Ferric chloride-catalyzed C–N bond cleavage for the cyclization of arylallenes leading to polysubstituted indenes

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Supporting information

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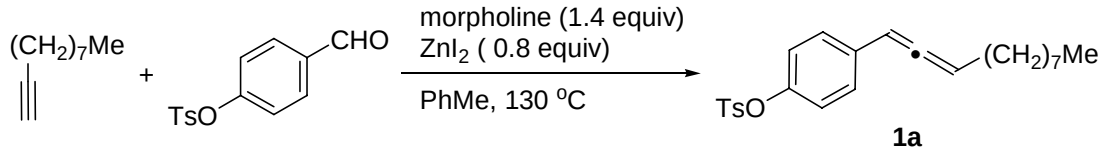
General information

^{1}H and ^{13}C NMR Spectra were recorded on a Bruker AC-400 FT spectrometer (400 MHz and 100 MHz, respectively) using tetramethylsilane as internal reference. Chemical shifts (δ) and coupling constants (J) were expressed in ppm and Hz, respectively. IR spectra were recorded on a Perkin-Elmer 2000 FTIR spectrometer. High resolution mass spectra were recorded on a LC-TOF spectrometer (Micromass). Melting points were uncorrected.

Sulfonamides **2a-l**, benzhydrylamine derivatives **2ab-ae**,[1] and allenes **1b-e**[2] and **1f**[3] were prepared according to literature procedures. The rest of chemicals were purchased from the Sinopharm Chemical Reagent Co., Meryer, Acros, and Alfa Aesar, and used as received. Solvents were dried over magnesium sulfate before use.

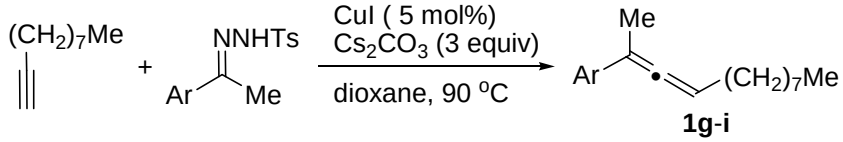
Preparation of allene 1a[2]



1a

Under a nitrogen atmosphere, morpholine (122 mg, 1.4 mmol) was added to a mixture of 1-decyne (138 mg, 1.0 mmol), 4-formylphenyl 4-methylbenzenesulfonate (497 mg, 1.8 mmol), and ZnI_2 (255 mg, 0.80 mmol) in toluene (5.0 mL). The mixture was stirred at 130 ^{o}C for 10 h, and cooled to room temperature. The mixture was purified by silica gel column chromatography, eluting with petroleum ether/ethyl acetate (20:1), to give allene **1a** (203 mg, 51%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (d, J = 8.4 Hz, 2H), 7.30 (d, J = 8.4 Hz, 2H), 7.17 (d, J = 8.8 Hz, 2H), 6.89 (d, J = 8.8 Hz, 2H), 6.07-6.03 (m, 1H), 5.58-5.53 (m, 1H), 2.44 (s, 3H), 2.14-2.07 (m, 2H), 1.46-1.40 (m, 2H), 1.38-1.25 (m, 10H), 0.87 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.4, 147.1, 144.2, 133.3, 131.5, 128.7, 127.5, 126.5, 121.5, 94.5, 92.5, 30.8, 28.1, 27.6, 21.6, 20.7, 13.1; IR (film) ν 3066, 2927, 1709, 1597, 1500, 1462 cm^{-1}; HRMS (EI) calcd for $C_{24}H_{30}O_3S$ (M) 398.1916, found 398.1922.

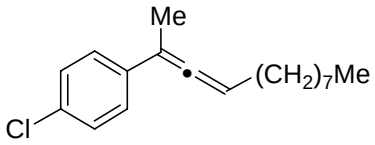
Preparation of allenes 1g-i[3]



1g-i

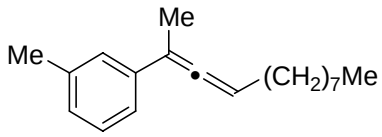
Under a nitrogen atmosphere, 1-decyne (276 mg, 2.0 mmol) was added to a mixture of CuI (19.0 mg, 0.10 mmol), Cs_2CO_3 (1.96 g, 6.0 mmol), and the corresponding *N*-alkylidene-*p*-tosylhydrazine (2.6 mmol) in dioxane (20 mL). The mixture was stirred at 90 ^{o}C until no further transformation was detected by TLC analysis. The solvent was then removed in vacuo to provide a crude mixture, which was purified by column chromatography on silica gel to give the corresponding

allene.



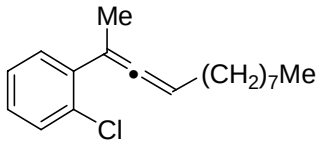
1g

Allene **1g** was obtained in 17% yield as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (d, J = 8.8 Hz, 2H), 7.26 (d, J = 8.8 Hz, 2H), 5.46-5.42 (m, 1H), 2.13-2.06 (m, 2H), 2.06 (s, 3H), 1.48-1.41 (m, 2H), 1.39-1.25 (m, 10H), 0.87 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.2, 136.4, 131.9, 128.3, 126.8, 99.5, 93.5, 31.9, 29.4, 29.3, 29.2, 28.9, 22.7, 19.2, 17.1, 14.1 ; IR (film) ν 3068, 3032, 2927, 1708, 1599, 1500, 1462 cm^{-1}; HRMS (EI) calcd for $C_{18}H_{25}Cl$ (M) 276.1645, found 276.1653.



1h

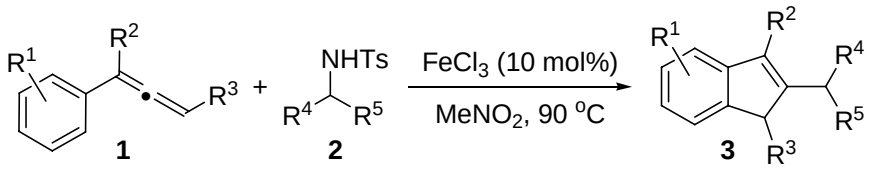
Allene **1h** was obtained in 19% yield as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21-7.17 (m, 3H), 7.00 (s, 1H), 5.44-5.39 (m, 1H), 2.34 (s, 3H), 2.26-2.22 (m, 1H), 2.13-2.07 (m, 1H), 2.07 (s, 3H), 1.52-1.26 (m, 12H), 0.87 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.1, 137.7, 131.3, 128.1, 127.1, 126.3, 122.7, 100.3, 92.9, 31.8, 29.2, 28.9, 28.4, 22.7, 21.5, 19.2, 17.2, 14.1; IR (film) ν 3023, 2925, 1712, 1607, 1487, 1463 cm^{-1}; HRMS (EI) calcd for $C_{19}H_{28}$ (M) 256.2191, found 256.2192.



1i

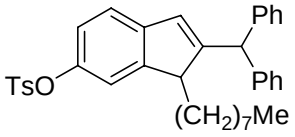
Allene **1i** was obtained in 14% yield as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37-7.34 (m, 1H), 7.30-7.27 (m, 1H), 7.21-7.12 (m, 2H), 5.24-5.18 (m, 1H), 2.31-2.29 (m, 1H), 2.09-2.02 (m, 4H), 1.45-1.36 (m, 2H), 1.32-1.25 (m, 10H), 0.88 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.9, 138.4, 130.0, 127.8, 126.6, 124.6, 121.7, 98.7, 91.3, 31.9, 29.7, 29.4, 29.3, 29.1, 28.7, 22.7, 20.2, 14.1; IR (film) ν 3063, 2924, 1730, 1594, 1463, 1435 cm^{-1}; HRMS (EI) calcd for $C_{18}H_{25}Cl$ (M) 276.1645, found 276.1648.

General procedure for the synthesis of polysubstituted indenes from arylallenes and *N*-alkyl sulfonamides



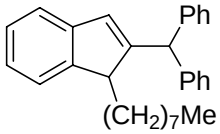
To a solution of *N*-alkyl sulfonamide **2** (0.20 mmol) in dry nitromethane (2.0 mL) under nitrogen were added arylallene **1** (0.30 mmol) and $FeCl_3$ (3.3 mg, 0.020 mmol). The resulting mixture was stirred at 90 °C until no further transformation was detected by TLC analysis. The mixture was cooled to room temperature, and purified by silica gel column chromatography, eluting with petroleum ether/ethyl acetate (50:1 to 10:1), to give indene **3**.

Analytical data for the products



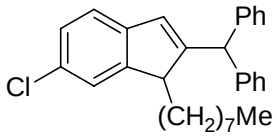
3a

Indene **3a**, colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, J = 8.4 Hz, 2H), 7.34-7.18 (m, 10H), 7.15 (d, J = 8.4 Hz, 2H), 7.02 (d, J = 8.0 Hz, 1H), 6.94-6.92 (m, 1H), 6.79-6.75 (m, 1H), 6.09 (s, 1H), 5.01 (s, 1H), 3.37-3.34 (m, 1H), 2.42 (s, 3H), 1.80-1.68 (m, 2H), 1.30-1.17 (m, 12H), 0.88 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.3, 149.0, 147.0, 145.0, 143.0, 142.6, 142.3, 132.7, 128.6, 126.5, 120.7, 120.5, 117.4, 51.9, 50.4, 31.9, 29.9, 29.7, 29.4, 29.3, 24.0, 22.7, 21.7, 14.1; IR (film) ν 3062, 3027, 2927, 1598, 1494, 1466 cm^{-1}; HRMS (EI) calcd for $C_{37}H_{40}O_3S$ (M) 564.2698, found 564.2695.



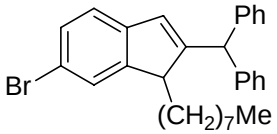
3b

Indene **3b**, colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33-7.09 (m, 14H), 6.16 (s, 1H), 5.06 (s, 1H), 3.43-3.39 (m, 1H), 1.97-1.85 (m, 1H), 1.82-1.73 (m, 1H), 1.28-1.19 (m, 12H), 0.87 (t, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.8, 147.6, 144.1, 142.7, 130.0, 129.2, 129.0, 128.5, 128.3, 126.6, 126.4, 124.2, 122.8, 120.5, 51.9, 50.2, 31.8, 30.0, 29.7, 29.4, 29.3, 24.1, 22.7, 14.1; IR (film) ν 3062, 3025, 2926, 1601, 1492, 1458 cm^{-1}; HRMS (EI) calcd for $C_{30}H_{34}$ (M) 394.2661, found 394.2662.



