A Neber approach for the synthesis of spiro-fused 2H-azirine-pyrazolones

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1. General Methods

Reagents were purchased from commercial sources and were used as received unless mentioned otherwise. Reactions were monitored by thinlayer chromatography (TLC), and column chromatography purifications were performed using 300-400 mesh silica gel.

\(^1\)H NMR and \(^{13}\)C NMR spectra (300 and 75 MHz, respectively) were recorded in CDCl\(_3\). \(^1\)H NMR chemical shifts are reported in parts per million (ppm) relative to tetramethylsilane (TMS) with the solvent resonance employed as the internal standard (CDCl\(_3\) at 7.26 ppm). Data are reported as follows: chemical shift, multiplicity (s=singlet, br s=broad singlet, d=doublet, t=triplet, q=quartet, m=multiplet), coupling constants (Hz) and integration. \(^{13}\)C NMR chemical shifts are reported in parts per million (ppm) relative to tetramethylsilane (TMS) with the solvent resonance as the internal standard (CDCl\(_3\) at 77.20 ppm).

2. General procedure for the synthesis of compounds 3

In an ordinary vial equipped with a magnetic stirring bar, compounds 1 (0.2 mmol, 1.0 equiv), compounds 2 (0.24 mmol, 1.2 equiv) and sodium carbonate (26 mg, 1.2 equiv) were dissolved in 4 mL of acetonitrile, and then the mixture was stirred at 25 °C for the indicated time. After completion of the reaction, as indicated by TLC, the products 3 were isolated by flash chromatography on silica gel (petroleum ether/ethyl acetate = 8/1).

7-methyl-2,5-diphenyl-1,5,6-triazaspiro[2.4]hepta-1,6-dien-4-one (3a). White solid; 48.8 mg, 88% yield; m.p. 129.7-131.1 °C; HPLC (AD-H, i-propanol/n-hexane = 10/90, flow rate = 1.0 mL/min, \(\lambda = 254\) nm) \(t\)\(_R\) = 16.4 min (major), 20.4 min (minor); \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\) 1.94 (s, 3H), 7.18-7.23 (m, 1H), 7.40-7.46 (m, 2H), 7.59-7.64 (m, 2H), 7.69-7.75 (m, 1H), 7.85-7.88 (m, 2H), 8.00 (d, \(J = 7.8\) Hz, 2H); \(^{13}\)C NMR (75 MHz, CDCl\(_3\)) \(\delta\) 13.1, 46.1, 118.4, 119.9, 125.0, 128.9, 129.8, 131.1, 135.2, 138.7, 157.5, 157.6, 168.9; HRMS (ESI-TOF) calcd. for C\(_{17}\)H\(_{14}\)N\(_3\)O [M + H]\(^+\) 276.1131; found: 276.1123.

7-methyl-5-phenyl-2-(m-tolyl)-1,5,6-triazaspiro[2.4]hepta-1,6-dien-4-one (3b). White solid; 49.1 mg, 84% yield; m.p. 116.2-117.9 °C; \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\) 1.95 (s, 3H), 2.45 (s, 3H), 7.17-7.23 (m, 1H), 7.40-7.46 (m, 2H), 7.49-7.52 (m, 2H), 7.65-7.71 (m, 2H), 8.00 (d, \(J = 8.1\) Hz, 2H); \(^{13}\)C NMR (75 MHz, CDCl\(_3\)) \(\delta\) 13.1, 21.1, 46.1, 118.4, 119.7, 125.0, 128.3, 128.9, 129.6, 131.6, 136.1, 138.7, 139.9, 157.5, 157.7, 169.0; HRMS (ESI-TOF) calcd. for C\(_{18}\)H\(_{16}\)N\(_3\)O [M + H]\(^+\) 290.1288; found: 290.1284.

7-methyl-5-phenyl-2-(p-tolyl)-1,5,6-triazaspiro[2.4]hepta-1,6-dien-4-one (3c). Yellow solid; 47.8 mg, 82% yield; m.p. 102.8-104.2 °C; \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\) 1.93 (s, 3H), 2.48 (s, 3H), 7.17-7.22 (m, 1H), 7.40-7.45 (m, 4H), 7.75 (d, \(J = 8.1\) Hz, 2H), 7.98-8.01 (m, 2H); \(^{13}\)C NMR (75 MHz, CDCl\(_3\)) \(\delta\) 13.1, 22.1, 46.0, 117.0, 118.4, 124.9, 128.8, 130.5, 131.2, 138.7, 146.7, 157.0, 157.8, 169.1; HRMS (ESI-TOF) calcd. for C\(_{18}\)H\(_{16}\)N\(_3\)O [M + H]\(^+\) 290.1288; found: 290.1277.

