**Palladium-Catalyzed [2+2+2] Cocyclotrimerization of Benzyne
with Bicyclic Alkenes: An Efficient Route to Anellated
9,10-Dihydrophenanthrene Derivatives and Polyaromatic Compounds**

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Supporting Information

Table of Contents

Page S2	General Section
Page S2 – S7	¹ H and ¹³ C NMR Data
Page S8 – S31	¹ H NMR Spectra

General: All reactions were conducted under nitrogen atmosphere on a dual-manifold Schlenk line unless otherwise mentioned and in oven-dried glass wares. All solvents were dried according to known methods and distilled prior to use.¹ Starting materials substituted oxabenzonorbornadiene **1a**, **g-i** azabenzonorbornadiene² **1f**, and benzyne precursors³ **2a-d** were synthesized according to the literature procedures. Pd(PPh₃)₄,⁴ PdCl₂(PPh₃)₂,⁵ and Pd(dba)₂⁶ were prepared by reported procedures. Other reagents were commercially available and used as purchased.

References:

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(2R*,15S*)Hexacyclo[14.6.1.0^{2,15}.0^{3,8}.0^{9,14}.0^{17,22}]tricos-3(8),4,6,9(14),10,12,17,19,21-nonaene (3d): ¹H NMR (600 MHz, CDCl₃): δ 8.13 (d, *J* = 6.0 Hz, 2 H), 7.64 (d, *J* = 7.8 Hz, 2 H), 7.57 (d, *J* = 8.4 Hz, 2 H), 7.49 – 7.48(m, 4 H), 7.39 (d, *J* = 8.4 Hz, 2 H), 3.68 (s, 2 H), 3.44 (s, 2 H), 1.98 (d, *J* = 12.0 Hz, 1 H), 1.77 (d, *J* = 12.0 Hz, 1 H) ; ¹³C NMR (150 MHz, CDCl₃): δ 148.4, 137.1, 131.7, 130.3, 127.8, 126.5, 126.0, 122.4, 120.9, 56.8, 43.6, 43.5; HRMS calcd for C₂₃H₁₈ 294.1409, found 294.1412.

Diethyl

(2R*,15S*)pentacyclo[14.2.1.0^{2,15}.0^{3,8}.0^{9,14}]nonadeca-3(8),4,6,9(14),10,12,17-heptaene-17,18-dicarboxylate(3e): ¹H NMR (600 MHz, CDCl₃): δ 7.88 (d, *J* = 9.0 Hz, 2 H), 7.36 (d, *J* = 2 Hz, 1 H), 7.23 (m, 4 H), 4.31 (q, *J*₁ = 11.4 Hz, *J*₂ = 7.1 Hz, 4 H), 3.36 (d, *J* = 4.8 Hz, 4 H), 1.43 (d, *J* = 10.8 Hz, 1 H), 1.39 – 1.34 (m, 8H); ¹³C NMR (150 MHz, CDCl₃): δ 164.7, 144.5, 136.2, 131.8, 130.2, 127.9, 126.8, 122.5, 61.1, 58.4, 41.5, 41.2, 14.2; HRMS calcd for C₂₅H₂₄O₄ 388.1675, found 388.1686.

(2R*,21S*)-29-Oxaocyclo[20.6.1.0^{2,21}.0^{3,11}.0^{5,9}.0^{12,20}.0^{14,18}.0^{23,28}]nonacos-3(11),4,9,12(20),13,18,23(28),24,26-nonaene (3h): ¹H NMR (600 MHz, CDCl₃): δ 7.78 (s, 2 H), 7.41 (d, *J* = 7.8 Hz, 2 H), 7.25 – 7.22 (m, 4 H), 5.36 (s, 2 H), 3.35 (s, 2 H), 2.96 – 2.90 (m, 8 H), 2.12 – 2.07 (m, 4 H); ¹³C NMR (150 MHz, CDCl₃): δ 145.9, 143.8, 143.1, 132.9, 130.4, 126.8, 125.5, 119.1, 118.2, 90.4, 44.1, 32.7, 32.6, 25.5; HRMS calcd for C₂₈H₂₄O 376.1827, found 376.1831.

