Practical Access to Spiroacetal Enol Ethers via Nucleophilic
Dearomatization of 2-Furylmethylene palladium Halides Generated
by Pd-Catalyzed Coupling of Furfural Tosylhydrazones with Aryl
Halides

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**General Experimental details**

IR spectra were recorded with FT-IR as a thin film or using KBr pellets and are expressed in cm\(^{-1}\). \(^1\)H (400 MHz) and \(^{13}\)C (100 MHz) NMR spectra were recorded using CDCl\(_3\) as a solvent. Chemical shifts are reported in ppm downfield to tetramethylsilane. Coupling constants are reported and expressed in Hz; splitting patterns are designated as s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), dd (double doublet), dt (double triplet), dq (double quartet). Infrared (IR) spectra were obtained on a Bruker Vector 22 spectrometer. Mass spectra were obtained from high resolution ESI mass spectrometer. All reactions were carried out using freshly distilled and dry solvents. Column chromatography was performed over silica gel (100-200 Mesh) using petroleum ether and ethyl acetate as the eluent.
Preparation of tosylhydrazone (4a)

To a stirred suspension of \( p \)-tosylhydrazide (1.86 g, 10 mmol) in methanol (20 mL) was added 5-(3-hydroxypropyl)furan-2-carbaldehyde (1.54 g, 10 mmol). The mixture was stirred for 2 h at room temperature, and then the solvent was removed under reduced pressure. The crude products could be obtained as precipitates. The precipitates were washed by petroleum ether then removed in vacuo to afford the pure product 4a. Yellow solid (3.09 g, 96%), m.p. 129–130 °C; IR (KBr) \( \nu \): 3438, 3047, 2386, 1438, 1162, 1105, 619, 550 cm\(^{-1}\); \(^1\)H NMR (400 MHz, DMSO) \( \delta \): 11.30 (s, 1H), 7.78–7.67 (m, 3H), 7.40 (d, \( J = 8.1 \) Hz, 2H), 6.70 (d, \( J = 3.3 \) Hz, 1H), 6.21 (d, \( J = 3.2 \) Hz, 1H), 3.42 (t, \( J = 6.3 \) Hz, 2H), 2.65 (t, \( J = 7.6 \) Hz, 2H), 2.37 (s, 3H), 1.76–1.62 (m, 2H); \(^{13}\)C NMR (101 MHz, DMSO) \( \delta \): 158.53, 147.02, 143.38, 137.11, 136.21, 129.65, 127.09, 115.22, 107.60, 59.76, 30.64, 24.07, 20.96; HRMS (ESI) m/z calcd for C\(_{15}\)H\(_{18}\)N\(_2\)NaO\(_4\)S: [M + Na]\(^+\) 345.0885, Found: 345.0882;

General procedure for the preparation of 7

Bromobenzene (0.36 mmol, 56 mg) was added to a mixture of \( \text{Pd}_2(\text{dba})_3 \) (5.0 mol%, 14 mg), tricyclohexyolphosphine (10 mol%, 9 mg), \( \text{LiO}t\text{Bu} \) (1.05 mmol, 84 mg), 4a (0.3 mmol, 97 mg), and toluene (3 mL) in a Schlenk tube under nitrogen. The mixture was stirred at 90 °C for 2 h, cooled to room temperature, and filtered through a short column of silica gel (ethyl acetate). The solvent was removed in vacuo, and the residue was purified by flash column chromatography on silica gel (ethyl acetate/petroleum ether= 1:15) to give 7.
Characterization of 7

**(Z)-2-benzylidene-1,6-dioxaspiro[4.4]non-3-ene (7a)**

Yellow oil (79 mg, 74%), IR (film) 2950, 1693, 1449, 1359, 1091, 944, 816, 754, 697 cm\(^{-1}\);
\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.54 (d, \(J = 7.3\) Hz, 2H), 7.21 (t, \(J = 7.7\) Hz, 2H), 7.09-7.02 (m, 1H), 6.26 (d, \(J = 5.6\) Hz, 1H), 5.96 (d, \(J = 5.6\) Hz, 1H), 5.32 (s, 1H), 4.21-3.91 (m, 2H), 2.30-2.14 (m, 2H), 2.04-1.97 (m, 2H); \(^13\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 156.0, 136.2, 131.0, 129.9, 128.3, 128.2, 125.7, 121.1, 101.3, 69.1, 36.0, 24.6; HRMS (ESI) m/z calcd for C\(_{14}\)H\(_{14}\)NaO\(_2\): [M + Na]\(^+\) 237.0891, Found: 237.0886.