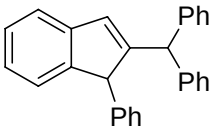
3c

Indene **3c**, white solid, m.p. 118-120 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34-7.24 (m, 9H), 7.17-7.15 (m, 3H), 7.08 (d, J = 8.0 Hz, 1H), 6.11 (s, 1H), 5.03 (s, 1H), 3.42-3.39 (m, 1H), 1.97-1.89 (m, 1H), 1.80-1.74 (m, 1H), 1.29-1.20 (m, 12H), 0.88 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.4, 149.4, 142.7, 142.6, 142.4, 129.1, 128.6, 126.8, 126.5, 123.3, 121.2, 51.9, 50.4, 31.8, 29.9, 29.5, 29.4, 29.3, 24.0, 22.7, 14.1; IR (film) ν 3062, 3026, 2956, 2926, 1598, 1561, 1493, 1462 cm^{-1}; HRMS (EI) calcd for $C_{30}H_{33}Cl$ (M) 428.2271, found 428.2279.



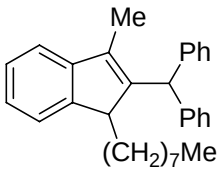
3d

Indene **3d**, colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (s, 1H), 7.32-7.23 (m, 5H), 7.23-7.20 (m, 4H), 7.17-7.14 (m, 2H), 7.02 (d, J = 8.0 Hz, 1H), 6.10 (s, 1H), 5.02 (s, 1H), 3.41-3.38 (m, 1H), 1.92-1.81 (m, 1H), 1.79-1.73 (m, 1H), 1.28-1.15 (m, 12H), 0.88 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.4, 149.7, 143.0, 142.7, 142.3, 129.4, 129.1, 128.6, 126.7, 126.1, 121.7, 118.3, 51.9, 50.4, 31.8, 29.9, 29.5, 29.3, 29.2, 24.0, 22.7, 14.1; IR (film) ν 3060, 3025, 2925, 1600, 1492, 1451 cm^{-1}; HRMS (EI) calcd for $C_{30}H_{33}Br$ (M) 472.1766, found 472.1773.



3e

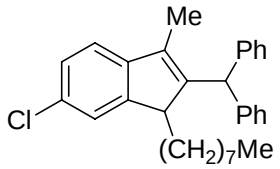
Indene **3e**, white solid, m.p. 96-98 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34-7.15 (m, 13H), 7.12-7.01 (m, 4H), 7.00-6.88 (m, 2H), 6.27 (s, 1H), 4.73 (s, 1H), 4.38 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.1, 148.9, 144.0, 143.0, 142.2, 139.6, 129.8, 129.3, 128.8, 128.6, 128.2, 126.9, 126.7, 126.3, 124.9, 124.0, 120.6, 57.7, 51.5; IR (film) ν 3029, 2927, 1599, 1502, 1459 cm^{-1}; HRMS (EI) calcd for $C_{28}H_{22}$ (M) 358.1722, found 358.1721.



3f

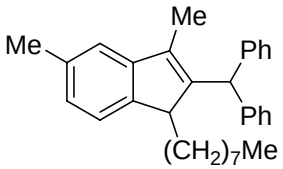
Indene **3f**, colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34-7.13 (m, 14H), 5.35

(s, 1H), 3.40-3.35 (m, 1H) 1.99-1.87 (m, 1H), 1.65 (s, 3H), 1.62-1.40 (m, 1H), 1.19-1.03 (m, 12H), 0.86 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.9, 146.8, 146.4, 143.3, 142.9, 134.9, 129.2, 128.4, 128.2, 126.4, 126.2, 124.3, 122.5, 118.3, 51.8, 50.7, 31.8, 30.2, 29.7, 29.4, 25.0, 22.7, 14.1, 11.4; IR (film) ν 3062, 3026, 2926, 1601, 1493, 1467 cm^{-1}; HRMS (EI) calcd for $C_{31}H_{36}$ (M) 408.2817, found 408.2825.



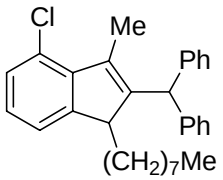
3g

Indene **3g**, colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31-7.10 (m, 13H), 5.32 (s, 1H), 3.40-3.33 (m, 1H), 1.94-1.86 (m, 1H), 1.63 (s, 3H), 1.61-1.56 (m, 1H), 1.22-1.02 (m, 12H), 0.87 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.5, 146.8, 145.4, 142.9, 142.6, 134.4, 130.4, 129.2, 128.5, 128.3, 126.5, 126.3, 122.9, 119.1, 51.8, 50.7, 31.8, 30.0, 29.8, 29.3, 24.8, 22.7, 14.1, 11.4; IR (film) ν 3064, 3027, 2927, 1649, 1597, 1490, 1465 cm^{-1}; HRMS (EI) calcd for $C_{31}H_{35}Cl$ (M) 442.2427, found 442.2420.



3h

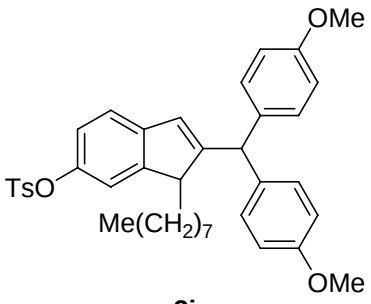
Indene **3h**, colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29-7.14 (m, 11H), 7.05 (s, 1H), 6.97 (d, J = 8.4 Hz, 1H), 5.34 (s, 1H), 3.38-3.30 (m, 1H), 2.39 (s, 3H), 1.96-1.85 (m, 1H), 1.64 (s, 3H), 1.51-1.45 (m, 1H), 1.26-0.95 (m, 12H), 0.86 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.1, 146.7, 143.9, 143.3, 142.9, 135.8, 134.7, 129.3, 128.3, 128.2, 126.3, 126.2, 125.0, 122.2, 119.1, 51.5, 50.7, 31.8, 30.3, 29.9, 29.4, 29.3, 25.1, 22.7, 21.6, 14.1, 11.4; IR (film) ν 3061, 3025, 2926, 1600, 1493, 1461, 1414 cm^{-1}; HRMS (EI) calcd for $C_{32}H_{38}$ (M) 422.2974, found 442.2979.



3i

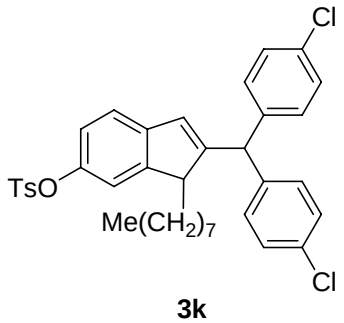
Indene **3i**, colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31-7.16 (m, 12H), 7.05-7.01 (m, 1H), 5.38 (s, 1H), 3.44-3.34 (m, 1H), 2.06 (s, 3H), 1.90-1.80 (m, 1H), 1.28-1.21 (m, 1H), 1.16-0.96 (m, 12H), 0.86 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.0, 148.1, 142.9, 142.6, 142.2, 135.8, 129.2, 129.1, 128.4, 126.6, 126.4, 126.3, 125.3, 121.0, 52.0, 50.6, 31.8, 29.8, 29.3, 29.2, 24.8, 22.7, 15.3, 14.1; IR (film)

v 3060, 3026, 2926, 1600, 1564, 1493, 1451 cm^{-1}; HRMS (EI) calcd for $C_{31}H_{35}Cl$ (M) 442.2427, found 442.2426.



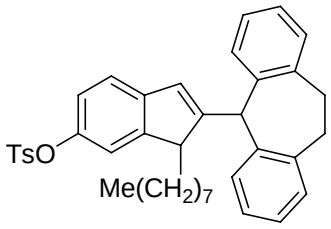
3j

Indene **3j**, colorless oil; ^1H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, J = 8.0 Hz, 2H), 7.27 (d, J = 8.0 Hz, 2H), 7.11 (d, J = 8.4 Hz, 2H), 7.06-6.98 (m, 3H), 6.91 (s, 1H), 6.86-6.81 (m, 4H), 6.79-6.76 (m, 1H), 6.08 (s, 1H), 4.92 (s, 1H), 3.78 (s, 6H), 3.35-3.31 (m, 1H), 2.42 (s, 3H), 1.81-1.68 (m, 2H), 1.22-1.16 (m, 12H), 0.88 (t, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.4, 158.2, 149.0, 147.0, 145.0, 143.1, 132.7, 129.6, 128.6, 122.0, 120.5, 117.3, 114.0, 113.7, 55.2, 50.3, 50.2, 31.9, 29.9, 29.7, 29.4, 29.3, 24.0, 22.7, 21.7, 14.1; IR (film) *v* 3062, 3026, 2926, 1597, 1491, 1455, 1408 cm^{-1}; HRMS (EI) calcd for $C_{39}H_{44}O_5S$ (M) 624.2909, found 624.2911.



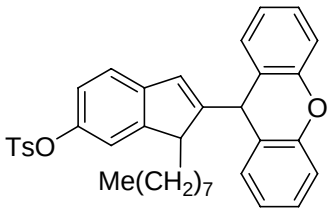
3k

Indene **3k**, colorless oil; ^1H NMR (400 MHz, $CDCl_3$) δ 7.70 (d, J = 8.4 Hz, 2H), 7.30-7.24 (m, 6H), 7.13 (d, J = 8.4 Hz, 2H), 7.06-7.02 (m, 3H), 6.95 (s, 1H), 6.80-6.77 (m, 1H), 6.07 (s, 1H), 4.96 (s, 1H), 3.35-3.30 (m, 1H), 2.43 (s, 3H), 1.82-1.76 (m, 1H), 1.70-1.63 (m, 1H), 1.25-1.09 (m, 12H), 0.88 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.1, 148.8, 147.3, 145.1, 142.6, 140.6, 140.4, 130.1, 129.6, 129.0, 128.6, 120.9, 120.7, 117.5, 50.6, 50.4, 31.8, 29.9, 29.7, 29.4, 29.3, 24.2, 22.7, 21.7, 14.1; IR (film) *v* 3062, 3028, 2927, 1606, 1509, 1464 cm^{-1}; HRMS (EI) calcd for $C_{37}H_{38}O_3SCl_2$ (M) 632.1919, found 632.1927.