(2R*,15S*)-20,22,26-Trioxaheptacyclo[14.9.1.0^{2,15}.0^{3,8}.0^{9,14}.0^{17,25}.0^{19,23}]hexacos-3(8),4,6,9(14),10,12,17(25),18,23-nonaene (3i): ¹H NMR (600 MHz, CDCl₃): δ 7.92 (d, *J* = 8.4 Hz, 2 H), 7.37 (d, *J* = 9.0 Hz, 2 H), 7.31 – 7.27 (m, 4 H), 6.93 (s, 2 H), 5.98 (d, *J* = 18.0 Hz, 2 H), 5.29 (s, 2 H), 3.34 (s, 2 H); ¹³C NMR (150 MHz, CDCl₃): δ 146.7, 139.4, 135.0, 131.7, 129.9, 128.0, 127.0, 122.7, 101.4, 90.3, 44.2; HRMS calcd for C₂₃H₁₆O₃ 340.1099, found 340.1102.

(2R*,15S*)-5,6,11,12-Tetramethyl-20,22,26-trioxaheptacyclo[14.9.1.0^{2,15}.0^{3,8}.0^{9,14}.0^{17,25}.0^{19,23}]hexacos-3(8),4,6,9(14),10,12,17(25),18,23-nonaene (3j): ¹H NMR (400MHz, CDCl₃): δ 7.64 (s, 2 H), 7.10 (s, 2 H), 6.93 (s, 2 H), 5.97 (d, *J* = 10.4 Hz, 2 H), 5.25 (s, 2 H), 3.22 (s, 2 H), 2.29 (s, 12 H); ¹³C NMR (100 MHz, CDCl₃): δ 146.5, 139.5, 135.9, 134.9, 132.2, 130.9, 129.5, 123.5, 101.4, 90.2, 43.7, 19.6, 19.4; HRMS calcd for C₂₇H₂₄O₃ 396.1725, found 396.1733.

(2R*,21S*)-26,28,32-Trioxanonacyclo[20.9.1.0^{2,21}.0^{3,11}.0^{5,9}.0^{12,20}.0^{14,18}.0^{23,31}.0^{25,29}]dotriaconta-3(11),4,9,12(20),13,18,23(31),24,29-nonaene (3k): ¹H NMR (400MHz, CDCl₃): δ 7.77 (s, 2 H), 7.21 (s, 2 H), 6.92 (s, 2 H), 5.96 (d, *J* = 8.0 Hz, 2 H), 5.26 (s, 2 H), 3.28 (s, 2 H), 2.94 - 2.88 (m, 8 H), 2.13 – 2.03 (m, 4 H); ¹³C NMR (100 MHz, CDCl₃): δ 146.5, 143.7, 143.1, 139.6, 132.9, 130.0, 125.4, 118.2, 101.4, 101.1, 90.5, 44.4, 32.7, 32.5, 25.5; HRMS calcd for C₂₉H₂₄O₃ 420.1725, found 420.1720.

(2R*,15S*)-19,20-Dimethoxy-23-oxahexacyclo[14.6.1.0^{2,15}.0^{3,8}.0^{9,14}.0^{17,22}]tricos-3(8),4,6,9(14),10,12,17(22),18,20-nonaene (3l): ¹H NMR (600 MHz, CDCl₃): δ 7.93 (d, *J* = 9.0 Hz, 2 H), 7.41 (d, *J* = 8.4 Hz, 2 H), 7.31 – 7.27 (m, 4 H), 7.03 (s, 2 H), 5.33 (s, 2 H), 3.93 (s, 6 H), 3.35 (s, 2 H); ¹³C NMR (150 MHz, CDCl₃): δ 148.2, 137.9, 135.2, 131.8,

129.9, 128.0, 126.9, 122.7, 103.9, 90.5, 56.3, 44.4; HRMS calcd for C₂₄H₂₀O₃ 356.1412, found 356.1416.