**(Z)-2-(2-methylbenzylidene)-1,6-dioxaspiro[4.4]non-3-ene (7b)**

Yellow oil (83 mg, 73%), IR (KBr) 2952, 2359, 1644, 1456, 1129, 944, 818, 750, 619 cm\(^{-1}\);
\(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.20-7.02 (m, 4H), 6.37 (d, \(J = 5.5\) Hz, 1H), 6.02 (d, \(J = 5.6\) Hz, 1H), 5.50 (s, 1H), 4.32-3.90 (m, 3H), 2.31 (s, 3H), 2.27-2.19 (m, 2H), 2.10-2.01 (m, 2H); \(^13\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 156.0, 135.0, 134.5, 134.1, 130.9, 130.1, 129.9, 128.5, 125.9, 125.8, 121.0, 98.0, 69.1, 49.4, 36.0, 24.7, 20.3; HRMS (ESI) m/z calcd for C\(_{15}\)H\(_{16}\)NaO\(_2\): [M + Na]\(^+\) 251.1048, Found: 251.1043.

**(Z)-2-(4-methylbenzylidene)-1,6-dioxaspiro[4.4]non-3-ene (7c)**

Yellow oil (86 mg, 76%), IR (film) 2925, 1640, 1446, 1106, 816 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.55 (d, \(J = 7.5\) Hz, 2H), 7.14 (d, \(J = 7.1\) Hz, 2H), 6.37 (d, \(J = 6.4\) Hz, 1H), 6.05 (d, \(J = 6.0\) Hz, 1H), 5.40 (s, 1H), 4.31-4.00 (m, 2H), 2.35 (s, 1H), 2.30-2.20 (m, 2H), 2.14-2.06(m,
2H); $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 155.4, 135.4, 133.3, 130.4, 129.9, 129.0, 128.1, 120.9, 101.3, 69.0, 36.0, 24.6, 21.2; HRMS (ESI) m/z calcd for C$_{15}$H$_{16}$NaO$_2$: [M + Na]$^+$ 251.1048, Found: 251.1043.

(Z)-2-(3,5-dimethylbenzylidene)-1,6-dioxaspiro[4.4]non-3-ene (7d)

Yellow oil (91 mg, 75 %), IR (film) 2978, 2874, 1518, 1488, 1372, 1280, 1158, 1116, 1063, 978, 876 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.92 (d, $J$ = 8.0 Hz, 1H), 6.96 (d, $J$ = 13.6 Hz, 2H), 6.37 (d, $J$ = 5.5 Hz, 1H), 6.01 (d, $J$ = 5.6 Hz, 1H), 5.48 (s, 1H), 4.23-3.99 (m, 2H), 2.30-2.22 (m, 12H), 2.12-2.04 (m, 3H); $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 155.46, 130.72, 130.30, 130.14, 128.45, 120.85, 100.68, 98.05, 69.04, 35.94, 24.63, 20.19; HRMS (ESI) m/z calcd for C$_{16}$H$_{18}$O: [M + H]$^+$ 243.1385, Found: 243.1390.

(Z)-2-(3-methoxybenzylidene)-1,6-dioxaspiro[4.4]non-3-ene (7e)

Yellow oil (85 mg, 70%), IR (film) 2942, 1648, 1487, 1266, 1093, 974, 874, 775, 688 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.36-7.18 (m, 3H), 6.75-6.69 (m, 1H), 6.38 (d, $J$ = 5.6 Hz, 1H), 6.08 (d, $J$ = 5.6 Hz, 1H), 5.42 (s, 1H), 4.31-4.01 (m, 2H), 3.85 (s, 3H), 2.44-0.59 (m, 2H); $^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 159.5, 156.3, 137.5, 131.1, 129.9, 129.1, 121.0, 113.3, 111.8, 101.2, 69.1, 55.0, 36.0, 24.6; HRMS (ESI) m/z calcd for C$_{15}$H$_{16}$NaO$_3$: [M + Na]$^+$ 267.0997, Found: 267.0991.

(Z)-2-(4-methoxybenzylidene)-1,6-dioxaspiro[4.4]non-3-ene (7f)

Yellow oil (94 mg, 77 %), IR (film) 2950, 1510, 1451, 1358, 1249, 1176, 1029, 840 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.59 (d, $J$ = 8.8 Hz, 2H), 6.88 (d, $J$ = 8.8 Hz, 2H), 6.36 (d, $J$ =
5.6 Hz, 1H), 6.01 (d, \( J = 5.5 \) Hz, 1H), 5.39 (s, 1H), 4.34-4.01 (m, 2H), 3.83 (s, 3H), 2.42-2.23 (m, 2H), 2.16-2.03 (m, 2H); \(^1\)H NMR (101 MHz, CDCl\(_3\)) \( \delta \) 157.7, 154.6, 133.8, 129.9, 129.8, 129.4, 129.1, 120.9, 114.0, 113.8, 109.9, 69.0, 55.2, 36.0, 24.7; HRMS (ESI) m/z calcd for C\(_{15}\)H\(_{16}\)NaO\(_3\): [M + Na]\(^+\) 267.0997, Found: 267.0992.