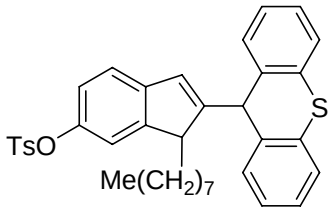
3l

Indene **3l**, colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (d, J = 8.4 Hz, 2H), 7.35-7.32 (m, 1H), 7.25 (d, J = 8.4 Hz, 2H), 7.19-7.08 (m, 7H), 6.97 (d, J = 8.4 Hz, 1H), 6.83 (s, 1H), 6.76-6.73 (m, 1H), 5.91 (s, 1H), 4.77 (s, 1H), 3.43-3.39 (m, 1H), 3.28-3.21 (m, 1H), 3.10-3.85 (m, 2H), 2.54-2.48 (m, 1H), 2.41 (s, 3H), 1.82-1.68 (m, 2H), 1.33-1.00 (m, 12H), 0.90 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.9, 149.0, 146.8, 145.0, 142.9, 141.4, 141.1, 138.7, 137.8, 132.7, 131.7, 131.6, 129.7, 129.6, 129.3, 128.3, 127.6, 126.9, 126.3, 125.9, 120.5, 117.2, 55.3, 49.7, 31.9, 29.8, 29.5, 29.3, 22.7, 21.7, 14.1; IR (film) ν 3061, 3017, 2927, 1598, 1494, 1464 cm^{-1}; HRMS (EI) calcd for $C_{39}H_{42}O_3S$ (M) 590.2855, found 590.2852.



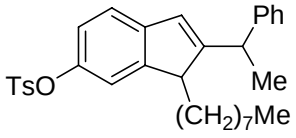
3m

Indene **3m**, colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (d, J = 8.4 Hz, 2H), 7.28-7.00 (m, 11H), 6.86 (s, 1H), 6.74-6.71 (m, 1H), 6.25 (s, 1H), 5.08 (s, 1H), 3.30-3.27 (m, 1H), 2.40 (s, 3H), 1.89-1.79 (m, 1H), 1.75-1.67 (m, 1H), 1.30-1.02 (m, 12H), 0.88 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.7, 151.8, 151.5, 149.0, 147.1, 145.0, 142.6, 132.5, 129.6, 129.5, 128.8, 128.6, 128.2, 128.1, 127.5, 123.4, 123.2, 123.1, 122.8, 120.9, 120.5, 117.2, 116.8, 116.7, 49.3, 39.7, 29.7, 29.4, 29.3, 23.8, 22.7, 21.7, 14.1; IR (film) ν 3065, 3041, 2926, 1599, 1575, 1476, 1458 cm^{-1}; HRMS (EI) calcd for $C_{37}H_{38}O_4S$ (M) 578.2491, found 578.2483.



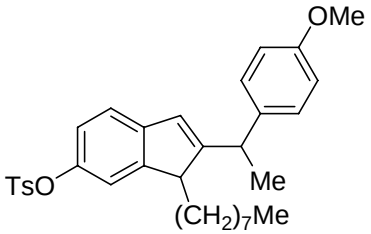
3n

Indene **3n**, colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (d, J = 8.4 Hz, 2H), 7.48-7.42 (m, 2H), 7.36-7.15 (m, 8H), 6.91-6.88 (m, 2H), 6.67-6.40 (m, 1H), 5.83 (s, 1H), 5.01 (s, 1H), 3.10-3.07 (m, 1H), 2.38 (s, 3H), 1.99-1.94 (m, 2H), 1.33-1.22 (m, 12H), 0.90 (t, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.0, 149.2, 147.0, 145.0, 142.8, 136.3, 135.7, 133.4, 133.1, 132.6, 129.6, 128.6, 127.4, 127.1, 127.0, 126.8, 126.7, 121.9, 120.8, 120.4, 117.2, 49.3, 48.9, 31.9, 30.0, 29.6, 29.4, 23.8, 22.7, 21.7, 14.2; IR (film) ν 3062, 3014, 2927, 1597, 1466 cm^{-1}; HRMS (EI) calcd for $C_{37}H_{38}O_3S_2$ (M) 594.2262, found 594.2266.



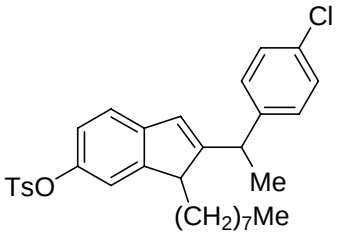
3o

Indene **3o**, 54:46 dr, colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, J = 8.0 Hz, 2H), 7.33-7.14 (m, 8H), 7.00-6.95 (m, 1H), 6.87-6.82 (m, 1H), 6.67 (s, 1H), 3.78-3.65 (m, 1H), 3.08-3.05 (m, 1H), 2.42 (s, 3H), 1.87-1.60 (m, 2H), 1.54 (d, J = 7.2 Hz, 3H), 1.29-1.11 (m, 12H), 0.87 (t, J = 7.2 Hz, 3H); Partial ^{1}H NMR for the minor diastereomer δ 6.03 (s, 1H), 3.51-3.48 (m, 1H), 1.52 (d, J = 7.2 Hz, 3H); IR (film) v 3062, 3027, 2927, 1599, 1496, 1464 cm^{-1}; HRMS (EI) calcd for $C_{32}H_{38}O_3S$ (M) 502.2542, found 502.2549.



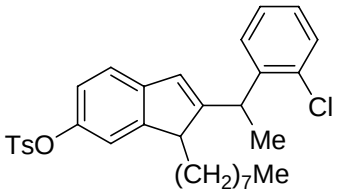
3p

Indene **3p**, 55:45 dr, colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, J = 8.0 Hz, 2H), 7.26 (d, J = 8.4 Hz, 2 H), 7.19-7.13 (m, 1H), 7.06 (d, J = 8.8 Hz, 1H), 7.00-6.95 (m, 1H), 6.89-6.73 (m, 4H), 6.64 (s, 1H), 3.77 (s, 3H), 3.73-3.62 (m, 1H), 3.13-3.02 (m, 1H), 2.42 (s, 3H), 1.88-1.59 (m, 2H), 1.51 (d, J = 7.6 Hz, 3H), 1.30-1.05 (m, 12H), 0.87 (t, J = 7.2 Hz, 3H); Partial ^{1}H NMR for the minor diastereomer δ 6.63 (s, 1H), 3.80 (s, 3H), 3.50-3.46 (m, 1H), 1.49 (d, J = 7.6 Hz, 3H); IR (film) v 3063, 3029, 2927, 1602, 1510, 1464 cm^{-1}; HRMS (EI) calcd for $C_{33}H_{40}O_4S$ (M) 532.2647, found 532.2642.



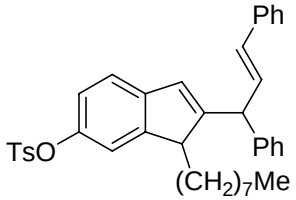
3q

Indene **3q**, 93:7 dr, colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, J = 8.4 Hz, 2H), 7.29-7.22 (m, 4H), 7.16 (d, J = 8.0 Hz, 1H), 7.08 (d, J = 8.4 Hz, 2H), 6.96-6.83 (m, 2H), 6.67 (s, 1H), 3.70-3.65 (m, 1H), 3.06-3.03 (m, 1H), 2.43 (s, 3H), 1.78-1.70 (m, 1H), 1.69-1.60 (m, 1H), 1.52 (d, J = 7.2 Hz, 3H), 1.29-1.13 (m, 12H), 0.87 (t, J = 7.2 Hz, 3H); Partial ^{1}H NMR for the minor diastereomer δ 6.03 (s, 1H), 3.50-3.47 (m, 1H); IR (film) v 3065, 3028, 2927, 1598, 1490, 1465 cm^{-1}; HRMS (EI) calcd for $C_{32}H_{37}ClO_3S$ (M) 536.2174, found 536.2155.



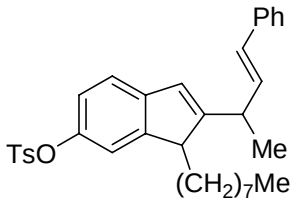
3r

Indene **3r**, 65:35 dr, colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (d, J = 8.4 Hz, 2H), 7.40-7.34 (m, 1H), 7.27 (d, J = 8.8 Hz, 2H), 7.21-7.10 (m, 3H), 7.03-6.95 (m, 1H), 6.90-6.75 (m, 2H), 6.71 (s, 1H), 4.35-4.26 (m, 1H), 3.10-3.06 (m, 1H), 2.42 (s, 3H), 1.90-1.59 (m, 2H), 1.50 (d, J = 6.8 Hz, 3H), 1.25-1.10 (m, 12H), 0.87 (t, J = 7.2 Hz, 3H); Partial ^{1}H NMR for the minor diastereomer δ 7.68 (d, J = 8.4 Hz, 2H), 6.00 (s, 1H), 3.52-3.48 (m, 1H), 1.51 (d, J = 7.2 Hz, 3H); IR (film) ν 3064, 2927, 1598, 1577, 1496, 1467 cm^{-1}; HRMS (EI) calcd for $C_{32}H_{37}ClO_3S$ (M) 536.2174, found 536.2160.



3s

Indene **3s**, 50:50 dr, colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.10-7.67 (m, 2H), 7.32-7.12 (m, 12H), 7.05-6.95 (m, 2H), 6.82-6.67 (m, 1H), 6.46 (s, 1H), 6.42-6.31 (m, 2H), 4.44 (d, J = 7.2 Hz, 1H), 3.21-3.18 (m, 1H), 2.42 (s, 3H), 1.91-1.73 (m, 2H), 1.30-1.15 (m, 12H), 0.88 (t, J = 7.2 Hz, 3H); Partial ^{1}H NMR for the other diastereomer δ 6.14 (s, 1H), 4.50 (d, J = 7.6 Hz, 1H), 3.57-3.54 (m, 1H); IR (film) ν 3060, 3027, 1598, 1496, 1464 cm^{-1}; HRMS (EI) calcd for $C_{39}H_{42}O_3S$ (M) 590.2855, found 590.2848.



