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An Expedient Total Synthesis of *cis*-(+)-Sertraline from D-Phenylglycine

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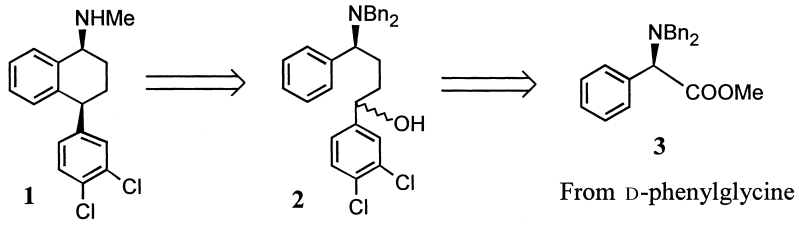
Abstract—An efficient and practical total synthesis of *cis*-(+)-Sertraline is developed involving intramolecular Friedel–Crafts cyclization of an appropriately tailored D-phenylglycine.

Depression is a serious and horrifying mental disorder and only a few drugs have been discovered with minimal side effects.[1] The slow onset of clinical improvement with these antidepressants has led to recent proposals that desensitization of norepinephrine transmission is involved in the therapeutic activity of the antidepressant.[2] Other investigations have suggested a role for serotonin in depression.[3,4] The search for more selective serotonin uptake blockers with reduced anticholinergic and cardiovascular liabilities of the tricyclic antidepressants has resulted in newer agents.[5] Sertraline **1** is one such clinical compound that acts on the central nervous system as a selective serotonin reuptake inhibitor (SSRI).[6] Sertraline **1**, which is marketed as Zoloft®, is one of the best selling second generation antidepressants.[7] The total synthesis of Sertraline has attracted quite a few synthetic organic chemistry laboratories, however, most efforts involve the synthesis of a racemic version and later resolution into the required *cis*-(+) form. Chiral syntheses of Sertraline are reported by Corey,[9a] and Lautens,[9c] involving asymmetric catalysis. Our continued interest in the development of new and efficient synthetic routes to clinically significant amino compounds[8] has prompted us to investigate the total synthesis of Sertraline. Described herein is an altogether different approach starting from cheap and readily accessible D-phenylglycine for the first time (Scheme 1).[9]



Scheme 1.

Reaction of the methyl ester of *N*,*N*-dibenzylphenylglycine **3**[10] with $LiAlH_4$ in anhydrous THF for 3 h afforded the glycinol derivative **4**. A one-pot[11] Swern oxidation and Wittig reaction with ethyltriphenylphosporane in CH_2Cl_2 yielded the unsaturated ester **5** (72%). To circumvent the poor yielding one step reduction of **5** to **7** with $LiAlH_4$, a high yielding two step protocol was adapted. Accordingly, the unsaturation in **5** is reduced with Mg in MeOH followed by reduction of the ester group with $LiAlH_4$ (76%) (Scheme 2).

The dichlorophenyl group was introduced through a Grignard reaction between in situ generated 3,4-dichlorophenylmagnesium bromide[12] and 4-*N*,*N*-dibenzyl-4-phenyl-*n*-butanal obtained upon oxidation of **7**. The crucial intramolecular Friedel–Crafts cyclization was attempted with various Lewis acids, however, $AlCl_3$ turned out to be best in yields and stereoselectivity ($AlCl_3$, CH_2Cl_2, RT, 78% yield from **2**, ds 80%).[13] The minor *trans*-isomer **8a** was eliminated by column chromatography using ethyl acetate/hexane (2:98). The *cis* tricyclic intermediate **8** upon debenzylation ($Pd\text{-}(OH)_2/H_2$),[14] protection of the free