**(Z)-2-(2,4-dimethoxybenzylidene)-1,6-dioxaspiro[4.4]non-3-ene (7g)**

Yellow oil (113 mg, 83 %), IR (film) 2941, 1608, 1504, 1460, 1291, 1158, 1035, 832 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \( \delta \) 8.03 (d, \( J = 8.6 \) Hz, 1H), 6.51 (dd, \( J = 8.6, 2.4 \) Hz, 1H), 6.42 (d, \( J = 2.4 \) Hz, 1H), 6.36 (d, \( J = 5.6 \) Hz, 1H), 5.95 (d, \( J = 5.5 \) Hz, 1H), 5.75 (s, 1H), 4.26-3.97 (m, 2H), 3.81 (s, 6H), 2.35-2.17 (m, 2H), 2.12-2.02 (m, 2H); \(^1\)C NMR (101 MHz, CDCl\(_3\)) \( \delta \) 158.9, 157.1, 154.7, 130.2, 129.8, 129.3, 120.8, 118.1, 104.6, 98.1, 94.2, 69.0, 55.6, 55.3, 36.0, 24.7; HRMS (ESI) m/z calcd for C\(_{16}\)H\(_{18}\)NaO\(_4\): [M + Na]\(^+\) 297.1103, Found: 297.1097.

**(Z)-2-(4-fluorobenzylidene)-1,6-dioxaspiro[4.4]non-3-ene (7h)**

Yellow oil (74 mg, 64 %), IR (film) 2983, 1693, 1507, 1359, 1315, 1090, 944, 771, cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \( \delta \) 7.63-7.52 (m, 2H), 6.96 (d, \( J = 8.8 \) Hz, 2H), 6.33 (d, \( J = 5.6 \) Hz, 1H), 6.03 (d, \( J = 5.6 \) Hz, 1H), 5.36 (s, 1H), 4.27-3.99 (m, 2H), 2.36-2.20 (m, 2H), 2.13-2.06 (m, 2H); \(^1\)C NMR (101 MHz, CDCl\(_3\)) \( \delta \) 134.7, 132.4, 132.3, 130.9, 129.7, 129.7, 129.6, 129.5, 129.4, 125.3, 121.1, 115.2, 115.0, 100.2, 69.2, 36.0, 24.6; HRMS (ESI) m/z calcd for C\(_{14}\)H\(_{13}\)FNaO\(_2\): [M + Na]\(^+\) 255.0797, Found: 255.0792.

**(Z)-2-(4-nitrobenzylidene)-1,6-dioxaspiro[4.4]non-3-ene (7i)**
Yellow oil (63 mg, 49 %); IR (film), 2983, 1642, 1388, 1111, 993, 619 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 8.14 (d, J = 8.8 Hz, 2H), 7.71 (d, J = 8.9 Hz, 2H), 6.38 (d, J = 5.6 Hz, 1H), 6.22 (d, J = 5.6 Hz, 1H), 5.48 (s, 1H), 4.34-3.99 (m, 3H), 2.33-2.22 (m, 2H), 2.20-2.11 (m, 2H); ¹³C NMR (101 MHz, CDCl₃) δ 175.1, 159.3, 134.0, 129.4, 128.1, 127.9, 123.7, 121.9, 99.5, 69.6, 49.4, 30.6; HRMS (ESI) m/z calcd for C₁₄H₁₃NNaO₄: [M + Na]⁺ 282.0742, Found: 282.0737.

(Z)-2-(4-(trifluoromethyl)benzylidene)-1,6-dioxaspiro[4.4]non-3-ene (7j)

Yellow oil (78 mg, 56 %), IR (film) 2895, 1653, 1419, 1363, 1243, 1068, 945, 849, 759 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.69 (d, J = 8.2 Hz, 2H), 7.52 (d, J = 8.3 Hz, 2H), 6.35 (d, J = 5.6 Hz, 1H), 6.12 (d, J = 5.5 Hz, 1H), 5.42 (s, 1H), 4.32-3.97 (m, 2H), 2.40-2.20 (m, 2H), 2.16-2.01 (m, 2H); ¹³C NMR (101 MHz, CDCl₃) δ 157.7, 139.8, 132.6, 129.6, 128.0, 127.2 (q, J_C·F = 32 Hz), 125.8, 125.1 (q, J_C·F = 23 Hz), 123.1, 121.5, 99.9, 77.4, 77.1, 76.7, 69.4, 35.9, 24.6; HRMS (ESI) m/z calcd for C₁₅H₁₃F₃NaO₂: [M + Na]⁺ 305.0765, Found: 305.0760.