2-(3-methoxyphenyl)-7-methyl-5-phenyl-1,5,6-triazaspiro[2.4]hepta-1,6-dien-4-one (3d). Yellow solid; 57.4 mg, 94% yield; m.p. 80.4-81.0 °C; \(^1\)H NMR (300 MHz, CDCl\(_3\)) \(\delta\) 1.95 (s, 3H), 3.88 (s, 3H), 7.20-7.24 (m, 2H), 7.37-7.54 (m, 5H), 7.98-8.01 (m, 2H); \(^{13}\)C NMR (75 MHz, CDCl\(_3\)) \(\delta\) 13.1, 46.3, 55.7, 114.8, 118.4, 121.0, 121.9, 123.8, 125.0, 128.9, 130.8, 138.7, 157.6, 157.7, 160.3, 168.9; HRMS (ESI-TOF) calcd. for C\(_{18}\)H\(_{16}\)O\(_2\) [M + H]\(^+\) 306.1237; found: 306.1237.
2-(4-methoxyphenyl)-7-methyl-5-phenyl-1,5,6-triazaspiro[2.4]hepta-1,6-dien-4-one (3e). Yellow solid; 48.5 mg, 79% yield; m.p. 135.1-136.3 °C; 1H NMR (300 MHz, CDCl3) δ 1.93 (s, 3H), 3.90 (s, 3H), 7.08 (d, J = 8.7 Hz, 2H), 7.17-7.22 (m, 1H), 7.39-7.45 (m, 2H), 7.80 (d, J = 8.7 Hz, 2H), 7.98-8.01 (m, 2H); 13C NMR (75 MHz, CDCl3) δ 13.1, 46.0, 55.8, 111.8, 115.4, 118.4, 124.9, 128.8, 133.4, 138.8, 156.0, 158.0, 165.1, 169.3; HRMS (ESI-TOF) calcd. for C19H16N2O2 [M + H]+ 306.1237; found: 306.1231.

2-(4-(tert-butyl)phenyl)-7-methyl-5-phenyl-1,5,6-triazaspiro[2.4]hepta-1,6-dien-4-one (3f). White solid; 51.6 mg, 77% yield; m.p. 126.5-127.7 °C; 1H NMR (300 MHz, CDCl3) δ 1.36 (s, 9H), 1.94 (s, 3H), 7.20-7.25 (m, 1H), 7.40-7.46 (m, 2H), 7.70-7.82 (m, 2H), 7.98-8.02 (m, 2H); 13C NMR (75 MHz, CDCl3) δ 13.1, 30.9, 35.6, 46.0, 117.0, 118.4, 124.9, 126.9, 128.9, 131.1, 138.8, 157.0, 157.8, 159.6, 169.1; HRMS (ESI-TOF) calcd. for C23H22N2O [M + H]+ 332.1757; found: 332.1766.

2-(3-fluorophenyl)-7-methyl-5-phenyl-1,5,6-triazaspiro[2.4]hepta-1,6-dien-4-one (3g). Yellow solid; 51.0 mg, 87% yield; m.p. 132.7-134.5 °C; 1H NMR (300 MHz, CDCl3) δ 1.95 (s, 3H), 7.20-7.23 (m, 1H), 7.40-7.46 (m, 3H), 7.57-7.69 (m, 3H), 7.98 (d, J = 7.8 Hz, 2H); 13C NMR (75 MHz, CDCl3) δ 13.1, 46.3, 117.5 (d, J = 23.1 Hz, 1C), 118.4, 121.8 (d, J = 8.2 Hz, 1C), 122.4 (d, J = 21.1 Hz, 1C), 125.0, 126.9 (d, J = 3.3 Hz, 1C), 126.9, 128.8, 131.7 (d, J = 8.0 Hz, 1C), 138.5, 157.0, 157.3 (d, J = 3.3 Hz, 1C), 162.8 (d, J = 250.0 Hz, 1C), 168.4; HRMS (ESI-TOF) calcd. for C20H19FN3O [M + H]+ 294.1037; found: 294.1032.

