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CONCISE SYNTHESIS OF DIARYLMETHYL-*1H*-PYRROLES

Meng-Yang Chang,* Tsun-Cheng Wu, and Ya-Jung Ko

Department of Applied Chemistry, National University of Kaohsiung, Kaohsiung 811, Taiwan. Email: mychang@nuk.edu.tw

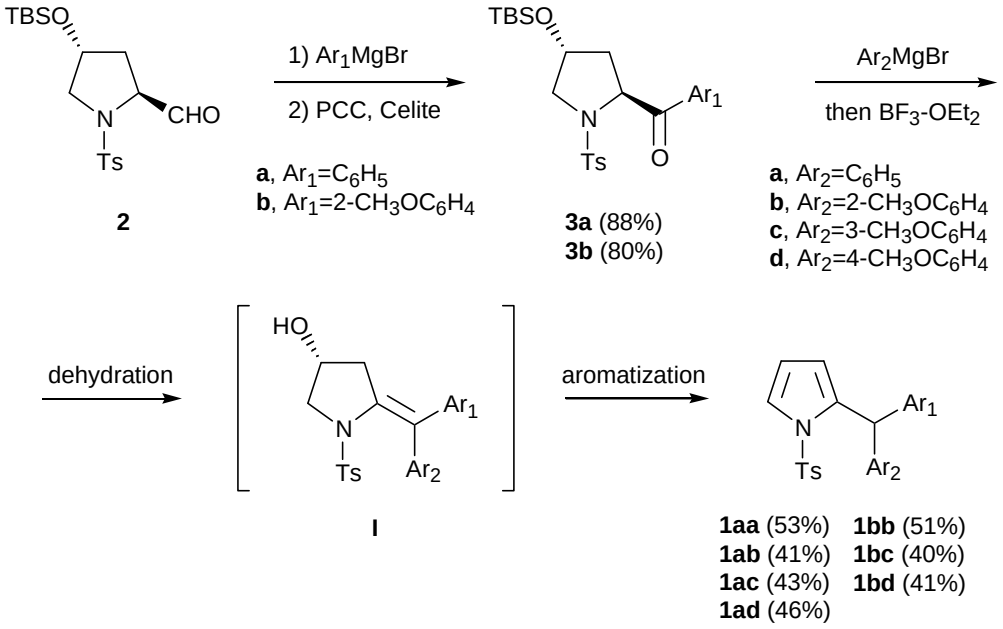
Abstract – An easy and straightforward synthesis of diarylmethyl-*1H*-pyrroles has been established starting from *trans*-(2*S*,4*R*)-4-hydroxyproline by the key combination of Grignard addition/aromatization.

Based on the structural framework of *trans*-(2*S*,4*R*)-4-hydroxyproline, it possesses three functional groups that can be easily modified.[1] The skeleton represents the significant feature for producing a series of different carbon framework using an efficient modification technique.[2] Recently we have introduced a straightforward approach to the (i) acyclic γ-amino acid (statine and vigabatrin®),[3a-b] (ii) monocyclic pyrrolidine (anisomycin)[3c] and piperidines (α-conhydrine, baikiain and pipecolic acid),[3d-e] (iii) bicyclic bridged 7-azabicyclo[2.2.1]heptane (epibatidine)[3f] and pyrrolophane (streptorubin B core)[3g] and fused hexahydro-1*H*-indol-3-one (pancracine)[3h] skeleton by employing *trans*-(2*S*,4*R*)-4-hydroxyproline as the starting material. To explore a new application of *trans*-(2*S*,4*R*)-4-hydroxyproline, synthetic studies toward diarylmethyl-*1H*-pyrrole were further investigated.

The wide-ranging applications of triaryl- or triheteroarylmethanes have been found in different areas of chemistry.[4] The classical dyestuff chemistry relies mainly on the triarylmethyl core, whereas the trityl group represents an important protective group for a range of functionalities.[4c] The related medicinal applications associated with various functional groups have attracted intense interests among synthetic and medicinal chemists in these compounds.[5] Accordingly, several useful synthetic strategies have been developed for the synthesis of triaryl- or triheteroarylmethanes with the facile and rapid controls.[6] Especially, there have been few reports in the synthesis of diarylmethyl-*1H*-pyrrole.[7] Here, we want to use the combination of Grignard addition/aromatization to achieve the structural framework of diarylmethyl-*1H*-pyrrole.

