

**Synthesis of [3]Ferrocenophanes via Samaruim Diiodide Promoted Reductive
Cyclizations of 1,1'-Bis(cinnamoyl)ferrocenes**

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Supporting Information:

Additional experimental procedures and spectral data of new compounds.

Experimental Section

Melting points are uncorrected. Chemical shifts are reported relative to CHCl_3 (δ_{H} 7.26) and CDCl_3 [δ_{C} (central line of t) 77.0]. All reactions requiring anhydrous conditions were conducted in flame-dried apparatus under an atmosphere of nitrogen. Syringes and needles for the transfer of reagents were dried at 120 °C and allowed to cool in a desiccator over P_2O_5 before use. Ethers were distilled from sodium benzophenone ketyl, and (chlorinated) hydrocarbons from CaH_2 . Column chromatography was carried out on Kieselgel 60 (40–63 μm). Merck silica gel 60F sheets were used for analytical thin-layer chromatography.

1,1'-Dicinnamoylferrocene (1a).¹ Compound **1a** was prepared in 63% yield by condensation of 1,1'-diacetylferrocene (2.02 g, 7.5 mmol) with benzaldehyde (2.06 g, 20 mmol) in the presence of NaOH (200 mg) in MeOH solution (20 mL) according to the known procedure.²

1a: Red solid, mp 180–182 °C; IR (KBr) 1657, 1599 cm^{-1} ; ^1H NMR (CDCl_3 , 200 MHz) δ 4.58 (4 H, s), 4.91 (4 H, s), 7.03(2 H, d, J = 15.6 Hz), 7.34–758 (10 H, m), 7.74 (2 H, d, J = 15.6 Hz); ^{13}C NMR (CDCl_3 , 50 MHz) δ 71.3 (4 ×), 74.2 (4 ×), 81.9 (2 ×), 122.4 (2 ×), 128.4 (4 ×), 128.8 (4 ×), 130.3 (2 ×), 134.8 (2 ×), 142.1 (2 ×), 192.1 (2 ×). FAB-MS m/z 446 (M^+).

1,1'-Bis(4-methylcinnamoyl)ferrocene (1b). Compound **1b** was prepared in 55% yield by condensation of 1,1'-diacetylferrocene (1.12 g, 4 mmol) with 4-methylbenzaldehyde (1.20 g, 10 mmol) in the presence of NaOH (200 mg) in MeOH solution (20 mL) by a procedure similar to that for **1a**.

1b: Red solid, mp 230 °C (decomposed); IR (KBr) 1653, 1597 cm^{-1} ; ^1H NMR (CDCl_3 , 200 MHz) δ 2.35 (6 H, s), 4.57 (4 H, s), 4.90 (4 H, s), 7.00 (2 H, d, J = 15.5 Hz), 7.12 (4 H, d, J = 7.6 Hz), 7.45 (4 H, d, J = 7.6 Hz), 7.73 (2 H, d, J = 15.5 Hz); ^{13}C NMR (CDCl_3 , 50 MHz) δ 21.5 (2 ×), 71.2 (4 ×), 74.1(4 ×), 77.2 (2 ×) 121.4 (2 ×), 128.4 (4 ×), 129.6 (4 ×), 132.0 (2 ×), 140.7 (2 ×), 142.1 (2 ×), 192.5 (2 ×). FAB-MS m/z 474 (M^+). HRMS Calcd for $\text{C}_{30}\text{H}_{26}\text{FeO}_2$:

474.1282. Found: 474.1279.

1,1'-Bis(3-methoxycinnamoyl)ferrocene (1c). Compound **1c** was prepared in 68% yield by condensation of 1,1'-diacetylferrocene (2.10 g, 7.8 mmol) with 3-methoxybenzaldehyde (2.60 g, 20 mmol) in the presence of NaOH (200 mg) in MeOH solution (20 mL) by a procedure similar to that for **1a**.