2-(4-fluorophenyl)-7-methyl-5-phenyl-1,5,6-triazaspiro[2.4]hepta-1,6-dien-4-one (3h). White solid; 41.3 mg, 70% yield; m.p. 123.3-125.2 °C; 1H NMR (300 MHz, CDCl3) δ 1.94 (s, 3H), 7.18-7.23 (m, 1H), 7.29-7.35 (m, 2H), 7.40-7.46 (m, 2H), 7.88-7.93 (m, 2H), 7.96-8.05 (m, 2H); 13C NMR (75 MHz, CDCl3) δ 13.1, 46.1, 116.3 (d, J = 3.1 Hz, 1C), 117.5 (d, J = 22.7 Hz, 1C), 118.4, 125.1, 128.9, 133.7 (d, J = 9.8 Hz, 1C), 138.6, 156.6, 157.4, 166.7 (d, J = 258.1 Hz, 1C), 168.8; HRMS (ESI-TOF) calcd. for C19H15FN3O [M + H]+ 294.1037; found: 294.1029.

2-(4-chlorophenyl)-7-methyl-5-phenyl-1,5,6-triazaspiro[2.4]hepta-1,6-dien-4-one (3i). White solid; 34.5 mg, 55% yield; m.p. 124.9-126.5 °C; 1H NMR (300 MHz, CDCl3) δ 1.94 (s, 3H), 7.18-7.23 (m, 1H), 7.40-7.46 (m, 2H), 7.60 (d, J = 8.1 Hz, 2H), 7.82 (d, J = 8.1 Hz, 2H), 7.96-8.05 (m, 2H); 13C NMR (75 MHz, CDCl3) δ 13.2, 46.1, 118.4, 125.1, 128.9, 130.3, 132.2, 138.6, 142.0, 157.0, 157.3, 168.6; HRMS (ESI-TOF) calcd. for C16H11ClN3O [M + H]+ 310.0742; found: 310.0730.

2-(3-bromophenyl)-7-methyl-5-phenyl-1,5,6-triazaspiro[2.4]hepta-1,6-dien-4-one (3j). White solid; 42.3 mg, 59% yield; m.p. 108.4-109.9 °C; 1H NMR (300 MHz, CDCl3) δ 1.95 (s, 3H), 7.20-7.25 (m, 1H), 7.40-7.46 (m, 2H), 7.52 (d, J = 7.8 Hz, 1H), 7.81-7.85 (m, 2H), 7.97-8.01 (m, 3H); 13C NMR (75 MHz, CDCl3) δ 13.2, 46.3, 118.5, 121.9, 123.8, 125.1, 128.9, 129.5, 131.2, 133.6, 138.1, 138.6, 157.1, 157.2, 168.5; HRMS (ESI-TOF) calcd. for C17H13BrN3O [M + H]+ 354.0237; found: 354.0227.

2-(4-bromophenyl)-7-methyl-5-phenyl-1,5,6-triazaspiro[2.4]hepta-1,6-dien-4-one (3k). White solid; 55.9 mg, 78% yield; m.p. 142.9-144.1 °C; 1H NMR (300 MHz, CDCl3) δ 1.93 (s, 3H), 7.17-7.23 (m, 1H), 7.40-7.46
(m, 2H), 7.71-7.80 (m, 4H), 7.96-8.00 (m, 2H); $^{13}$C NMR (75 MHz, CDCl$_3$) δ 13.2, 46.1, 118.4, 118.8, 125.1, 128.9, 130.7, 132.2, 133.3, 138.6, 157.1, 157.2, 168.6; HRMS (ESI-TOF) calcd. for C$_{17}$H$_{13}$BrN$_2$O [M + H]$^+$ 354.0237; found: 354.0230.

7-methyl-2-(4-nitrophenyl)-5-phenyl-1,5,6-triazaspiro[2.4]hepta-1,6-dien-4-one (3l). Yellow solid; 49.6 mg, 77% yield; m.p. 141.5-142.3 °C; $^1$H NMR (300 MHz, CDCl$_3$) δ 1.95 (s, 3H), 7.19-7.24 (m, 1H), 7.40-7.46 (m, 2H), 7.94-7.98 (m, 2H), 8.09 (d, $J$ = 8.7 Hz, 2H), 8.45 (d, $J$ = 8.7 Hz, 2H); $^{13}$C NMR (75 MHz, CDCl$_3$) δ 13.2, 46.5, 118.4, 124.8, 125.3, 125.5, 128.9, 131.9, 138.4, 151.4, 156.5, 157.6, 168.0; HRMS (ESI-TOF) calcd. for C$_{17}$H$_{13}$N$_2$O [M + H]$^+$ 321.0972.