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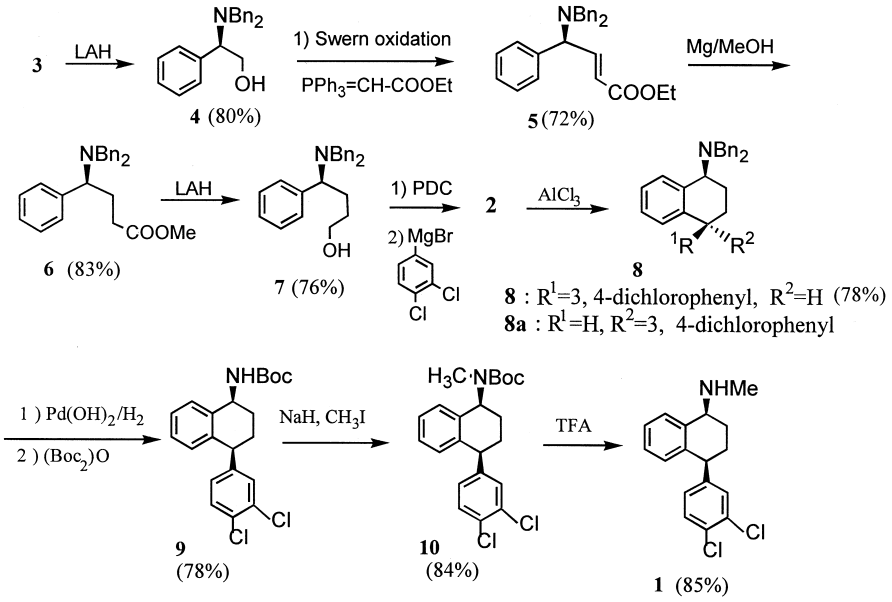

Scheme 2.

amino group with $(Boc)_2O$ and methylation (NaH, MeI) followed by Boc removal furnished uneventfully the desired targeted compound (+)-**1** whose data was comparable with literature.[9]

In conclusion, a practical and enantiopure synthesis of *cis*-(+)-Sertraline is described starting from chiral pool D-phenylglycine for the first time.[15]

Experimental

General methods

Crude products were purified by column chromatography on Silica gel (60–120 mesh). ^{1}H NMR were obtained in $CDCl_3$ at 200 MHz. Chemical shifts are given in ppm, with respect to internal TMS, *J* values are quoted in Hz. Infrared spectra were obtained neat, only the most significant absorptions in cm^{-1} are indicated. DMSO, triethylamine and CH_2Cl_2 were distilled from CaH_2 and stored over molecular sieves. Benzene and THF were dried over sodium, benzophenone. All reactions were carried out under nitrogen atmosphere using dry glassware.

(2*S*)-*N*,*N*-(Dibenzyl)-2-amino-2-phenyl-1-ethanol (4). A solution of ester **3** (15 g, 43 mmol) in THF (20 mL) was added to a suspension of $LiAlH_4$ (2 g, 52 mmol) in dry THF (100 mL) at 0°C. The suspension was stirred for 30 min at the same temperature and at room temperature overnight. The reaction mixture was then cooled to 0°C and the excess reagent was quenched by using 15% aqueous NaOH solution (4 mL) and (water 6 mL). After stirring for 30 min, the solution was filtered through a pad of silica and Na_2SO_4 and the filtrate was concentrated under vacuum. The oily residue was purified by column chromatography (ethyl acetate/hexane 1:4) to yield **4** (11 g, 80%) as a colorless viscous oil. [Anal. Calcd for $C_{22}H_{23}NO$: C, 83.24; H, 7.30; N, 4.41. Found: C, 83.40; H, 7.25; N, 4.50]; R_f (20% ethyl acetate/hexane) 0.5; $[\alpha]_D^{25}=-116.7$ (*c* 2, $CHCl_3$); IR (neat): 3500, 3030, 1600, 1350 cm^{-1}; ^{1}H NMR (200 MHz, $CDCl_3$); δ 7.45–7.25 (15H, m, 3×*Ph*–CH_2N) 4.15 (1H, t, *J*=10.6 Hz, *CH*–NBn_2), 3.95 (2H, d AB system, *J*=13.8 Hz, N–*CH*$_2$–Ph), 3.6 (2H, dd, *J*=4.2, 10.6 Hz, *CH*$_2$–OH), 3.15 (2H, d AB system, *J*=13.8 Hz, N–*CH*$_2$Ph); EI MS *m*/*z*: 299[M−18]$^+$, 286, 91.