1c: Red solid, mp 155–157 °C; IR (KBr) 1653, 1597 cm⁻¹; ¹H NMR (CDCl₃, 200 MHz) δ 3.83 (6 H, s), 4.89 (4 H, s), 4.90 (4 H, s), 6.91 (4 H, d, *J* = 7.8 Hz), 7.03 (2 H, d, *J* = 15.5 Hz), 7.11 (2 H, s), 7.18 (4 H, d, *J* = 7.8 Hz), 7.27 (4 H, t, *J* = 7.8 Hz), 7.70 (2 H, d, *J* = 15.5 Hz); ¹³C NMR (CDCl₃, 50 MHz) δ 55.3 (2 ×), 71.4 (4 ×), 74.1 (4 ×), 81.9 (2 ×), 113.4 (2 ×), 116.1 (2 ×), 121.0 (2 ×), 122.6 (2 ×), 129.8 (2 ×), 136.1 (2 ×), 141.9 (2 ×), 159.8 (2 ×), 192.0 (2 ×). FAB-MS *m/z* 506 (M⁺). HRMS Calcd for C₃₀H₂₆FeO₄: 506.1180. Found: 506.1154.

1,1'-Bis[(3-trifluoromethyl)cinnamoyl]ferrocene (1d). Compound **1d** was prepared in 86% yield by condensation of 1,1'-diacetylferrocene (1.12 g, 4 mmol) with 3-trifluoromethylbenzaldehyde (1.74 g, 10 mmol) in the presence of NaOH (200 mg) in MeOH solution (20 mL) by a procedure similar to that for **1a**.

1d: Red solid, mp 158–160 °C; IR (KBr) 1665, 1611 cm⁻¹; ¹H NMR (CDCl₃, 200 MHz) δ 4.63 (4 H, t, *J* = 1.9 Hz), 4.92 (4 H, t, *J* = 1.9 Hz), 7.03 (2 H, d, *J* = 15.7 Hz), 7.44 (2 H, t, *J* = 7.8 Hz), 7.57 (2 H, d, *J* = 7.8 Hz), 7.70 (2 H, d, *J* = 7.8 Hz), 7.72 (2 H, d, *J* = 15.7 Hz), 7.80 (2 H, s); HRMS Calcd for C₃₀H₂₀F₆FeO₂: 582.0717. Found: 582.0672.

1,1'-Bis(4-fluorocinnamoyl)ferrocene (1e). Compound **1e** was prepared in 91% yield by condensation of 1,1'-diacetylferrocene (2.18 g, 8 mmol) with 4-fluorobenzaldehyde (2.48 g, 20 mmol) in the presence of NaOH (200 mg) in MeOH solution (20 mL) by a procedure similar to that for **1a**.

1e: Red solid, mp 230 °C (decomposed); IR (KBr) 1662, 1602 cm⁻¹; ¹H NMR (CDCl₃, 200 MHz) δ 4.59 (4 H, t, *J* = 1.9 Hz), 4.89 (4 H, t, *J* = 1.9 Hz), 6.92 (2 H, d, *J* = 15.6 Hz), 7.13 (4 H, dd, *J* = 4.4, 8.8 Hz), 7.52 (4 H, dd, *J* = 5.4, 8.8 Hz), 7.67 (2 H, d, *J* = 15.6 Hz); FAB-MS

m/z 482 (M^+). HRMS Calcd for $C_{28}H_{20}F_2FeO_2$: 482.0781. Found: 482.0783.

1,1'-Bis(4-chlorocinnamoyl)ferrocene (1f). Compound **1f** was prepared in 71% yield by condensation of 1,1'-diacetylferrocene (2.18 g, 8 mmol) with 4-chlorobenzaldehyde (2.80 g, 20 mmol) in the presence of NaOH (200 mg) in MeOH solution (20 mL) by a procedure similar to that for **1a**.

1f: Red solid, mp >240 °C; IR (KBr) 1662, 1606 cm^{-1} ; ^1H NMR (CDCl_3 , 200 MHz) δ 4.59 (4 H, s), 4.89 (4 H, s), 6.95 (2 H, d, J = 15.4 Hz), 7.17–7.29 (4 H, m), 7.44 (4 H, d, J = 7.8 Hz), 7.64 (2 H, d, J = 15.4 Hz); FAB-MS *m/z* 514 (M^+). HRMS Calcd for $C_{28}H_{20}(^{35}\text{Cl})_2FeO_2$: 514.0190. Found: 514.0162.