(2R*,15S*)-19,20-Dimethoxy-5,6,11,12-tetramethyl-23-oxahexacyclo[14.6.1.0^{2,15}.0^{3,8}.0^{9,14}.0^{17,22}]tricoso-3(8),4,6,9(14),10,12,17(22),18,20-nonaene (3m): ¹H NMR (400 MHz, CDCl₃): δ 7.64 (s, 2 H), 7.13 (s, 2 H), 7.04 (s, 2 H), 5.29 (s, 2 H), 3.92 (s, 6 H), 3.24 (s, 2 H), 2.29 (s, 12 H); ¹³C NMR (100 MHz, CDCl₃): δ 148.2, 138.2, 135.9, 134.9, 132.4, 130.9, 129.6, 123.5, 104.0, 90.4, 56.3, 43.9, 19.7; HRMS calcd for C₂₈H₂₈O₃ 412.2038, found 412.2031.

(2R*,15S*)-19-Methyl-23-oxahexacyclo[14.6.1.0^{2,15}.0^{3,8}.0^{9,14}.0^{17,22}]tricoso-3(8),4,6,9(14),10,12,17(22),18,20-nonaene (3n): ¹H NMR (600 MHz, CDCl₃): δ 7.92 (d, *J* = 7.8 Hz, 2 H), 7.40 (d, *J* = 5.4 Hz, 2 H), 7.30 – 7.25 (m, 6 H), 7.05 (s, 1 H), 5.34 (d, *J* = 11.4 Hz, 2 H), 3.39 (s, 2 H), 2.39 (s, 3 H); ¹³C NMR (150 MHz, CDCl₃): δ 146.0, 142.9, 136.8, 135.1, 131.7, 130.0, 128.0, 127.3, 126.9, 122.7, 120.0, 118.9, 90.2, 44.1, 21.5; HRMS calcd for C₂₃H₁₈O 310.1358, found 310.1365.

(2R*,15S*)-5,6,11,12,19-Pentamethyl-23-oxahexacyclo[14.6.1.0^{2,15}.0^{3,8}.0^{9,14}.0^{17,22}]tricoso-3(8),4,6,9(14),10,12,17(22),18,20-nonaene (3o): ¹H NMR (400 MHz, CDCl₃): δ 7.66 (s, 1 H), 7.31 (d, *J* = 8.0 Hz, 1 H), 7.26 (s, 1 H), 7.14 (s, 2 H), 7.06 (d, *J* = 8.0 Hz, 1 H), 5.32 (d, *J* = 8.0 Hz, 2 H), 3.29 (s, 2 H), 2.40 (s, 3 H), 2.30 (s, 12 H); ¹³C NMR (100 MHz, CDCl₃): δ 146.3, 143.1, 139.6, 135.9, 134.9, 132.3, 130.9, 129.6, 127.2, 123.5, 119.9, 118.8, 90.2, 90.0, 43.7, 21.4, 19.7, 19.4; HRMS calcd for C₂₇H₂₆O 366.1984, found 366.1996.

Benzo[*b*]triphenylene (8a): colorless needles, mp: 204-205 °C, ¹H NMR (400 MHz, *d*₆-DMSO): δ 9.36 (s, 2 H), 8.93 (d, *J* = 8.0 Hz, 2 H), 8.71 (d, *J* = 8.0 Hz, 2 H), 8.21 – 8.18 (m, 2 H), 7.71 – 7.68 (m, 4 H), 7.62 – 7.59 (m, 2 H); ¹³C NMR (100 MHz, *d*₆-DMSO): δ 132.0, 129.5, 129.4, 128.0, 127.8, 127.7, 126.3, 123.9, 123.7, 122.3; HRMS calcd for C₂₂H₁₄ 278.1096, found 278.1094.