(4*S*)-Ethyl-4-*N*,*N*-(Dibenzyl)-4-amino-4-phenyl-(*E*)-2-butenoate (5). To a stirred solution of oxalyl chloride (2.14 g, 17 mmol) in dichloromethane (15 mL) at −78°C under nitrogen atmosphere was added DMSO (1.6 mL, 18.9 mmol) dropwise. After stirring for 30 min a solution of the amino alcohol **4** (3 g, 9.46 mmol) in dichloromethane (25 mL) was added over 15 min. The mixture was warmed to −45°C and stirring was continued for 1 h at this temperature, then triethylamine (4.8 g, 47 mmol) was added. The reaction mixture was brought to 0°C and maintained for 15 min, then ethyltriphenyl phosphorane (3.9 g, 11.3 mmol) in benzene (15 mL) was added and the resulting solution was stirred for 15 h at room temperature. The solvent was removed under vacuum, the residue was washed with water, brine solution and dried over Na_2SO_4. The residue was purified by column chromatography (ethyl acetate/hexane 4:96) to yield the amino α,βunsaturated ester **5** (2.62 g, 72%) as a pale yellow viscous oil. [Anal. Calcd for $C_{26}H_{27}NO_2$: C, 81.01; H, 7.06; N, 3.63. Found: C, 81.05; H, 7.10; N, 3.53]; R_f (15% ethyl acetate/hexane) 0.75; $[\alpha]_D^{25}=+0.80$ (*c* 2, $CHCl_3$); IR (neat): 3030, 1700, 1600, 1350 cm^{-1}; ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.45–7.2 (16H, m, 3×*Ph*–*CH*=C), 6.0 (1H, d, *J*=16.2 Hz, *CH*=CH), 4.45 (1H, d, *J*=8.5 Hz, *CH*–NBn_2), 4.25 (2H, q, *J*=7.5 Hz, O–*CH*$_2$), 3.62 (4H, s, 2×*CH*$_2$Ph), 1.38 (3H, t, *J*=7.5 Hz, *CH*$_3$CH$_2$); EI MS *m*/*z*: 294 [M−91]$^+$, 131, 91.

(4*S*)-Methyl-*N*,*N*-(Dibenzyl)-4-amino-4-phenylbutanoate (6). To a stirred solution of ester **5** (2.0 g, 5.1 mmol) in dry methanol (50 mL) was added magnesium turnings (0.375 g, 15.5 mmol) and stirred for 4 h under nitrogen

atmosphere. The total reaction was taken into water (25 mL) and to this was added dilute acetic acid and stirred vigorously to get a clear solution. The acidic solution was treated with NH_4OH solution to adjust pH to 8.5 and extracted with ether (50 mL). The total extracts were washed with water (25 mL) saturated $NaHCO_3$ solution (20 mL) and dried over Na_2SO_4. The solvent was removed under vacuum to get the crude oily compound and was purified by column chromatography (ethyl acetate/hexane 5:95) to afford the pure ester **6** (1.6 g, 83%) as a yellow viscous oil. [Anal. Calcd for $C_{25}H_{27}NO_2$: C, 80.40; H, 7.29; N, 3.75. Found: C, 80.52; H, 7.40; N, 3.92]; R_f (20% ethyl acetate/hexane) 0.6; $[\alpha]_D^{25}$ −41.4 (*c* 1, $CHCl_3$); IR (neat): 2990, 1740, 1600, 1350 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$); δ 7.45–7.15 (15H, m, 3×*Ph*), 3.85 (2H, d AB system, *J*=13.8 Hz, N–*CH*$_2$Ph), 3.70 (1H, t, *J*=7.5 Hz, *CH*–NBn_2), 3.62 (3H, s, O*Me*), 3.12 (2H, d AB system, *J*=13.8 Hz, N–*CH*$_2$Ph), 2.45–2.30 (2H, m, *CH*$_2$), 2.15–2.0 (2H, m, *CH*$_2$–CHN); EI MS *m*/*z*: 373 $[M]^+$.