2-(3-fluoromethylphenyl)-7-methyl-5-phenyl-1,5,6-triazaspiro[2.4]hepta-1,6-dien-4-one (3m). White solid; 46.8 mg, 76% yield; m.p. 128.8-130.4 °C; $^1$H NMR (300 MHz, CDCl$_3$) δ 1.94 (s, 3H), 2.40 (d, $J$ = 9.2 Hz, 3H), 7.18-7.23 (m, 1H), 7.40-7.47 (m, 3H), 7.52-7.57 (m, 2H), 7.97-8.00 (m, 2H); $^{13}$C NMR (75 MHz, CDCl$_3$) δ 13.1, 15.2 (d, $J$ = 3.5 Hz, 1C), 46.2, 117.1 (d, $J$ = 24.1 Hz, 1C), 118.4, 119.0 (d, $J$ = 8.3 Hz, 1C), 125.0, 126.8 (d, $J$ = 3.2 Hz, 1C), 128.8, 132.9 (d, $J$ = 5.2 Hz, 1C), 133.8 (d, $J$ = 17.1 Hz, 1C), 138.6, 156.8 (d, $J$ = 3.1 Hz, 1C), 157.3, 161.4 (d, $J$ = 248.2 Hz, 1C), 168.6; HRMS (ESI-TOF) calcd. for C$_{18}$H$_{13}$N$_2$O [M + H]$^+$ 308.1194; found: 308.1191.

2-(furan-2-yl)-7-methyl-5-phenyl-1,5,6-triazaspiro[2.4]hepta-1,6-dien-4-one (3n). Yellow solid; 46.9 mg, 78% yield; m.p. 139.2-140.6 °C; $^1$H NMR (300 MHz, CDCl$_3$) δ 1.95 (s, 3H), 6.74 (dd, $J$ = 1.7 Hz, 3.6 Hz, 1H), 7.17-7.23 (m, 1H), 7.36-7.37 (m, 1H), 7.39-7.45 (m, 2H), 7.90-7.91 (m, 1H), 7.95-7.99 (m, 2H); $^{13}$C NMR (75 MHz, CDCl$_3$) δ 13.1, 45.5, 113.6, 118.5, 124.1, 125.1, 128.9, 136.5, 138.6, 147.2, 150.6, 157.2, 168.3; HRMS (ESI-TOF) calcd. for C$_{16}$H$_{12}$N$_2$O [M + H]$^+$ 266.0924; found: 266.0924.

7-methyl-2-(napthalen-2-yl)-5-phenyl-1,5,6-triazaspiro[2.4]hepta-1,6-dien-4-one (3o). White solid; 47.4 mg, 72% yield; m.p. 143.4-144.2 °C; $^1$H NMR (300 MHz, CDCl$_3$) δ 1.98 (s, 3H), 7.19-7.25 (m, 1H), 7.42-7.48 (m, 2H), 7.60-7.65 (m, 1H), 7.67-7.73 (m, 1H), 7.93-8.00 (m, 3H), 8.03-8.08 (m, 3H), 8.25 (s, 1H); $^{13}$C NMR (75 MHz, CDCl$_3$) δ 13.2, 46.3, 117.1, 118.4, 124.8, 125.0, 127.8, 128.2, 128.9, 129.3, 129.9, 130.0, 132.5, 134.2, 136.2, 138.8, 157.6, 157.8, 169.0; HRMS (ESI-TOF) calcd. for C$_{21}$H$_{13}$N$_2$O [M + H]$^+$ 326.1288; found: 326.1275.