3t

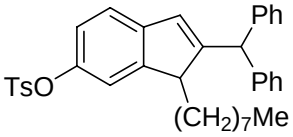
Indene **3t**, 55:45 dr, colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, J = 8.4 Hz, 2H), 7.40 (d, J = 8.4 Hz, 1H), 7.35-7.15 (m, 6H), 7.13-6.91 (m, 2H), 6.83-6.76 (m, 1H), 6.46-6.33 (m, 3H), 3.48-3.40 (m, 1H), 3.35-3.27 (m, 1H), 2.43 (s, 3H), 1.90-1.78 (m, 1H), 1.76-1.68 (m, 1H), 1.35 (d, J = 6.8 Hz, 3H), 1.30-1.09 (m, 12H), 0.86 (t, J = 7.2 Hz, 3H); Partial ^{1}H NMR for the minor diastereomer δ 6.13-6.06 (m, 1H), 3.34-3.28 (m, 1H), 1.42 (d, J = 6.8 Hz, 3H); IR (film) ν 3060, 3026, 2927, 1598, 1496, 1465 cm^{-1}; HRMS (EI) calcd for $C_{34}H_{40}O_3S$ (M) 528.2698, found 528.2672.

References

1 C.-R. Liu, F.-L. Yang, Y.-Z. Jin, X.-T. Ma, D.-J. Cheng, N. Li and S.-K. Tian, *Org. Lett.*, 2010, **12**, 3832.

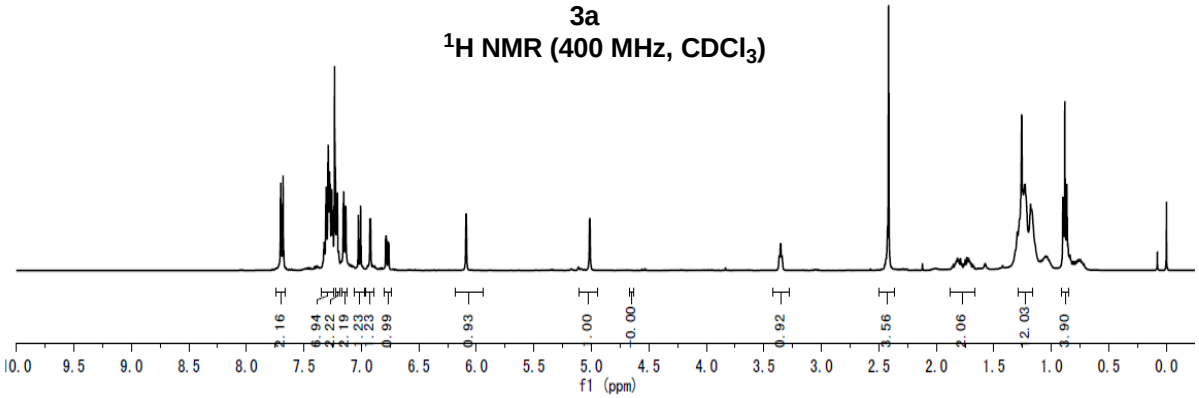
2 J. Kuang and S. Ma, *J. Am. Chem. Soc.*, 2010, **132**, 1786.

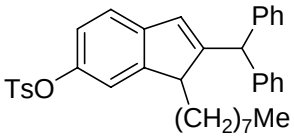
3 Q. Xiao, Y. Xia, H. Li, Y. Zhang and J. Wang, *Angew. Chem., Int. Ed.*, 2011, **50**, 1114.



3a

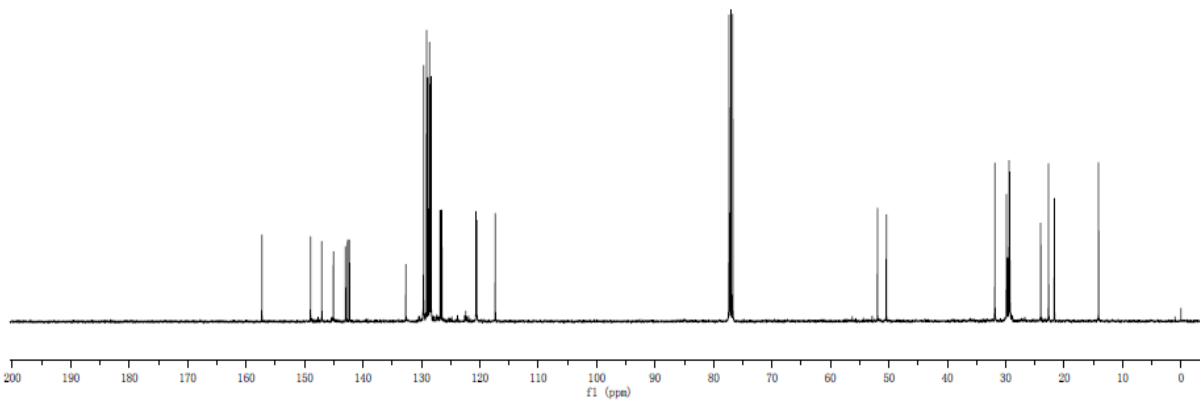
^{1}H NMR (400 MHz, $CDCl_3$)