Representative Procedure for the SmI₂ Promoted Reductive Cyclization of 1,1'-Dicinnamoylferrocenes. A deep blue SmI₂ solution (0.1 M, 1.8 mmol) was prepared by treatment of Sm (331 mg, 2.2 mmol) with 1,2-diiodoethane (507 mg, 1.8 mmol) in anhydrous THF (18 mL) for 1.5 h at room temperature. The SmI₂ solution was cooled in an ice bath, and a THF (100 mL) solution of 1,1-dicinnamolyferrocene (178 mg, 0.4 mmol) was added dropwise via a syringe pump over a period of 3 h. The mixture was stirred at 0 °C for an additional 3.5 h, and then filtered through a pad of silica gel by elution with EtOAc/hexane (1:1). The filtrate was concentrated, and chromatographed on a silica gel column by elution with gradients of EtOAc/hexane (0–25%) to give two isomers of [3]ferrocenophane diols, **4a**-minor (58 mg, 32 %, less retained in the column) and **4a**-major (88 mg, 49%).

When a THF solution of **1a** (0.5 mmol) was added dropwise to the freshly prepared SmI₂ solution (1.8 mmol) over a period of 1 h, followed by stirring at 0 °C for a brief period (10 min), dione **2a** and aldon **3a** were obtained in 23% and 44% yields. The ^1H NMR analyses indicated that both products existed as single isomers. The starting material was entirely recovered when 0.8 mmol of **1a** was treated with 0.45 mmol of SmI₂ at 0 °C for 2 h.

1,1'-Bis(3-phenylpropanoyl)ferrocene (2a). IR (KBr) 1681 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz) δ 2.62 (2 H, dd, J = 14.5, 2.8 Hz), 3.40 (2 H, dd, 14.5, 13.6 Hz), 3.97 (2 H, dd, J = 13.6,

2.8 Hz), 4.50 (2 H, br s), 4.60 (2 H, br s), 4.92 (2 H, br s), 5.01 (2 H, br s), 6.94–6.98 (4 H, m), 7.13–7.26 (6 H, m); ^{13}C NMR (CDCl_3 , 75 MHz) δ 42.7, 45.8, 70.3, 73.4, 73.5, 73.8, 79.9, 82.4, 126.6, 127.8, 128.3, 129.0, 141.2, 203.8; FAB-MS m/z 448 (M^+).

2,3-(1,2-Diphenylpropane-1,3-diyl)-3-hydroxy[3]ferrocenophane-1-one (3a). IR (KBr) 3397, 1676 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz) δ 2.42 (1 H, dd, $J = 13.6, 7.8$ Hz), 2.55 (1 H, dd, $J = 13.6, 9.7$ Hz), 2.82 (1 H, s, OH), 4.03 (1 H, br s), 4.04–4.40 (4 H, m), 4.49 (1 H, br s), 4.70 (1 H, br s), 4.91 (1 H, d, $J = 11.5$ Hz), 4.93 (1 H, br s), 5.00 (1 H, br s), 5.02 (1 H, br s), 5.20 (1 H, br s), 6.85–7.08 (10 H, m); ^{13}C NMR (CDCl_3 , 75 MHz) δ 29.7, 46.1, 47.8, 48.7, 67.2, 67.7, 68.7, 70.0, 70.7, 72.1, 72.4, 74.0, 79.0, 88.1, 95.6, 125.7, 127.7, 128.5, 128.6, 140.0, 142.1, 207.6; FAB-MS m/z 448 (M^+).

2,3-[1,2-Bis(3-methoxyphenyl)propane-1,3-diyl]-3-hydroxy[3]ferrocenophane-1-one (3c). A solution of **1c** (202 mg, 0.4 mmol) in THF (100 mL) was added dropwise to the freshly prepared SmI_2 solution (0.1 M, 0.9 mmol) at 0 °C via a syringe pump over a period of 1.5 h. The mixture was stirred for additional 1 h, and worked up as usual to give **30c** (143 mg, 70%) as a mixture of two isomers (69:31).