2-benzyl-7-methyl-5-phenyl-1,5,6-triazaspiro[2.4]hepta-1,6-dien-4-one (3p). Yellow solid; 53.6 mg, 92% yield; m.p. 79.8-81.4 °C; $^1$H NMR (300 MHz, CDCl$_3$) δ 1.56 (s, 3H), 4.20 (d, $J$ = 16.6 Hz, 1H), 4.40 (d, $J$ = 16.6 Hz, 1H), 7.16-7.22 (m, 1H), 7.31-7.44 (m, 7H), 7.91-7.94 (m, 2H); $^{13}$C NMR (75 MHz, CDCl$_3$) δ 12.7, 33.1, 45.8, 118.4, 125.0, 128.5, 128.8, 129.1, 129.4, 130.0, 138.6, 157.3, 160.6, 169.0; HRMS (ESI-TOF) calcd. for C$_{19}$H$_{14}$N$_2$O [M + H]$^+$ 290.1288; found: 290.1277.

7-methyl-2-phenethyl-5-phenyl-1,5,6-triazaspiro[2.4]hepta-1,6-dien-4-one (3q). White solid; 56.8 mg, 93% yield; m.p. 104.9-106.2 °C; $^1$H NMR (300 MHz, CDCl$_3$) δ 1.68 (s, 3H), 3.07-3.22 (m, 2H), 3.28-3.34 (m, 2H), 3.716-7.30 (m, 6H), 7.38-7.44 (m, 2H), 7.89-7.93 (m, 2H); $^{13}$C NMR (75 MHz, CDCl$_3$) δ 12.9, 28.2, 30.2, 45.6, 118.5, 125.0, 127.1, 128.2, 128.8, 128.9, 138.1, 138.6, 157.4, 161.3, 169.2; HRMS (ESI-TOF) calcd. for C$_{19}$H$_{14}$N$_2$O [M + H]$^+$ 304.1444; found: 304.1437.
(E)-7-methyl-5-phenyl-2-styryl-1,5,6-triazaspiro[2.4]hepta-1,6-dien-4-one (3r). Yellow solid; 54.3 mg, 90% yield; m.p. 79.7-81.6 °C; 1H NMR (300 MHz, CDCl₃) δ 1.57 (s, 3H), 4.20 (d, J = 16.5 Hz, 1H), 4.39 (d, J = 16.5 Hz, 1H), 7.18-7.26 (m, 1H), 7.31-7.43 (m, 7H), 7.91-7.94 (m, 2H); 13C NMR (75 MHz, CDCl₃) δ 12.7, 33.1, 45.8, 118.4, 125.0, 128.5, 128.8, 129.1, 129.4, 130.0, 138.6, 157.3, 160.6, 169.0; HRMS (ESI-TOF) calcd. for C₁₉H₁₃N₅O [M + H]+ 301.1215; found: 301.1384.

7-methyl-2-(naphthalen-2-ylmethyl)-5-phenyl-1,5,6-triazaspiro[2.4]hepta-1,6-dien-4-one (3s). Yellow solid; 51.4 mg, 75% yield; m.p. 124.3-126.1 °C; 1H NMR (300 MHz, CDCl₃) δ 1.56 (s, 3H), 4.37 (d, J = 16.8 Hz, 1H), 4.55 (d, J = 16.8 Hz, 1H), 7.16-7.22 (m, 1H), 7.38-7.44 (m, 3H), 7.51-7.56 (m, 2H), 7.81-7.90 (m, 4H), 7.91-7.95 (m, 2H); 13C NMR (75 MHz, CDCl₃) δ 12.8, 33.3, 45.9, 118.4, 125.0, 126.3, 126.7, 126.9, 127.3, 127.7, 127.8, 128.4, 128.8, 129.4, 132.8, 133.4, 138.6, 157.3, 160.7, 169.1; HRMS (ESI-TOF) calcd. for C₂₂H₁₈N₅O [M + H]+ 340.1444; found: 340.1434.

2,7-dimethyl-5-phenyl-1,5,6-triazaspiro[2.4]hepta-1,6-dien-4-one (3t). Yellow oil; 38.7 mg, 90% yield; 1H NMR (300 MHz, CDCl₃) δ 1.88 (s, 3H), 2.63 (s, 3H), 7.13-7.18 (m, 1H), 7.34-7.40 (m, 2H), 7.87-7.91 (m, 2H); 13C NMR (75 MHz, CDCl₃) δ 12.4, 13.1, 45.3, 118.4, 124.9, 128.8, 138.5, 157.4, 158.6, 169.2; HRMS (ESI-TOF) calcd. for C₁₇H₁₂N₅O [M + H]+ 214.0975; found: 214.0968.