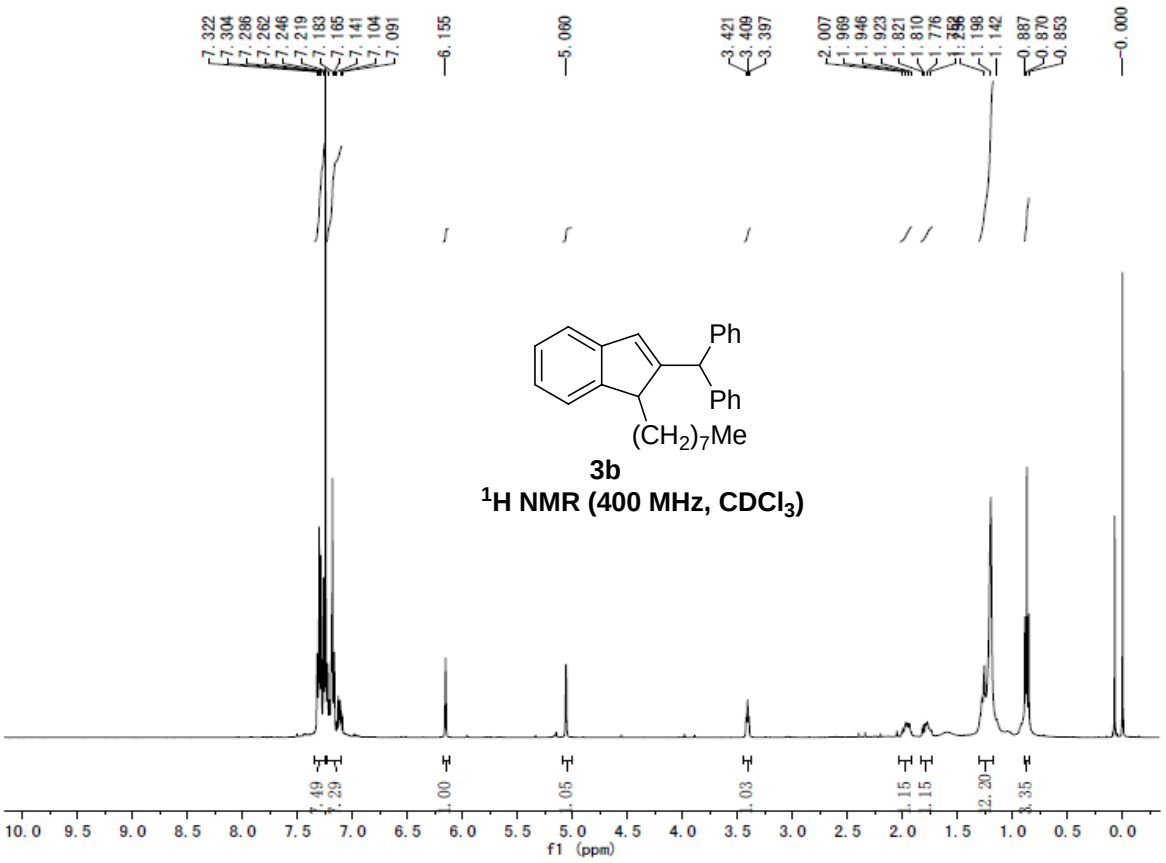




3a

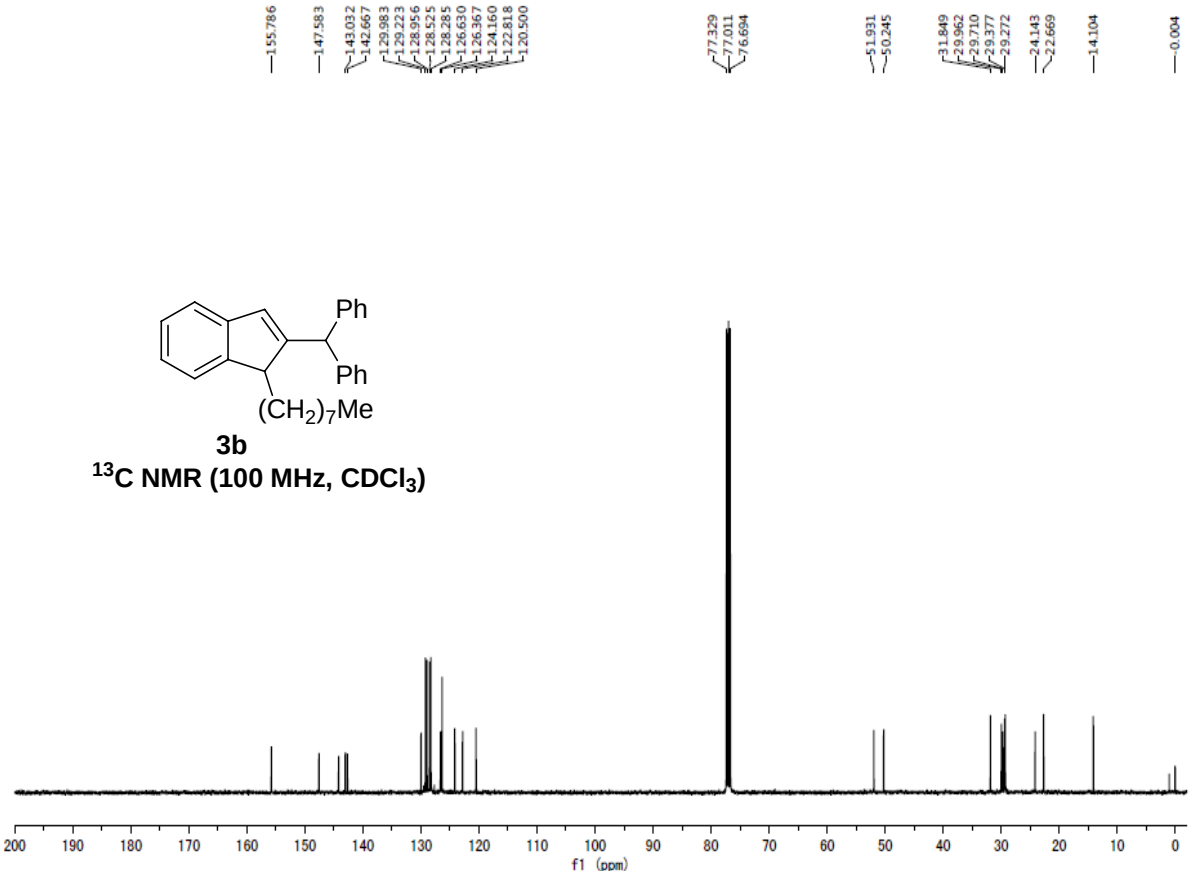
^{13}C NMR (100 MHz, $CDCl_3$)





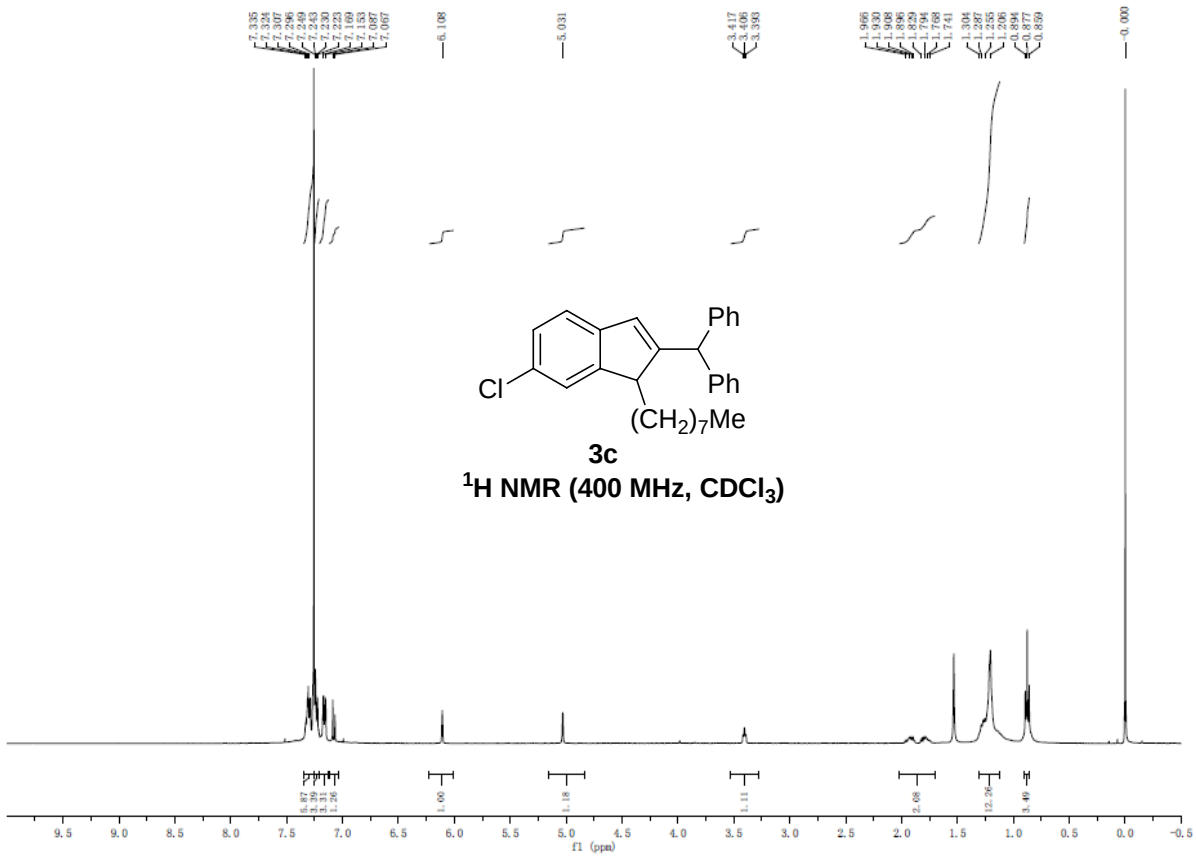
3b

^{1}H NMR (400 MHz, $CDCl_3$)



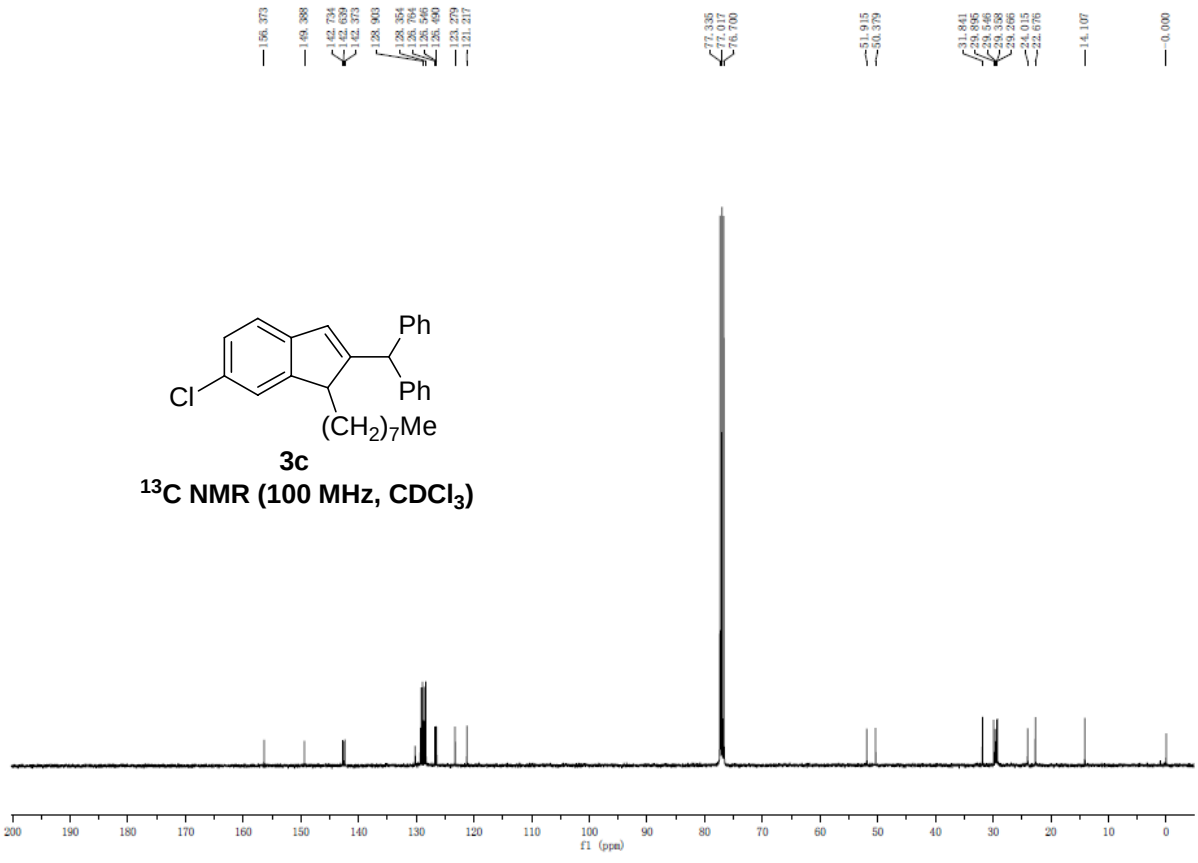
3b

^{13}C NMR (100 MHz, $CDCl_3$)



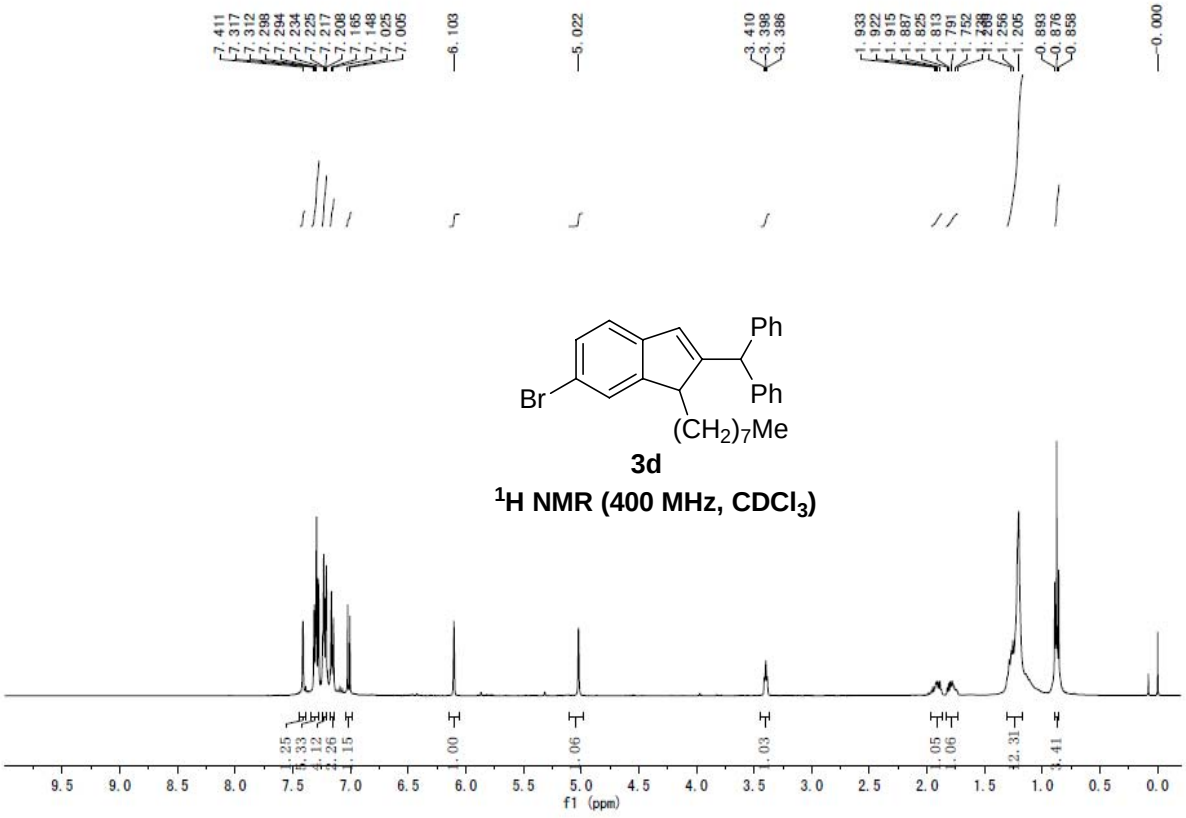
3c

^{1}H NMR (400 MHz, $CDCl_3$)