(4*S*)-*N*,*N*-(Dibenzyl)-4-amino-4-phenyl-1-butanol (7). A solution of ester **6** (1.5 g, 4 mmol) in THF (20 mL) was added to a suspension of $LiAlH_4$ (0.25 g, 6.1 mmol) in dry THF (50 mL) at 0°C and stirred for 30 min at the same temperature and at room temperature for overnight. The reaction mixture was cooled to 0°C and excess reagent was quenched by careful addition of 15% aqueous NaOH solution (0.5 mL) and (water 1 mL). After stirring for 30 min the solution was filtered through a pad of silica and Na_2SO_4 and filtrate was concentrated under vacuum. The crude product was purified by column chromatography (ethyl acetate/hexane 2:8) to afford the pure alcohol **7** (1.05 g, 76%) as a semisolid. [Anal. Calcd for $C_{24}H_{27}NO$: C, 83.44; H, 7.88; N, 4.05. Found: C, 83.53; H, 7.90; N, 4.07]; R_f (30% ethyl acetate/hexane) 0.35; $[\alpha]_D^{25}$=−49.20 (*c* 1, $CHCl_3$); IR (neat): 3500, 3030, 2990, 1600, 1350 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$); δ 7.40–7.40 (15H, m, 3×*Ph*), 3.8 (2H, d AB system, *J*=13.8 Hz, N–*CH*$_2$Ph), 3.7 (1H, t, *J*=5.9 Hz, *CH*–NBn_2), 3.55 (2H, t, *J*=5.9 Hz, *CH*$_2$–OH), 3.15 (2H, d AB system, *J*=13.8 Hz, N–*CH*$_2$–Ph), 1.95–1.5 (4H, m, *CH*$_2$–*CH*$_2$); HRMS (FAB) Calcd for $C_{24}H_{27}NO$ 346.2129; Found 346.2126.

(1*S*,*R*4*S*)-*N*,*N*-(Dibenzyl)-4-amino-1-(3,4-dicholrophenyl)-4-phenyl-1-butanol (2). To a stirred solution of **7** (1.0 g, 2.8 mmol) in dichloromethane (25 mL) was added pyridinium dichromate (2.1 g, 5.7 mmol) in portions with stirring at room temperature, after 5 h the reaction mixture was diluted with ether (50 mL) and filtered through a short silica gel column. The column was washed with ether (50 mL) and the filtrates were concentrated at low temperature to afford the aldehyde (0.85 g, 2.4 mmol). The aldehyde was added to a solution of 3,4 dichloro phenylmagnesium bromide {The solution of 3,4-dichloro phenylmagnesium bromide was prepared from 3,4-dichlorobromo benzene (1.1 g, 4.9 mmol and magnesium (0.209 g, 8.7 mmol) in THF (20 mL)}. The reaction mixture was stirred for 5 h, quenched with saturated ammonium chloride solution (15 mL), and compound was extracted with ether (50 mL). The combined extracts were washed with water (25 mL) brine (25 mL) and dried over Na_2SO_4. The solvent was removed under vacuum to get the residue, which was purified by column chromatography (ethyl acetate/hexane 2:8) to yield **2** (1.0 g, 83%) as a diastereomeric mixture. [Anal. Calcd for $C_{30}H_{29}Cl_2NO$: C, 73.47; H, 5.96; N, 2.86; Cl, 14.46 Found: C, 73.50; H, 5.89; N, 2.85; Cl, 14.50]; R_f (25% ethyl acetate/hexane) 0.4; IR (neat): 3400, 2990, 1600, 1350, 825 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$); δ 7.4–6.95 (18H, m, 3×*Ph*, *Ph*–Cl_2), 4.5 (1H, m, *CH*–$PhCl_2$), 3.75 (*2H* dd, AB system, *J*=6.5, 13.7 Hz, *CH*$_2$Ph), 3.65 (1H, m, *CH*–NBn_2), 3.05 (2H, dd, AB system *J*=6.5 13.7 Hz, N–*CH*$_2$Ph), 1.9–1.5 (4H, m, *CH*$_2$–*CH*$_2$); HRMS (FAB) Calcd for $C_{30}H_{29}Cl_2NO$; 490.1691. Found 490.1704.