Dibenzo[5,6:7,8]anthra[2,3-*d*][1,3]dioxole (8b): white solid, mp: 262-263 °C, ¹H NMR (600 MHz, CDCl₃): δ 8.85 (s, 2 H), 8.69 (d, *J* = 4.0 Hz, 2 H), 8.57 (d, *J* = 5.0 Hz, 2 H), 7.63 – 7.59 (m, 4 H), 7.28 (s, 2 H), 6.07 (s, 2 H); ¹³C NMR (150 MHz, CDCl₃): δ 148.2, 130.0, 129.7, 127.3, 127.2, 127.1, 123.4, 123.3, 120.9, 103.3, 101.1; HRMS calcd for C₂₃H₁₄O₂ 322.0994, found 322.0996.

11,12-Dimethoxy-2,3,6,7-tetramethylbenzo[*b*]triphenylene (8e): white solid, mp: 277-278 °C ¹H NMR (600 MHz, CDCl₃): δ 8.77 (s, 2 H), 8.37 (s, 2 H), 8.20 (s, 2 H) 7.48 (s, 2 H), 4.03 (s, 6 H), 2.50 (d, *J* = 9.6 Hz, 12 H); ¹³C NMR (150 MHz, CDCl₃): δ 150.2, 135.3, 135.0, 128.5, 128.3, 128.1, 127.2, 124.2, 124.0, 119.8, 105.9, 20.4, 14.4; HRMS calcd for C₂₈H₂₆O₂ 394.1933, found 394.1932.

2,3,6,7,11-Pentamethylbenzo[*b*]triphenylene (8f): colorless needles, mp: 236-237 °C, ¹H NMR (600 MHz, *d*₆-DMSO): δ 9.17 (s, 1 H), 9.12 (s, 1 H), 8.61 (s, 2 H) 8.42 (s, 2 H), 8.07 (d, *J* = 8.0 Hz, 1 H), 7.92 (s, 1 H), 7.42 (d, *J* = 8.0 Hz, 1 H), 2.55 (s, 3 H), 2.47 (s, 12 H); ¹³C NMR (150 MHz, *d*₆-DMSO): δ 136.5, 136.4, 135.8, 135.1, 130.1, 128.4, 127.8, 127.5, 127.3, 127.2, 127.1, 126.3, 124.3, 124.2, 123.8, 121.4, 120.7; HRMS calcd for C₂₇H₂₄ 348.1878, found 348.1879.

Diethyl 11-oxatricyclo[6.2.1.0^{2,7}]undeca-2,4,6,9-tetraene-9,10-dicarboxylate (9): ¹H NMR (400 MHz, CDCl₃): δ 7.40 (dd, *J*₁ = 5.2 Hz, *J*₂ = 2.8 Hz, 2 H) 7.06 (dd, *J*₁ = 5.2 Hz, *J*₂ = 2.8 Hz, 2 H), 5.94 (s, 2 H), 4.23 (q, *J*₁ = 14.0 Hz, *J*₂ = 7.2 Hz, 4 H), 1.29 (t, *J* = 7.2 Hz, 6 H); ¹³C NMR (100 MHz, CDCl₃): δ 162.5, 150.9, 146.3, 126.0, 121.4, 84.8, 61.4, 14.0; HRMS calcd for C₁₆H₁₆O₅ 288.0998, found 288.1004.

2,3,6,7-Tetramethyl-9,10-dihydrophenanthrene (10): colorless needles, mp: 186-187 °C, ¹H NMR (400 MHz, CDCl₃): δ 8.43 (s, 2 H), 7.63 (s, 2 H), 7.60 (s, 2 H), 2.55 (s, 6 H), 2.48 (s, 6 H); ¹³C NMR (100 MHz, CDCl₃): δ 135.4, 135.1, 130.3, 128.5, 128.3, 125.4, 122.6, 20.4, 19.8; HRMS calcd for C₁₈H₁₈ 234.1409, found 234.1405.

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