-O-Me
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040124
Time 12.02
INSTRUM dxs500
PROBHD 5 mm FXI 13C X
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 64
DS 2
SWH 8012.820 Hz
FIDRES 0.244512 Hz
AQ 2.0447731 sec
RG 80.6
DM 62.400 usec
DE 7.50 usec
TE 300.0 K
D1 1.0000000 sec
MCREST 0.0000000 sec
MCWRR 0.0150000 sec
===== CHANNEL f1 =====
NUC1 1H
P0 4.70 usec
PL1 0.00 dB
SFO1 500.1335009 MHz
F2 - Processing parameters
SI 16384
SF 500.1300138 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 32.00 cm
CY 21.00 cm
FLP 10.000 ppm
F1 5001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
FREQM 0.31250 ppm/cm
HZCM 156.29062 Hz/cm

F2 - Acquisition Parameters
 Date_ 20040119
 Time 11:52
 INSTRUM drx500
 PROBHD 5 mm Maltinu
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 64
 DS 4
 SWH 7507.507 Hz
 FIDRES 0.229111 Hz
 AQ 2.1823988 sec
 RG 362
 DW 66.600 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P0 5.80 usec
 PL1 -4.00 dB
 SFO1 500.0335002 MHz

F2 - Processing parameters
 SI 16384
 SF 500.0330011 MHz
 MDW EN
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 32.00 cm
 F1P 10.000 ppm
 F1 5000.30 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.31250 ppm/cm
 HZCM 156.25937 Hz/cm

Current Data Parameters
NAME Beta-mCMe-f
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040119
Time 11.31
INSTRUM dirx500
PROBHD 5 mm Maltinu
PULPROG zg0
TD 32768
SOLVENT CDCl3
NS 64
DS 4
SWH 7507.507 Hz
FIDRES 0.229111 Hz
AQ 2.1823988 sec
RG 181
DM 66.600 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P0 5.80 usec
PL1 -4.00 dB
SF01 500.0335002 MHz

F2 - Processing parameters
SI 16384
SF 500.0300107 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
FIP 10.000 ppm
F1 5000.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.31250 ppm/cm
HZCM 156.25937 Hz/cm

Current Data Parameters
 NAME Deca-H-butyl-
 EXPRNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040211
 Time_ 11.35
 INSTRUM dxt500
 PROBHD 5 mm Multinu
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 64
 DS 4
 SWH 7507.507 Hz
 FIDRES 0.229111 Hz
 AQ 2.1823988 sec
 RG 90.5
 DW 66.600 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P0 5.80 usec
 PL1 -4.00 dB
 SFO1 500.0335002 MHz

F2 - Processing parameters
 SI 16384
 SF 500.0300107 MHz
 WDW EN
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR Plot Parameters
 CX 32.00 cm
 F1P 10.000 ppm
 F1 5000.30 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.31250 ppm/cm
 HZCM 156.25937 Hz/cm

Current Data Parameters
 NAME atobe
 EXPNO 26
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20040216
 Time 19.51
 INSTRUM QNP400
 PROBHD 5 mm QNP 1H
 PULPROG zgpg30
 TD 32768
 SOLVENT CDCl3
 NS 64
 DS 0
 SWE 6793.478 Hz
 FIDRES 0.207320 Hz
 AQ 2.4117749 sec
 RG 645.1
 DW 73.600 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====

NUC1 1H
 P1 7.40 usec
 PL1 -6.00 dB
 SFO1 400.1326354 MHz

F2 - Processing parameters

SI 16384
 SF 400.1300095 MHz
 WDW EM
 SSF 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters

CA 12.00 cm
 FI 10.000 ppm
 F1 400.130 Hz
 F2 0.000 ppm
 F3 0.00 Hz
 FPMCH 0.31250 ppm/cm
 FSCN 125.04063 Hz/cm

Current Data Parameters
 NAME arsyne
 EXPNO 12
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040306
 Time_ 15.12
 INSTRUM dpx400
 PROBHD 5 mm QNP 1H
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 64
 DS 0
 SWH 6793.478 Hz
 FIDRES 0.207320 Hz
 AQ 2.411749 sec
 RG 256
 DW 73.600 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 7.40 usec
 PL1 -6.00 dB
 SFO1 400.1326354 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300095 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 32.00 cm
 KY 10.000 gpm
 F1P 4001.30 Hz
 F1 0.000 gpm
 F2P 0.00 Hz
 F2 0.000 gpm
 FREQM 0.31250 ppm/cm
 HZCM 125.04663 Hz/cm

Current Data Parameters
NAME t-Bu-amine
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040228
Time 17.44
INSTRUM dx500
PROBHD 5 mm Multinu
PULPROG zg0
TD 32768
SOLVENT CDCl3
NS 64
DS 4
SWH 7507.507 Hz
FIDRES 0.22911 Hz
AQ 2.182398 sec
RG 16
DW 66.600 usec
DE 6.00 usec
TE 300.0 K
D1 1.0000000 sec

===== CHANNEL f1 =====
NUC1 1H
P0 5.80 usec
PL1 -4.00 dB
SFO1 500.035002 MHz

F2 - Processing parameters
SI 16384
SF 500.030000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
FLP 10.000 ppm
F1 5000.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.31250 ppm/cm
HZCM 156.25937 Hz/cm

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8. Configuration of this material was assigned to be *S* by conversion of (*S*)-**43** to (*S*)-2-(1-amino-2,2-dimethyl-propyl)phenol: $[\alpha]_{\text{D}}^{20} +20.7$ (*c* 0.2, CDCl₃) [for lit. (*R*)-2-(1-amino-2,2-dimethyl-propyl)phenol: $[\alpha]_{\text{D}}^{21} -21.1$ (*c* 2.06, CDCl₃) (Bernardinelli, G.; Fernandez, D.; Gosmini, R.; Meier, P.; Ripa, A.; Schüpfer, P.; Treptow, B.; Kündig, E. P. *Chirality* **2000**, *12*, 529–539)].