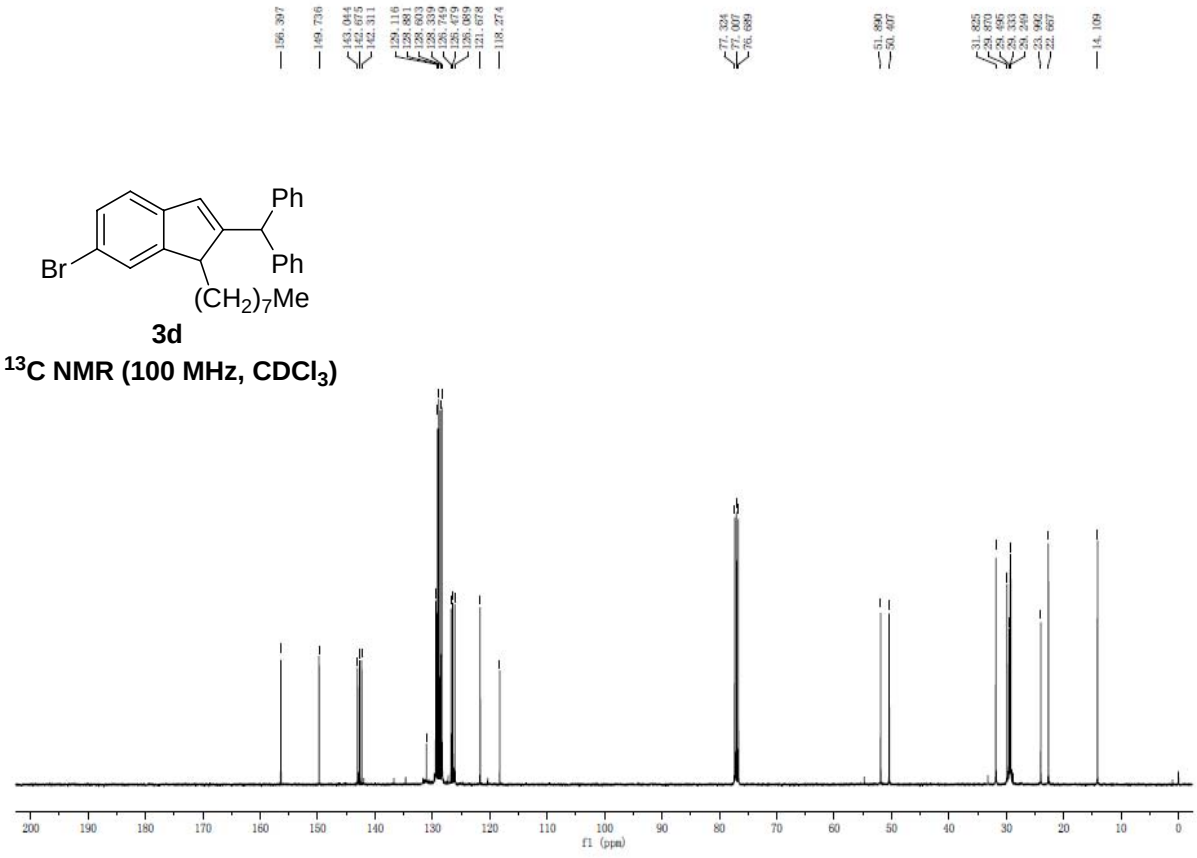
3c

^{13}C NMR (100 MHz, $CDCl_3$)



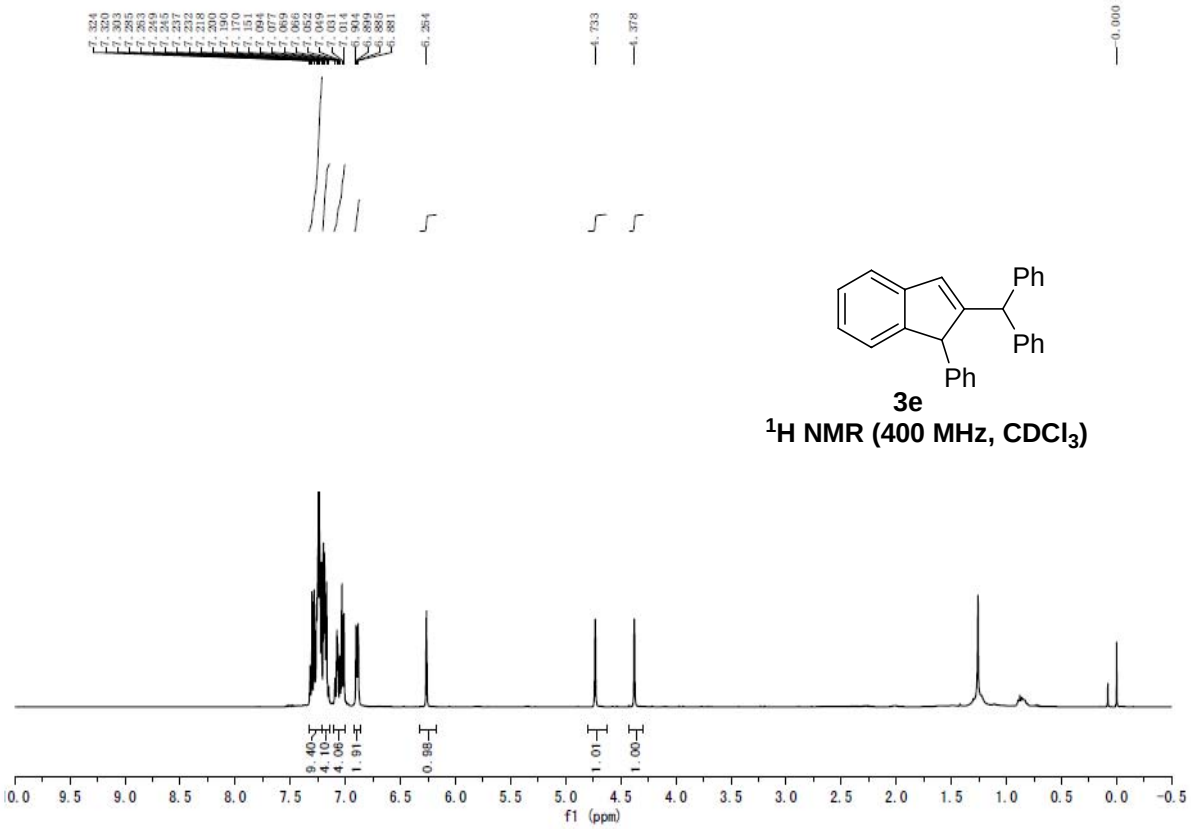
3d

^{1}H NMR (400 MHz, $CDCl_3$)



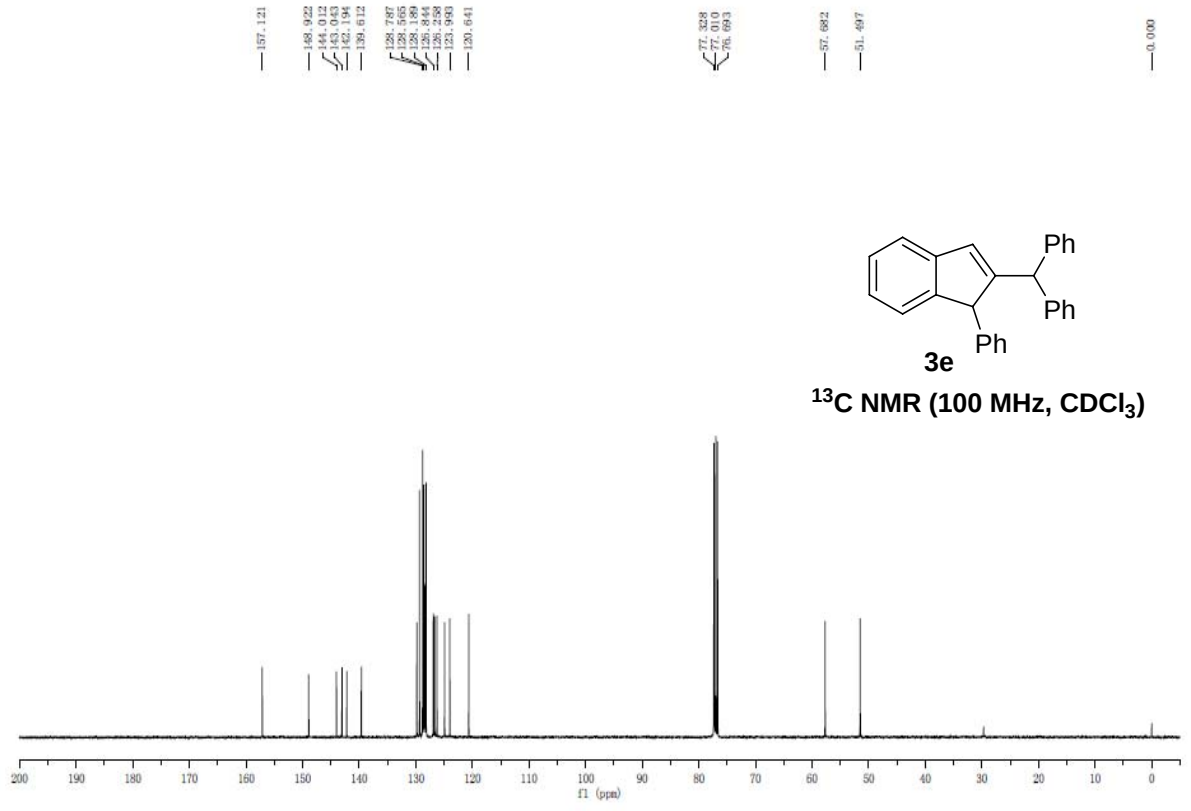
3d

^{13}C NMR (100 MHz, $CDCl_3$)