The approach toward aldehyde (**2**) from *trans*-(2*S*,4*R*)-4-hydroxyproline via our previous procedure was reported.[3] As shown in Scheme 1, the easy synthesis of seven compounds (**1aa**~**1bd**) was described as follows: (i) compounds (**3a**~**3b**) were provided via Grignard addition of aldehyde (**2**) with two

arylmagnesium bromide reagents (Ar_1, C_6H_5 and 2-$CH_3OC_6H_4$) and oxidation of the resulting alcohols with pyridinium chlorochromate and Celite in 88% and 80% yields; (ii) Grignard addition of ketones (**3a**~**3b**) with four arylmagnesium bromide reagents (Ar_2, C_6H_5, 2-$CH_3OC_6H_4$, 3-$CH_3OC_6H_4$ and 4-$CH_3OC_6H_4$) followed by boron trifluoride etherate-mediated dehydration gave tertiary alcohols in 40~53% overall yields.[8] Based on the experimental simplicity, the preparation of compound (**1bb**) was also conducted in a multigram scale (10 mmole) with 51% yield from ketone (**3b**).



Scheme 1. Synthesis of seven diarylmethyl-*1H*-pyrroles (**1aa**~**bd**)

How is the procedure initiated by boron trifluoride etherate? Intermediate (**I**) yielded by dehydration of the resulting tertiary alcohols. During the aromatization of intermediate (**I**), compounds (**1aa**~**bd**) were given immediately as the major products. The most likely explanation would be that it is controlled by involvement of the nitrogen lone pair. As shown in diagram 1, the structure of compound **(1bb**) was determined by a single-crystal X-ray analysis.[9]

Generally, the major approach to the triheteroarylmethanes having pyrrole motif was applied in the condensation of aldehyde and pyrrole and it could provide *meso*-substituted dipyrromethanes along with oligomeric byproducts under a variety of conditions (boron trifluoride etherate, propionic acid and trifluoroacetic acid).[10] The specific selectivity to products was not performed very well and the given yields were reduced due to the formation of oligmers and this calls for stringent purification methods to remove the byproducts. In comparison with the reported literature by us, this presented method could provide a flexible protocol to three different aromatic rings on a methane skeleton with unique selectivity. Although the synthesis of substituted triaryl- or triheteroarylmethanes have been developed, the present work is complementary to existing methodology.[11]

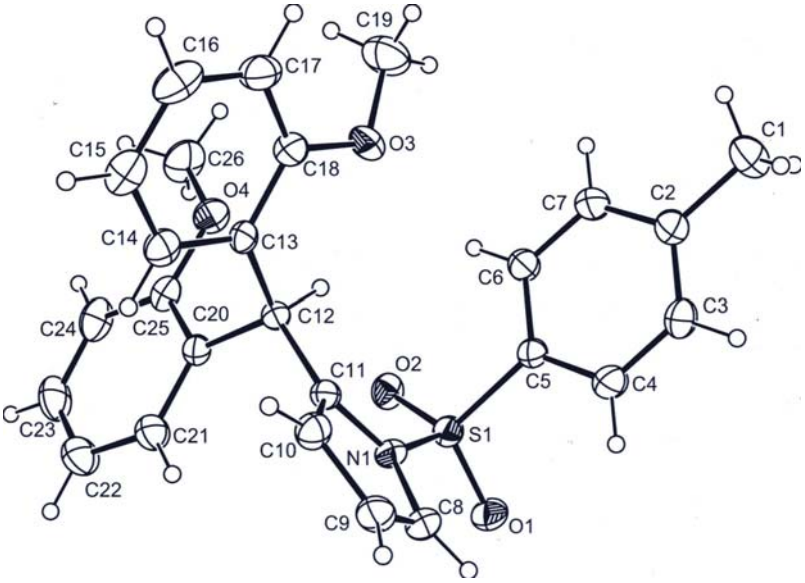


Diagram 1. X-ray crystallography of compound (**1bb**)

In summary, we developed a concise synthetic approach to sole diarylmethyl-*1H*-pyrrole skeleton based on the key combination of Grignard addition/aromatization. The photochemical reaction of the yielded compounds was further investigated.