3c-major: Orange solid, mp 155–157 °C; TLC (EtOAc/hexane (1:4)) R_f 0.13; IR (KBr) 3411, 1654 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz) δ 2.39 (1 H, dd, $J = 7.6, 13.6$ Hz), 2.50 (1 H, dd, $J = 9.8, 13.6$ Hz), 2.79 (1 H, s), 3.61 (3 H, s), 3.64 (3 H, s), 3.70–5.10 (11 H, m), 6.41–7.21 (8 H, m); FAB-MS m/z 508 (M^+); HRMS Calcd for $\text{C}_{30}\text{H}_{28}\text{FeO}_4$: 508.1337. Found: 508.1324.

3c-minor: Orange solid, mp 188–190 °C; IR (KBr) 3440, 1679 cm^{-1} ; ^1H NMR (CDCl_3 , 500 MHz) δ 2.20 (1 H, dd, $J = 4.7, 14.5$ Hz), 2.69 (1 H, dd, $J = 12.0, 14.5$ Hz), 2.86 (1 H, s, D₂O exchangeable), 3.55 (1 H, m), 3.69 (3 H, s), 3.74 (3 H, s), 3.89 (1 H, dd, $J = 12.4, 9.7$ Hz), 4.00 (1 H, br s), 4.21 (1 H, br s), 4.29 (1 H, br s), 4.47 (1 H, br s), 4.63 (1 H, d, $J = 12.4$ Hz), 4.67 (1 H, br s), 4.90 (1 H, br s), 4.96 (1 H, br s), 5.15 (1 H, br s), 6.65 (1 H, s), 6.67 (1

H, d, J = 7.4 Hz), 6.69 (1 H, d, J = 7.4 Hz), 6.74 (1 H, d, J = 7.4 Hz), 6.85 (1 H, d, J = 7.4 Hz), 6.89 (1 H, s), 7.11-7.15 (2 H, m); ^{13}C NMR (CDCl_3 , 75 MHz) δ 49.6, 51.9, 52.2, 55.0, 55.1, 67.1, 67.9, 68.6, 70.6, 70.8, 71.7 (2 \times), 72.2, 72.5, 74.0, 88.6, 96.6, 111.7, 111.8, 113.5, 119.5, 120.3, 142.4, 146.4, 159.5, 207.7; FAB-MS m/z 508 (M^+); HRMS Calcd for $\text{C}_{30}\text{H}_{28}\text{FeO}_4$: 508.1337. Found: 508.1324.

2,3-[1,2-Bis(3-chlorophenyl)propane-1,3-diyl]-3-hydroxy[3]ferrocenophane-1-one (3f).

This reaction required an inverse addition due to the low solubility of **1f** in THF. The solution of **1f** (206 mg, 0.4mmol) in THF (100 mL) was cooled in an ice bath, and the SmI_2 solution was added. The mixture was stirred for 3.5 h, and worked up as usual to give **3f** (32 mg, 15%) along with a recovery of **1f**.

3f: Orange solid, mp 222 °C (decomposed); IR (KBr) 3422, 1674 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz) δ 2.38 (1 H, dd, J = 8.0, 13.7 Hz), 2.45 (1 H, dd, J = 9.9, 13.7 Hz), 2.71 (1 H, s), 4.03-5.14 (11 H, m), 6.77-7.58 (8 H, m); FAB-MS m/z 516 (M^+); HRMS Calcd for $\text{C}_{28}\text{H}_{22}\text{Cl}_2\text{FeO}_2$: 516.0346. Found: 516.0371.

2,3-(1,2-Diphenylpropane-1,3-diyl)[3]ferrocenophane-1,3-diol (4a).

4a-major: Orange solid, mp 229–231 °C; IR (KBr) 3437 cm^{-1} ; ^1H NMR (CDCl_3 , 200 MHz) δ 2.37 (1 H, dd, J = 9.3, 13.8 Hz), 2.46 (1 H, dd, J = 7.5, 13.8 Hz), 2.80 (1 H, dd, J = 1.9, 11.0 Hz), 3.23 (1 H, d, J = 5.7 Hz, OH), 3.93–4.64 (12 H, m), 6.87–7.10 (10 H, m); ^{13}C NMR (CDCl_3 , 50 MHz) δ 46.0 (2 \times), 47.7, 47.9, 65.5, 65.7 (2 \times), 67.1, 68.4, 68.9 (2 \times), 69.7, 70.2, 79.1, 90.0, 93.8, 125.5, 125.8, 127.5 (2 \times), 127.8 (2 \times), 128.7 (2 \times), 129.1 (2 \times), 141.1, 142.2. FAB-MS m/z 450 (M^+). HRMS. Calcd for $\text{C}_{28}\text{H}_{26}\text{FeO}_2$: 450.1280. Found: 450.1279.