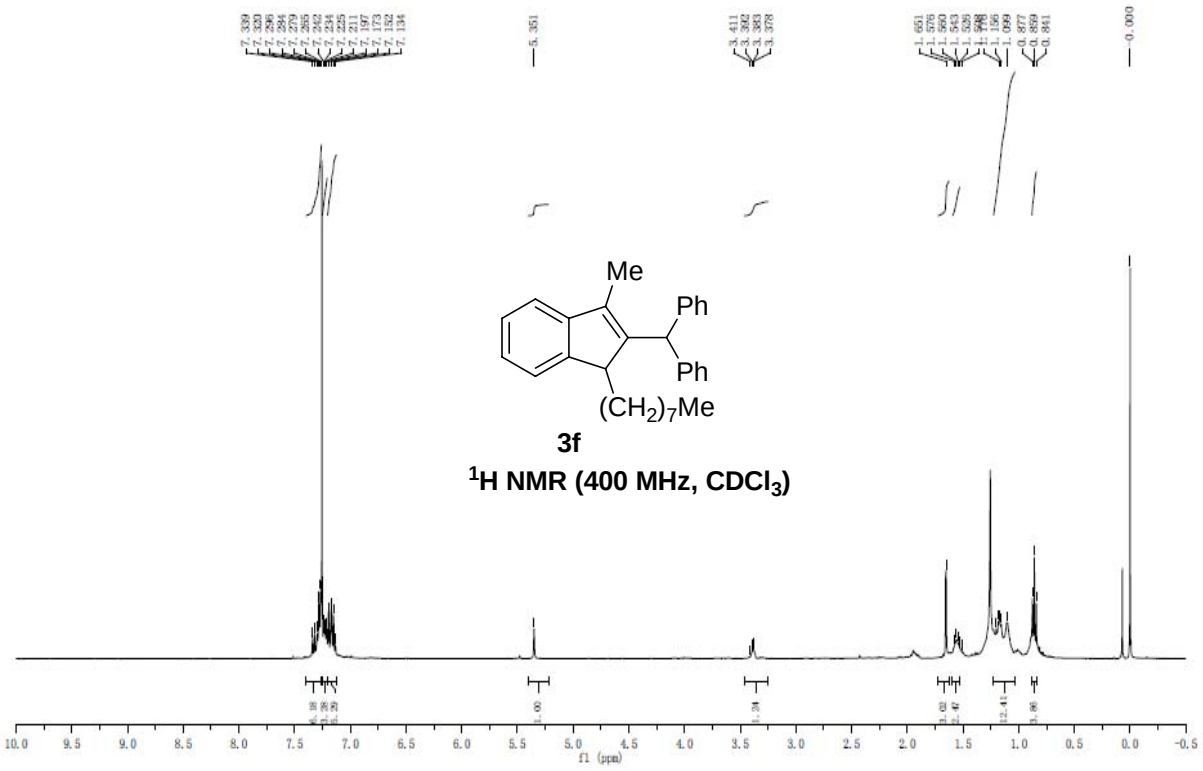
3e

^{1}H NMR (400 MHz, $CDCl_3$)



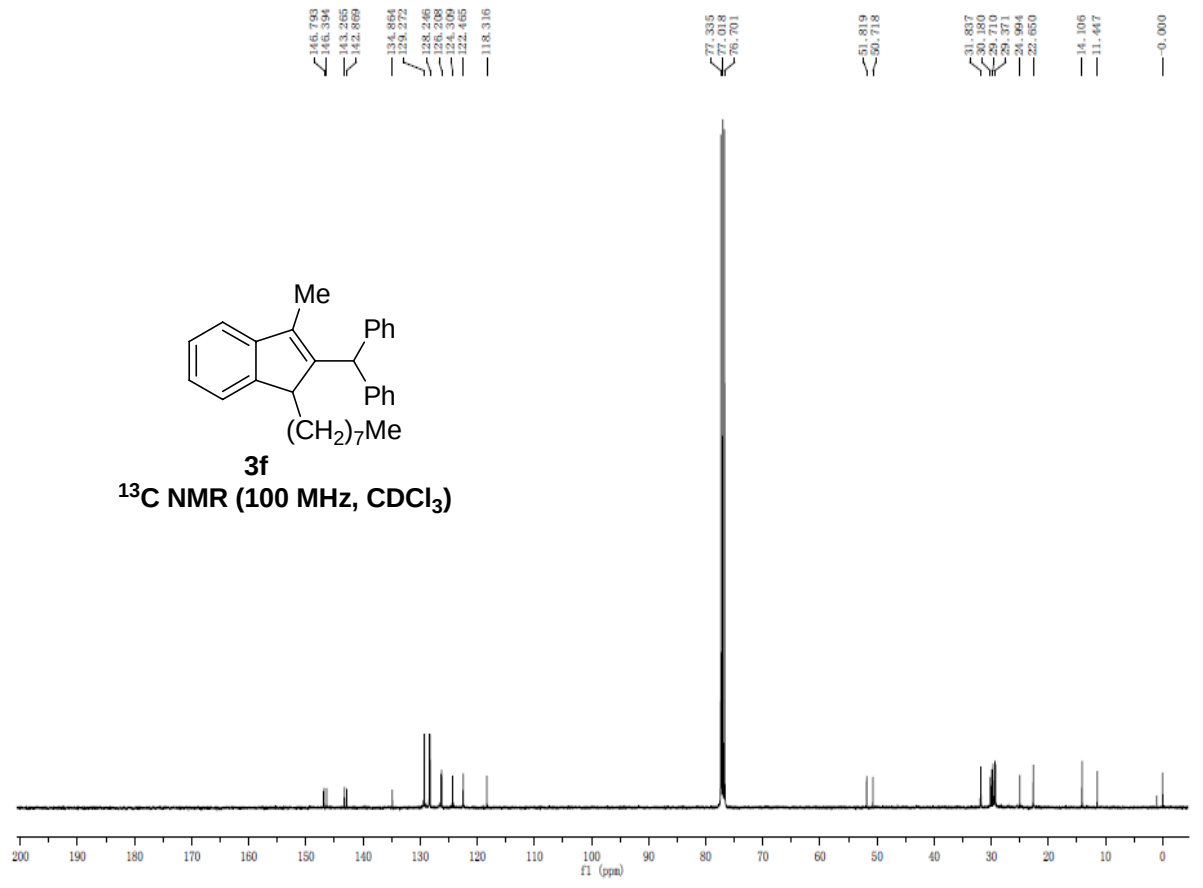
3e

^{13}C NMR (100 MHz, $CDCl_3$)



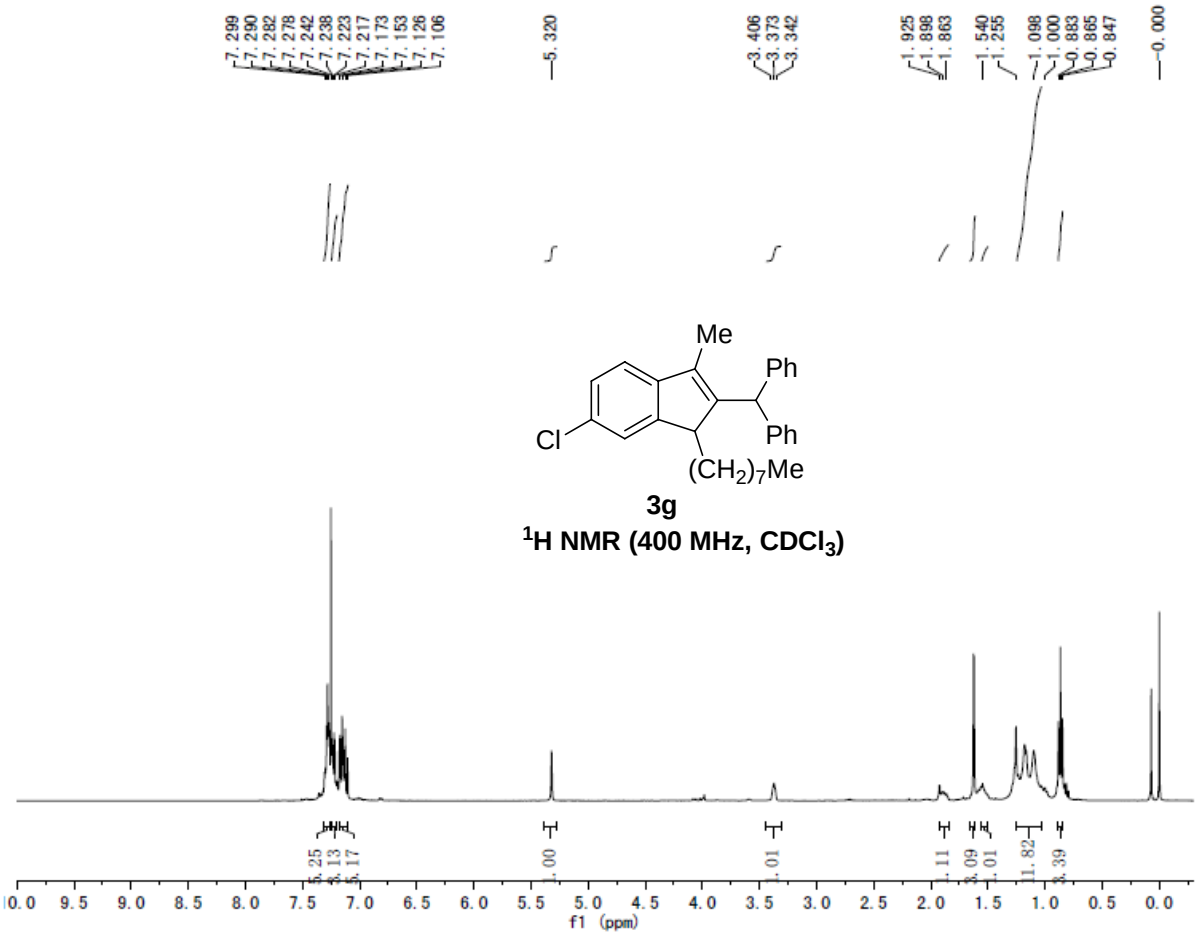
3f

^{1}H NMR (400 MHz, $CDCl_3$)