(Z)-4-(1,6-dioxaspiro[4.4]non-3-en-2-ylidenemethyl)benzaldehyde (7k)

Yellow syrup (60 mg, 50 %), IR (film) 2936, 1644, 1598, 1387, 1110, 848, 619 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 9.85 (s, 1H), 7.72 (d, J = 8.2 Hz, 2H), 7.66 (d, J = 8.3 Hz, 2H), 6.30 (d, J = 5.6 Hz, 1H), 6.10 (d, J = 5.6 Hz, 1H), 5.39 (s, 1H), 4.24-3.95 (m, 2H), 2.34-2.26 (m, 2H), 2.11-2.03 (m, 2H); ¹³C NMR (101 MHz, CDCl₃) δ 191.7, 158.6, 142.8, 133.3, 129.9, 129.6, 128.3, 128.1, 121.7, 100.4, 69.5, 35.9, 24.6; HRMS (ESI) m/z calcd for C₁₅H₁₄NaO₃: [M + Na]⁺ 265.0841, Found: 265.0835.
(Z)-2-(naphthalen-2-ylmethylene)-1,6-dioxaspiro[4.4]non-3-ene (7m)

Yellow oil (89 mg, 68 %), IR (film) 2952, 2892, 1650, 1591, 1440, 1089, 943, 705 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.99 (s, 1H), 7.82 (dd, \(J = 8.6, 1.6\) Hz, 1H), 7.78-7.71 (m, 3H), 7.43-7.34 (m, 2H), 6.36 (d, \(J = 5.6\) Hz, 1H), 6.05 (d, \(J = 5.6\) Hz, 1H), 5.54 (s, 1H), 4.34-3.97 (m, 2H), 2.42-2.21 (m, 2H), 2.16-2.02 (m, 2H); \(^13\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 156.5, 133.9, 133.8, 132.0, 131.2, 129.9, 127.9, 127.7, 127.6, 127.0, 126.6, 125.9, 125.2, 121.3, 101.5, 69.3, 36.0, 24.7; HRMS (ESI) m/z calcd for C\(_{18}\)H\(_{16}\)NaO\(_2\): [M + Na]\(^+\) 287.1048, Found: 287.1043.

(Z)-2-(thiophen-2-ylmethylene)-1,6-dioxaspiro[4.4]non-3-ene (7n)

Yellow oil (72 mg, 66 %), IR (film) 2945, 1642, 1440, 1356, 1056, 912, 669 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.17 (d, \(J = 5.1\) Hz, 1H), 7.05 (d, \(J = 3.4\) Hz, 1H), 6.96 (dd, \(J = 5.1, 3.6\) Hz, 1H), 6.35 (d, \(J = 5.6\) Hz, 1H), 6.06 (d, \(J = 5.6\) Hz, 1H), 5.75 (s, 1H), 4.37-3.94 (m, 2H), 2.47-2.18 (m, 2H), 2.16-1.98 (m, 2H); \(^13\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 154.4, 139.3, 131.4, 128.5, 126.7, 125.0, 124.2, 120.8, 95.3, 69.0, 36.0, 24.4; HRMS (ESI) m/z calcd for C\(_{12}\)H\(_{12}\)NaO\(_2\)S: [M + Na]\(^+\) 243.0456, Found: 243.0450.

(Z)-2-(1,6-dioxaspiro[4.4]non-3-en-2-ylidenemethyl)pyridine (7o)

Yellow syrup (47 mg, 44 %), IR (film) 2955, 1648, 1587, 1345, 1257, 1085, 936, 741 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.52-8.44 (m, 1H), 7.69 (d, \(J = 5.7\) Hz, 1H), 7.51 (td, \(J = 7.7, 1.9\) Hz, 1H), 7.07-6.92 (m, 2H), 6.26 (dd, \(J = 5.8, 1.7\) Hz, 1H), 5.93 (d, \(J = 1.2\) Hz, 1H), 4.42-3.83 (m, 2H), 2.32-2.17 (m, 2H), 2.12-2.03 (m, 2H); \(^13\)C NMR (101 MHz, CDCl\(_3\)) \(\delta\) 160.6, 156.0, 149.2, 136.0, 135.7, 127.6, 122.8, 119.8, 118.1, 101.1, 69.3, 35.5, 24.7 cm\(^{-1}\); HRMS (ESI) m/z
calcd for C_{13}H_{14}NO_2: [M + H]^+ 216.1025, Found: 216.1019