The structure of **4a-major** was confirmed by an X-ray diffraction analysis.

4a-minor: Orange solid, mp 216 °C (decomposed); IR (KBr) 3512 cm^{-1} ; ^1H NMR (CDCl_3 , 500 MHz) δ 1.66 (1 H, s, OH), 2.37 (1 H, dd, J = 14.8, 4.9 Hz), 2.67 (1 H, t, J = 14.8 Hz), 2.69 (1 H, d, J = 12.6 Hz), 2.90 (1 H, d, J = 5.0 Hz, D_2O exchangeable), 3.64 (1 H, m),

4.00 (1 H, br d, J = 12.6 Hz), 4.02 (1 H, br s), 4.15 (1 H, br s), 4.20 (1 H, br s), 4.23 (2 H, br s), 4.26 (1 H, br s), 4.31 (1 H, br d, J = 5.0 Hz), 4.63 (1 H, br s), 4.73 (1 H, br s), 7.20–7.40 (10 H, m); ^{13}C NMR (CDCl_3 , 75 MHz) δ 29.7, 43.0, 52.9, 54.0, 65.1, 65.5, 67.0, 67.7, 68.2, 68.9, 69.0, 69.7, 72.2, 77.2, 91.0, 93.5, 126.0, 126.6, 127.7 (2 \times), 127.8 (2 \times), 128.2 (2 \times), 128.6 (2 \times), 141.7, 145.3. FAB-MS m/z 450 (M^+). HRMS. Calcd for $\text{C}_{28}\text{H}_{26}\text{FeO}_2$: 450.1280. Found: 450.1327.

2,3-(1,2-Bis(4-methylphenyl)propane-1,3-diyl)[3]ferrocenophane-1,3-diol (4b). By a procedure similar to that for **4a**, treatment of **1b** (190 mg, 0.4 mmol) with SmI_2 (1.8 mmol) gave **4b** (138 mg, 72%) as a mixture of two isomers (62:38).

4b-major: Orange solid, mp 220 °C (decompose); IR (KBr) 3543, 3485 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz) δ 2.23 (1 H, m), 2.27 (3 H, s), 2.30 (3 H, s), 2.53 (1 H, dd, J = 12.1, 14.2 Hz), 2.97 (1 H, d, J = 5.3 Hz, OH), 3.48 (1 H, m), 3.84 (1 H, dd, J = 9.8, 12.7 Hz), 3.90–4.80 (11 H, m), 6.77–7.24 (8 H, m); FAB-MS m/z 478 (M^+). HRMS Calcd for $\text{C}_{30}\text{H}_{30}\text{FeO}_2$: 478.1595. Found: 478.1595.

4b-minor: Orange solid, mp 176–178 °C; IR (KBr) 3423 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz) δ 2.18 (6 H, s), 2.38 (3 H, m), 2.72 (1 H, d, J = 8.8 Hz), 3.95–4.75 (12 H, m), 6.77–7.24 (8 H, m); ^{13}C NMR (CDCl_3 , 75 MHz) δ 20.8, 20.9, 29.7, 46.9, 47.3, 47.5, 65.5, 65.7, 67.1, 68.0, 68.4, 69.0, 69.1, 69.8, 70.6, 79.1, 90.2, 93.9, 128.2 (2 \times), 128.5 (2 \times), 128.6 (2 \times), 128.9 (2 \times), 134.8, 135.0, 138.1, 139.2. FAB-MS m/z 478 (M^+). HRMS. Calcd for $\text{C}_{30}\text{H}_{30}\text{FeO}_2$: 478.1595. Found: 478.1597.

2,3-(1,2-Bis(3-methoxyphenyl)propane-1,3-diyl)[3]ferrocenophane-1,3-diol (4c). By a procedure similar to that for **4a**, treatment of **1c** (202 mg, 0.4 mmol) with SmI_2 (1.8 mmol) gave **4c** (198 mg, 97%) as a mixture of two isomers (86:14).