EXPERIMENTAL

General. Tetrahydrofuran (THF) was distilled prior to use from a deep-blue solution of sodium-benzophenone ketyl. All other reagents were obtained from commercial sources and used without further purification. Reaction was routinely carried out under an atmosphere of dry nitrogen with magnetic stirring. Crude product was purified using column chromatography on SiO_2 (MN Kieselgel 60, 70~230 mesh).

A representative procedure of compounds (1aa-1bd) is as follows: A solution of arylmagnesium bromide (0.5 M in THF, 1 mL, 0.5 mmol) was added to a stirred solution of ketones (**3a**) or (**3b**) (0.3 mmol) in THF (5 mL) at -78 ^{o}C. The reaction mixture was stirred at rt for 2 h. The total procedure was monitored by TLC until the reaction was completed. Without further purification, boron trifluoride etherate (0.5 mL) was added to a stirred solution of the resulting reaction mixture at 0 ^{o}C. The reaction mixture was stirred at rt for 15 min. Saturated $NaHCO_{3(aq)}$ (1 mL) was added to the reaction mixture and the solvent was concentrated. Water (2 mL) and AcOEt (10 mL) was added to the residue and the mixture was filtered through a short plug of Celite. The filtrate was concentrated. The residue was extracted with AcOEt (3 x 20 mL). The combined organic layers were washed with brine, dried, filtered and evaporated to afford crude product. Purification on silica gel (hexane/AcOEt = 8/1~4/1) afforded compounds (**1aa-bd**).

2-Benzhydryl-1-(4-methylphenylsulfonyl)-*1H*-pyrrole (1aa): Viscous gum; IR ($CHCl_3$) 3027, 2924, 1598, 1367, 1173, 729 cm^{-1}; HRMS (ESI, M^++1) calcd for $C_{24}H_{22}NO_2S$ 388.1371, found 388.1370; ^1H NMR (500 MHz, $CDCl_3$) δ 7.37 (dd, *J* = 1.5, 3.5 Hz, 1H), 7.29 (d, *J* = 8.5 Hz, 2H), 7.17-7.14 (m, 6H), 7.01 (d, *J* = 8.5 Hz, 2H), 6.93 (dd, *J* = 3.5, 7.0 Hz, 4H), 6.21 (t, *J* = 3.5 Hz, 1H), 6.07 (s, 1H), 5.72-5.71 (m, 1H), 2.34 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.16, 142.40 (2x), 137.30, 135.85, 129.50 (2x), 128.99 (4x), 128.16 (4x), 126.76 (2x), 126.28 (2x), 122.91, 116.07, 111.04, 48.39, 21.49; Anal. Calcd for $C_{24}H_{21}NO_2S$: C, 74.39; H, 5.46; N, 3.61. Found: C, 74.69; H, 5.67; N, 3.88.

2-[(2-Methoxyphenyl)-phenyl-methyl]-1-(4-methylphenylsulfonyl)-*1H*-pyrrole (1ab): Viscous gum; IR ($CHCl_3$) 3027, 2917, 1597, 1491, 1367, 1246, 1149, 755 cm^{-1}; HRMS (ESI) *m*/*z* calcd for $C_{25}H_{24}NO_3S$ (M^++1) 418.1477, found 418.1481; ^1H NMR (500 MHz, $CDCl_3$) δ 7.42 (d, *J* = 8.0 Hz, 2H), 7.35 (dd, *J* = 2.0, 3.5 Hz, 1H), 7.17-7.14 (m, 4H), 7.05 (d, *J* = 8.0 Hz, 2H), 6.93 (d, *J* = 8.0 Hz, 1H), 6.93-6.91 (m, 1H), 6.81 (dd, *J* = 1.0, 8.0 Hz, 1H), 6.68 (dt, *J* = 1.0, 8.0 Hz, 1H), 6.61 (dd, *J* = 1.0, 8.0 Hz, 1H), 6.34 (s, 1H), 6.18 (t, *J* = 7.0 Hz, 1H), 5.68-5.66 (m, 1H), 3.68 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 154.50, 141.80, 140.28, 135.03, 133.83, 128.94, 127.30, 127.17 (2x), 126.64 (2x), 125.76 (2x), 125.27, 124.67 (2x), 123.81, 120.51, 117.84, 113.49, 108.64, 108.33, 53.32, 39.67, 19.27; Anal. Calcd for $C_{25}H_{23}NO_3S$: C, 71.92; H, 5.55; N, 3.35. Found: C, 71.66; H, 5.79; N, 3.63.