(Z)-2-(1,6-dioxaspiro[4.4]non-3-en-2-ylidenemethyl)quinolone (7p)

![Chemical Structure](image)

Yellow oil (77 mg, 58 %), IR (film) 3048, 2924, 1648, 1593, 1498, 1429, 1345, 1170, 977, 754 cm^{-1}; \(^1\)H NMR (400 MHz, CDCl\(_3\)) δ 7.96 (dd, \(J = 6.8, 4.8\) Hz, 3H), 7.69 (d, \(J = 8.2\) Hz, 1H), 7.63 (t, \(J = 7.6\) Hz, 1H), 7.40 (t, \(J = 7.4\) Hz, 1H), 7.17 (d, \(J = 8.5\) Hz, 1H), 6.36 (d, \(J = 5.8\) Hz, 1H), 6.08 (s, 1H), 4.23–3.97 (m, 2H), 2.36–2.19 (m, 2H), 2.22–2.03 (m, 2H); \(^1^3\)C NMR (101 MHz, CDCl\(_3\)) δ 162.0, 156.1, 148.3, 136.7, 135.7, 129.3, 128.8, 128.0, 127.4, 126.2, 125.2, 122.3, 118.4, 101.2, 69.5, 35.5, 24.7; HRMS (ESI) m/z calcd for C_{17}H_{16}NO_2: [M + H]^+ 266.1181, Found: 266.1176.

(Z)-2-(1,6-dioxaspiro[4.4]non-3-en-2-ylidenemethyl)pyrimidine (7q)

![Chemical Structure](image)

Yellow syrup (64 mg, 58 %), IR (KBr) 3043, 2916, 1648, 1568, 1425, 1124, 985, 767 cm^{-1}; \(^1\)H NMR (400 MHz, CDCl\(_3\)) δ 8.57 (d, \(J = 4.8\) Hz, 2H), 7.81 (d, \(J = 5.8\) Hz, 1H), 6.91 (t, \(J = 4.9\) Hz, 1H), 6.36 (dd, \(J = 5.8, 1.6\) Hz, 1H), 6.07 (s, 1H), 4.28–3.96 (m, 2H), 2.36–2.19 (m, 2H), 2.15–2.07 (m, 2H); \(^1^3\)C NMR (101 MHz, CDCl\(_3\)) δ 165.8, 164.7, 156.7, 137.4, 127.6, 118.6, 116.4, 101.5, 69.7, 35.4, 24.7; HRMS (ESI) m/z calcd for C_{12}H_{13}N_2O_2: [M + H]^+ 217.0977, Found: 217.0972.
NOESY of 7a
COSY of 7o
NOESY of 7o
$^1$H NMR of 4a

$^{13}$C NMR of 4a
$^1$H NMR of 7a (Z/E=4:1)

$^{13}$C NMR of 7a (Z/E=4:1)
$^1$H NMR of 7b (Z/E = 4.5:1)

$^{13}$C NMR of 7b (Z/E = 4.5:1)
$^1$H NMR of 7c (Z/E = 5:1)

$^{13}$C NMR of 7c (Z/E = 5:1)
$^1$H NMR of 7d

$^{13}$C NMR of 7d
$^1$H NMR of 7e (Z/E =3.3:1)

$^{13}$C NMR of 7e (Z/E =3.3:1)
$^1$H NMR of 7f (Z/E = 10:1)

$^{13}$C NMR of 7f (Z/E = 10:1)
$^{1}H$ NMR of $7g$

$^{13}C$ NMR of $7g$
$^1$H NMR of 7h (Z/E = 3:1)

$^{13}$C NMR of 7h (Z/E = 3:1)
$^1$H NMR of 7i (Z/E =4/1)

$^{13}$C NMR of 7i (Z/E =4/1)
$^1$H NMR of 7j

$^{13}$C NMR of 7j
$^1$H NMR of 7k (Z/E =5:1)

$^{13}$C NMR of 7k (Z/E =5:1)
\(^1\)H NMR of 7m (Z/E =10:1)

\(^{13}\)C NMR of 7m (Z/E =10:1)
$^1$H NMR of 7n

$^{13}$C NMR of 7n
$^1$H NMR of 7o

$^{13}$C NMR of 7o
$^1$H NMR of 7p

$^{13}$C NMR of 7p
$^1$H NMR of 7q

$^{13}$C NMR of 7q