4c-major: Orange solid, mp 89–91 °C; IR (KBr) 3395 cm^{-1} ; ^1H NMR (CDCl_3 , 300 MHz) δ 2.32 (1 H, dd, J = 9.7, 14.0 Hz), 2.43 (1 H, dd, J = 7.6, 14.0 Hz), 2.73 (1 H, dd, J = 2.1, 11.4

Hz), 3.55 (1 H, d, J = 5.6 Hz), 3.61–4.62 (18 H, m), 6.44–6.99 (8 H, m); ^{13}C NMR (CDCl₃, 50 MHz) δ 29.6, 46.4, 47.6, 47.8, 55.1, 65.5, 65.7, 67.1, 67.9, 68.4, 68.9, 69.7, 70.2, 89.9, 93.8, 111.1, 111.3, 114.8, 115.0, 121.0, 121.5, 128.4, 128.7, 142.8, 143.8, 158.9, 159.0; FAB-MS m/z 510 (M $^+$). HRMS Calcd for C₃₀H₃₀FeO₄: 510.1493. Found: 510.1496.

4c-minor: Orange solid, mp 86–88 °C; IR (KBr) 3422 cm $^{-1}$; ^1H NMR (CDCl₃, 300 MHz) δ 2.24 (1 H, dd, J = 5.0, 14.7 Hz), 2.53 (2 H, m), 3.04 (1 H, d, J = 5.3 Hz), 3.50 (1 H, m), 3.62–4.60 (17 H, m), 6.66–7.22 (8 H, m); ^{13}C NMR (CDCl₃, 75 MHz) δ 29.7, 48.9, 52.7, 53.7, 55.1 (2 \times), 65.0, 65.5, 65.9, 67.0, 67.7, 68.2, 68.9, 69.0, 69.7, 72.1, 91.0, 93.5, 111.3, 111.5, 113.7, 114.0, 120.1, 120.3, 129.1, 129.6 (2 \times), 143.6, 159.5, 159.7. FAB-MS m/z 510 (M $^+$); HRMS Calcd for C₃₀H₃₀FeO₄: 510.1493. Found: 510.1481.

2,3-[1,2-Bis(3-trifluoromethylphenyl)propane-1,3-diyl]-[3]ferrocenophane-1,3-diol (4d). By a procedure similar to that for **4a**, treatment of **1d** (233 mg, 0.4 mmol) with SmI₂ (1.8 mmol) gave **4d** (143 mg, 61%) as a mixture of two isomers (52:48).

4d (a mixture of two isomers (52:48)): Orange solid, mp 95–97 °C; IR (KBr) 3395 cm $^{-1}$; ^1H NMR (CDCl₃, 200 MHz) δ 2.57–2.02 (2 H, m), 3.01 (1 H, d, J = 8.9 Hz)/2.78 (1 H, d, J = 11.1 Hz), 4.90–3.02 (13 H, m), 7.20–6.94 (8 H, m); FAB-MS m/z 586 (M $^+$); HRMS Calcd for C₃₀H₂₄F₆FeO₂: 586.1030. Found: 586.1020.

2,3-[1,2-Bis(4-fluorophenyl)propane-1,3-diyl]-[3]ferrocenophane-1,3-diol (4e). By a procedure similar to that for **4a**, treatment of **1e** (193 mg, 0.4 mmol) with SmI₂ (1.8 mmol) gave **4e** (93 mg, 47%).

4e: Orange solid, mp 197–199 °C; IR (KBr) 3405 cm $^{-1}$; ^1H NMR (CDCl₃, 200 MHz) δ 2.26 (1 H, dd, J = 10.1, 13.9 Hz), 2.42 (1 H, m), 2.68 (1 H, dd, J = 2.1, 11.5 Hz), 3.28 (1 H, d J = 5.5 Hz), .3.89-5.13 (12 H, m), 6.67-6.92 (8 H, m); FAB-MS m/z 486(M $^+$); HRMS Calcd for C₂₈H₂₄F₂FeO₂: 486.1094. Found: 486.1106.

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