(1*S*,4*S*)-*N*,*N*-(Dibenzyl)-4-(3,4-dicholrophenyl)-1,2,3,4-tetrahydro-1-napthalenamine (8). Compound **2** (0.5 g, 1 mmol) was dissolved in dry dichloromethane (10 mL) to obtain a clear solution. To this was added a solution of aluminium chloride (0.1 g, 0.2 mmol) in dry dichloromethane (5 mL) and the resulting solution was stirred for 1 h at room temperature, and washed with water (20 mL) and brine (15 mL) dried over Na_2SO_4. The solvent was removed under vacuum. The residue was purified by column chromatography (ethyl acetate/hexane 2:98) to give the aryl compounds **8** and **8a**, the major being *syn* **8** (0.375 g, 78%), as a yellow viscous oil [Anal. Calcd for **8**; $C_{30}H_{27}Cl_2N$; C, 76.27; H, 5.76; N, 2.96; Cl, 15.01 Found: C, 76.40; H, 5.65; N, 2.85; Cl, 15.05]; R_f (15% ethyl acetate/hexane) 0.7; $[\alpha]_D^{25}$=−17.60 (*c* 1, $CHCl_3$); IR: 2990, 1600, 1350, cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$); δ 7.4–7.0 (15H, m, 3×*Ph*, *Ph*–Cl_2), 6.85 (1H, dd, *J*=8, 2 Hz, *Ph*–Cl_2), 6.7 (1H, d, *J*=8 Hz, *Ph*–Cl_2), 4.45–4.0 (2H, m, *CH*–NBn_2,*CH*–Ph–Cl_2), 3.8 (2H, d, AB system, *J*=13.7 Hz, N–*CH*$_2$–Ph), 3.45 (2H, d, AB system, *J*=13.7 Hz, N–*CH*$_2$–Ph), 2.25–2.15 (2H, m, *CH*$_2$–CHN), 1.9–1.6 (2H, m, *CH*$_2$–CH–Ph); HRMS (FAB): Calcd for 472.1443; Found 472.1412.

***cis*-(1*S*,4*S*)-*N*-(*tert*-Butoxycarbonyl)-4-(3,4-dichlorophenyl)-1,2,3,4-tetrahydro-1-napthalenamine (9).** Solution of compound **8** (0.25 g, 0.53 mmol) in methanol (10 mL) was hydrogenated in presence of $Pd(OH)_2$ under hydrogen atmosphere. After stirring the mixture vigorously for 3 h, $(Boc)_2O$ (0.138 g, 0.63 mmol) was added to the reaction mixture and stirred for 3 h. The reaction mass was filtered and the filtrate was evaporated under vacuum to get the crude product which was purified by column chromatography (ethyl acetate/hexane 2:8) to afford a white solid **9** (0.162 g, 78%). mp 136–137°C; [Anal. Calcd for $C_{21}H_{23}Cl_2NO_2$ C, 64.29; H, 5.91; N, 3.57; Cl, 18.07. Found: C, 64.35; H, 6.00; N, 3.50; Cl, 18.10]; R_f (25% ethyl acetate/hexane) 0.5; $[\alpha]_D^{25}$=−1.80 (*c* 1, $CHCl_3$); IR (KBr): 3300, 1697, 1350, 1160 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 7.4–7.1 (5H, m, *Ph*–*Ph*Cl_2), 6.9 (1H, dd, *J*=8, 2 Hz, *Ph*–Cl_2), 6.8 (1H, d, *J*=6.8 Hz, *Ph*–Cl_2), 4.9 (1H, bs, *NH* Boc), 4.8 (1H, distorted t, *J*=2.5 Hz, *CH*–NH), 4.1 (1H, br t, *J*=6.8 Hz, *CH*–$PhCl_2$), 2.25–2.1 (2H, m, *CH*$_2$CHN), 2.0–1.80 (2H, m, *CH*$_2$–CHPh), 1.50 (9H, s, O–C– $(CH_3)_3$), EI MS *m*/*z* 334 $(M-57)^+$, 290, 249, 77.