2,5,7-triphenyl-1,5,6-triazaspiro[2.4]hepta-1,6-dien-4-one (3u). Yellow solid; 43.5 mg, 64% yield; m.p. 155.4-157.2 °C; 1H NMR (300 MHz, CDCl₃) δ 7.22-7.28 (m, 1H), 7.29-7.35 (m, 3H), 7.45-7.51 (m, 2H), 7.59-7.64 (m, 4H), 7.69-7.75 (m, 1H), 7.91-7.94 (m, 2H), 8.11-8.15 (m, 2H); 13C NMR (75 MHz, CDCl₃) δ 45.1, 118.7, 120.0, 125.3, 126.2, 128.8, 128.9, 129.7, 129.8, 130.3, 131.3, 135.2, 138.7, 154.9, 158.0, 169.3; HRMS (ESI-TOF) calcd. for C₂₅H₂₀N₅O [M + H]+ 338.1288; found: 338.1301.

7-methyl-2-phenyl-5-(p-tolyl)-1,5,6-triazaspiro[2.4]hepta-1,6-dien-4-one (3v). Yellow solid; 46.4 mg, 80% yield; m.p. 157.1-158.9 °C; 1H NMR (300 MHz, CDCl₃) δ 1.94 (s, 3H), 2.36 (s, 3H), 7.21-7.26 (m, 2H), 7.58-7.64 (m, 2H), 7.69-7.72 (m, 1H), 7.84-7.89 (m, 4H); 13C NMR (75 MHz, CDCl₃) δ 13.1, 20.9, 46.1, 118.5, 120.0, 129.4, 129.7, 131.1, 134.6, 135.1, 136.3, 157.4, 157.7, 168.7; HRMS (ESI-TOF) calcd. for C₂₃H₁₉N₅O [M + H]+ 300.1288; found: 300.1285.

5-(4-chlorophenyl)-7-methyl-2-phenyl-1,5,6-triazaspiro[2.4]hepta-1,6-dien-4-one (3w). Yellow solid; 48.9 mg, 79% yield; m.p. 166.7-168.6 °C; 1H NMR (300 MHz, CDCl₃) δ 1.94 (s, 3H), 7.36-7.40 (m, 2H), 7.59-7.65 (m, 2H), 7.70-7.73 (m, 1H), 7.85-7.89 (m, 2H), 7.95-7.99 (m, 2H); 13C NMR (75 MHz, CDCl₃) δ 13.2, 46.1, 119.5, 119.8, 128.9, 129.8, 130.0, 131.2, 135.3, 137.3, 157.4, 157.9, 168.9; HRMS (ESI-TOF) calcd. for C₁₇H₁₃ClN₅O [M + H]+ 310.0742; found: 310.0730.
3. The copies of $^1$H NMR, $^{13}$C NMR spectra for compounds 3

$^1$H NMR, $^{13}$C NMR spectra of 3a
$^1$H NMR, $^{13}$C NMR spectra of 3b
$^1$H NMR, $^{13}$C NMR spectra of 3c
$^1$H NMR, $^{13}$C NMR spectra of 3d
$^1$H NMR, $^{13}$C NMR spectra of 3f
$^1$H NMR, $^{13}$C NMR spectra of 3g
$^1$H NMR, $^{13}$C NMR spectra of 3h
$^1$H NMR, $^{13}$C NMR spectra of 3i
$^{1}H$ NMR, $^{13}$C NMR spectra of 3j
$^1$H NMR, $^{13}$C NMR spectra of 3k
$^1$H NMR, $^{13}$C NMR spectra of 3l

![NMR Spectra Image]

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$^{1} \text{H NMR, } ^{13} \text{C NMR spectra of 3m}$
$^1$H NMR, $^{13}$C NMR spectra of 3n
$^1$H NMR, $^{13}$C NMR spectra of 3o
$^1$H NMR, $^{13}$C NMR spectra of 3p
$^1$H NMR, $^{13}$C NMR spectra of 3q
$^1$H NMR, $^{13}$C NMR spectra of 3r
$^{1}H$ NMR, $^{13}C$ NMR spectra of 3s
$^1$H NMR, $^{13}$C NMR spectra of 3t
$^1$H NMR, $^{13}$C NMR spectra of 3u
$^{1}H$ NMR, $^{13}C$ NMR spectra of 3v
$^1$H NMR, $^{13}$C NMR spectra of 3w
HPLC spectra for chiral compounds 3a

Detector A Ch1 254nm

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