2-[(3-Methoxyphenyl)-phenyl-methyl]-1-(4-methylphenylsulfonyl)-*1H*-pyrrole (1ac): Viscous gum; IR ($CHCl_3$) 2913, 1597, 1489, 1365, 1173, 1148, 730 cm^{-1}; HRMS (ESI) *m*/*z* calcd for $C_{25}H_{24}NO_3S$ (M^++1) 418.1477, found 418.1482; ^1H NMR (500 MHz, $CDCl_3$) δ 7.37 (dd, *J* = 2.0, 3.5 Hz, 1H), 7.29 (d, *J* = 8.0 Hz, 2H), 7.17-7.14 (m, 3H), 7.08 (t, *J* = 8.0 Hz, 1H), 7.01 (d, *J* = 8.0 Hz, 2H), 6.94 (d, *J* = 3.5 Hz, 1H), 6.93 (d, *J* = 2.5 Hz, 1H), 6.68 (dd, *J* = 2.5, 8.5 Hz, 1H), 6.55 (d, *J* = 8.0 Hz, 1H), 6.39 (s, 1H), 6.21 (t, *J* = 3.5 Hz, 1H), 6.03 (s, 1H), 5.73-5.72 (m, 1H), 3.65 (s, 3H), 2.33 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.40, 144.12, 143.91, 142.31, 137.11, 135.81, 129.43 (2x), 129.09, 128.96 (2x), 128.17 (2x), 126.77 (2x), 126.33, 122.92, 121.63, 116.08, 114.75, 111.58, 111.04, 54.95, 48.34, 21.49.

2-[(4-Methoxyphenyl)-phenyl-methyl]-1-(4-methylphenylsulfonyl)-*1H*-pyrrole (1ad): Viscous gum; IR ($CHCl_3$) 2913, 1594, 1510, 1365, 1248, 1172 cm^{-1}; HRMS (ESI) *m*/*z* calcd for $C_{25}H_{24}NO_3S$ (M^++1) 418.1477, found 418.1477; ^1H NMR (500 MHz, $CDCl_3$) δ 7.36 (dd, *J* = 2.0, 3.5 Hz, 1H), 7.29 (d, *J* = 8.5 Hz, 2H), 7.16-7.13 (m, 4H), 7.01 (d, *J* = 8.5 Hz, 2H), 6.93-6.91 (m, 1H), 6.82 (d, *J* = 8.5 Hz, 2H), 6.68 (d, *J* = 8.5 Hz, 2H), 6.20 (t, *J* = 3.5 Hz, 1H), 6.00 (s, 1H), 5.70-5.69 (m, 1H), 3.76 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 158.12, 144.05, 142.73, 137.78, 135.91, 134.53, 129.93 (2x), 129.44 (2x), 128.91 (2x), 128.14 (2x), 126.76 (2x), 126.22, 122.91, 115.89, 113.49 (2x), 110.98, 55.15, 47.60, 21.49; Anal. Calcd for $C_{25}H_{23}NO_3S$: C, 71.92; H, 5.55; N, 3.35. Found: C, 71.80; H, 5.64; N, 3.60.

2-[Bis-(2-methoxyphenyl)-methyl]-1-(4-methylphenylsulfonyl)-*1H*-pyrrole (1bb): mp = 201-202 ^oC; IR ($CHCl_3$) 2941, 1598, 1490, 1462, 1368, 1244, 1175, 1149, 754 cm^{-1}; HRMS (ESI, M^++1) calcd for