***cis*-(1*S*,4*S*)-*N*-(*tert*-Butoxycarbonyl)-*N*-methyl-4-(3,4-dichlorophenyl)-1,2,3,4-tetrahydro-1-napthalenamine (10).** A solution of compound **9** (0.15 g, 0.38 mmol) in THF (2 mL) was added dropwise to a well-stirred suspension of oil free NaH (0.02 g, 76 mmol) in THF (10 mL). After 30 min, methyl iodide (0.136 g, 0.95 mmol) was added and the reaction mixture was stirred for 6 h. The reaction

mass was quenched with saturated NH_4Cl solution (5 mL) and the solution was extracted with ether (25 mL). The organic layers was washed with water (15 mL), brine (10 mL) and dried over Na_2SO_4. Removal of volatiles gave the crude compound, which was purified by column chromatography (ethyl acetate/hexane, 1:9) to give the compound **10** (0.13 g, 84%) as a semi solid. [Anal. Calcd for $C_{22}H_{25}Cl_2NO_2$; C, 65.03; H, 6.20; N, 3.45; Cl, 17.45. Found. C, 65.05; H, 6.30; N, 3.40; Cl, 17.60.]; R_f (15% ethyl acetate/hexane) 0.6; $[\alpha]_D^{25}=-19.0$ (*c* 1, $CHCl_3$); IR (neat): 1693, 1600, 1350, 1160 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 7.4–7.05 (5H, m, *Ph*, *Ph*Cl_2). 6.9 (1H, br d, *J*=8 Hz, *Ph*–Cl_2), 6.8 (1H, d, *J*=7 Hz, *Ph*–Cl_2) 5.4 (1H, br t, *J*=4.8 Hz, *CH*–NH), 4.15 (1H, distorted t, *CH*–PhCl_2), 2.65 (3H, s, N–CH_3), 2.4–2.2 (2H, m, CH_2–CHN), 2.05–1.9 (2H, m, CH_2–CHPh), 1.5 (9H, s, O–C–(CH_3)$_3$); FAB MS *m*/*z* 348 $(M-57)^+$, 275, 130, 77.

***cis*-(1*S*,4*S*)-*N*-Methyl-4-(3,4-dichlorophenyl)-1,2,3,4-tetrahydro napthalenamine hydrochloride (1).** The compound **10** (0.1 g, 0.3 mmol) was dissolved in dry dichloromethane (10 mL) to obtain a clear solution. To this was added a cold solution of trifluoroacetic acid (0.068 g, 0.6 mmol) in dry dichloromethane (5 mL). The resulting solution was stirred for 1 h at room temperature. The reaction mixture was poured into cold water (10 mL), washed with brine and dried over Na_2SO_4. Removal of the volatiles gave compound **1** (0.064 g, 85%). The compound **1** was dissolved in dry ether and dry HCl gas was passed through the solution to form the hydrochloride salt of compound **1** $[\alpha]_D^{25}=+37.9$ (*c* 2, CH_3OH). (Lit.[9] $[\alpha]_D^{25}=+37.9$) mp 242–244°C (lit.[9] 243–245°C) The material was found to be identical in all other aspects to that reported earlier.[9]

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