$C_{26}H_{26}NO_4S$ 448.1583, found 448.1585; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.57 (d, *J* = 8.0 Hz, 2H), 7.33 (dd, *J* = 1.5, 3.5 Hz, 1H), 7.16 (dt, *J* = 1.5, 8.0 Hz, 2H), 7.11 (d, *J* = 8.0 Hz, 2H), 6.82 (d, *J* = 8.0 Hz, 2H), 6.71 (t, *J* = 7.5 Hz, 2H), 6.63 (dd, *J* = 1.5, 7.5 Hz, 2H), 6.57 (s, 1H), 6.15 (t, *J* = 3.5 Hz, 1H), 5.62 (s, 1H), 3.66 (s, 6H), 2.36 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 156.91 (2x), 143.92, 137.34, 136.26, 131.29 (2x), 129.32 (2x), 129.12 (2x), 127.29 (2x), 127.09 (2x), 122.49, 120.00 (2x), 115.17, 110.73, 111.72 (2x), 55.66 (2x), 35.91, 21.49; Anal. Calcd for $C_{26}H_{25}NO_4S$: C, 69.78; H, 5.63; N, 3.13. Found: C, 69.62; H, 5.52; N, 3.42. Single-crystal X-ray diagram: crystal of compound (**1bb**) was grown by slow diffusion of CH_2Cl_2. The compound crystallizes in the triclinic crystal system, space group P -1 (No. 2), *a*=7.8767(16) Å, *b*=9.4870(19) Å, *c*=16.389(3) Å, *V*=1115.0(4) Å^3, *Z*=2, d_{calcd}=1.333 mg/m^3, absorption coefficient 0.179 mm^{-1}, *F*(000)=472, 2θ range (2.35~26.00^o), final R indices (all data), R_1=0.1771 and wR_2=0.1328.

2-[(2-Methoxyphenyl)-(3-methoxyphenyl)-methyl]-1-(4-methylphenylsulfonyl)-*1H*-pyrrole (1bc): mp = 127-128 ^{o}C; IR ($CHCl_3$) 3014, 2916, 1598, 1490, 1367, 1246, 1174, 1149, 752 cm^{-1}; HRMS (ESI) *m*/*z* calcd for $C_{26}H_{26}NO_4S$ 448.1583, found 448.1585; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 (d, *J* = 8.0 Hz, 2H), 7.35 (br s, 1H), 7.15 (t, *J* = 7.5 Hz, 1H), 7.08 (t, *J* = 7.5 Hz, 1H), 7.04 (d, *J* = 8.0 Hz, 2H), 6.81 (d, *J* = 8.0 Hz, 1H), 6.70-6.67 (m, 2H), 6.62 (d, *J* = 7.5 Hz, 1H), 6.54 (d, *J* = 7.5 Hz, 1H), 6.41 (s, 1H), 6.32 (s, 1H), 6.17 (t, *J* = 3.5 Hz, 1H), 5.69 (br s, 1H), 3.70 (s, 3H), 3.66 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.32, 156.73, 144.10, 144.00, 137.09, 136.05, 131.07, 129.51, 129.35 (2x), 128.89, 127.55, 126.91 (2x), 122.75, 121.56, 120.10, 115.66, 114.72, 111.37, 110.88, 110.57, 55.59, 54.94, 41.80, 21.50; Anal. Calcd for $C_{26}H_{25}NO_4S$: C, 69.78; H, 5.63; N, 3.13. Found: C, 69.58; H, 5.28; N, 3.47.

2-[(2-Methoxyphenyl)-(4-methoxyphenyl)-methyl]-1-(4-methylphenylsulfonyl)-*1H*-pyrrole (1bd): Viscous gum; IR ($CHCl_3$) 2907, 1597, 1510, 1366, 1245, 1174, 755 cm^{-1}; HRMS (ESI) *m*/*z* calcd for $C_{26}H_{26}NO_4S$ 448.1583, found 448.1586; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 (d, *J* = 8.5 Hz, 2H), 7.34 (dd, *J* = 1.5, 3.0 Hz, 1H), 7.14 (dt, *J* = 1.5, 7.5 Hz, 1H), 7.05 (d, *J* = 8.5 Hz, 2H), 6.83 (d, *J* = 8.5 Hz, 2H), 6.80-6.78 (m, 2H), 6.69 (d, *J* = 8.0 Hz, 2H), 6.60 (dd, *J* = 1.5, 7.5 Hz, 1H), 6.28 (s, 1H), 6.17 (t, *J* = 3.5 Hz, 1H), 5.66 (s, 1H), 3.76 (s, 3H), 3.69 (s, 3H), 2.35 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 157.93, 156.73, 143.95, 137.74, 136.11, 134.57, 131.49, 129.81 (2x), 129.44, 129.36 (2x), 127.44, 126.89 (2x), 122.74, 120.07, 115.48, 113.37 (2x), 110.83, 110.57, 55.58, 55.11, 41.10, 21.50; Anal. Calcd for $C_{26}H_{25}NO_4S$: C, 69.78; H, 5.63; N, 3.13. Found: C, 69.69; H, 5.52; N, 3.32.

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