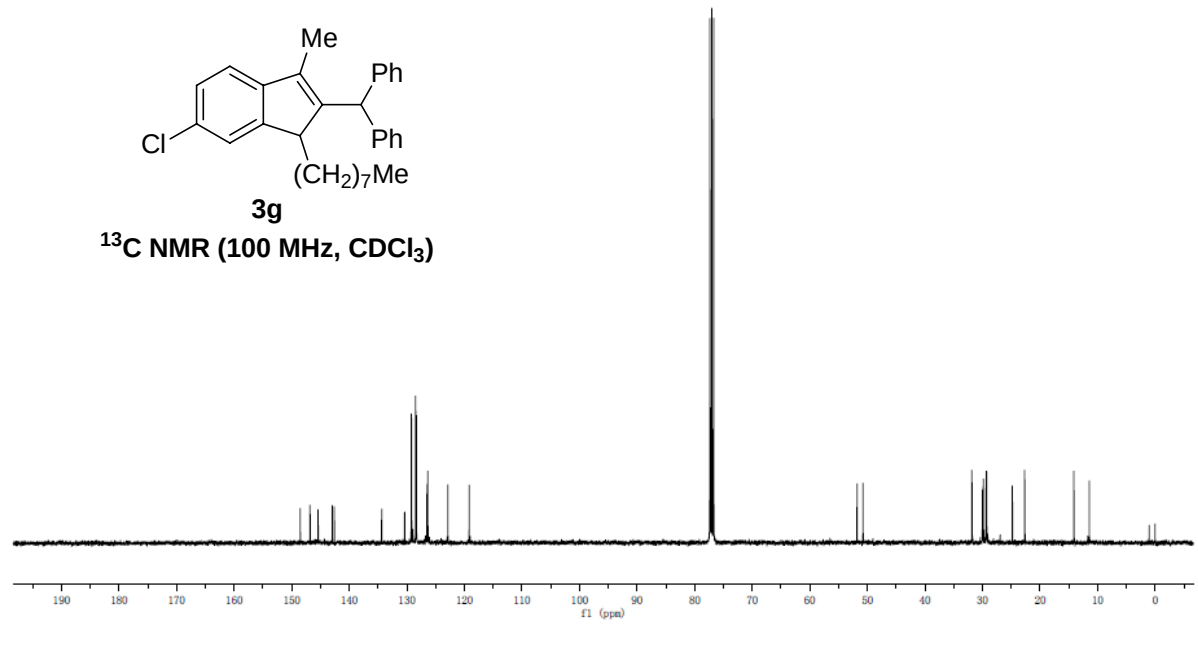
3f

^{13}C NMR (100 MHz, $CDCl_3$)



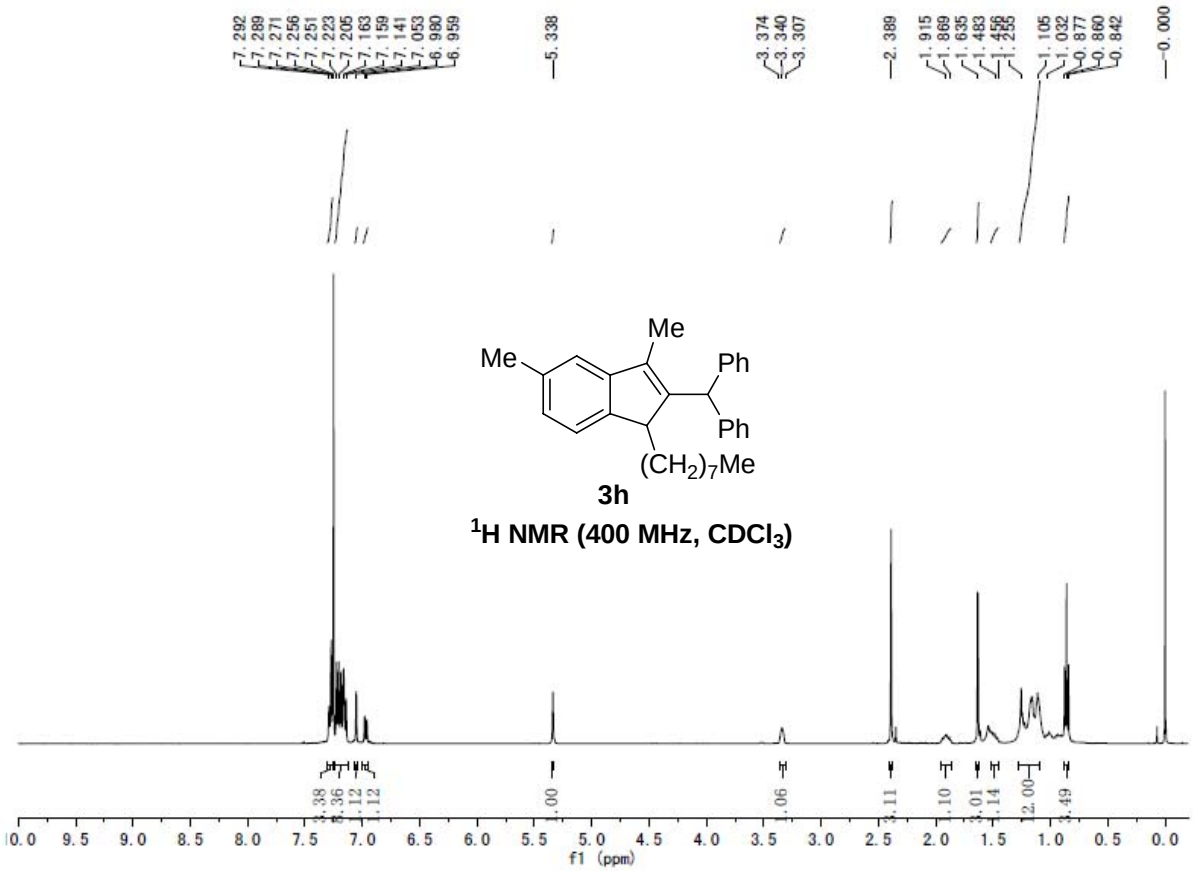
3g

^{1}H NMR (400 MHz, $CDCl_3$)



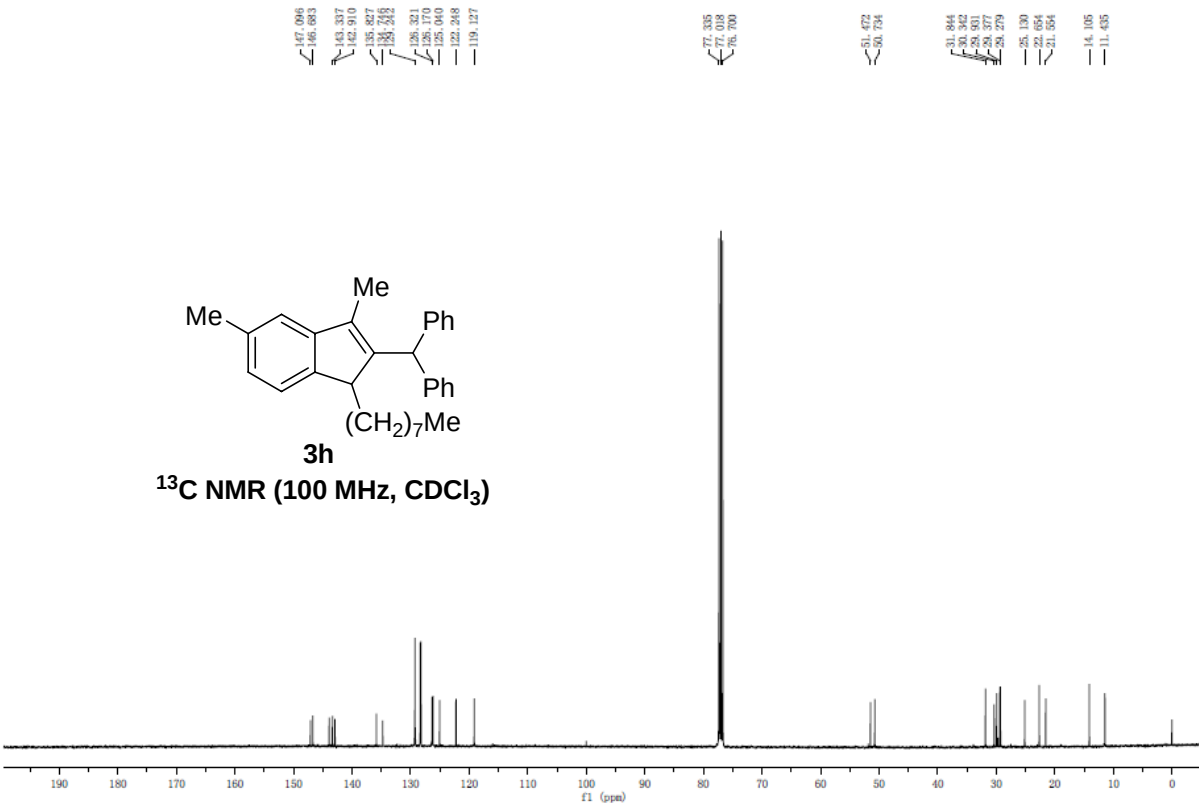
3g

^{13}C NMR (100 MHz, $CDCl_3$)



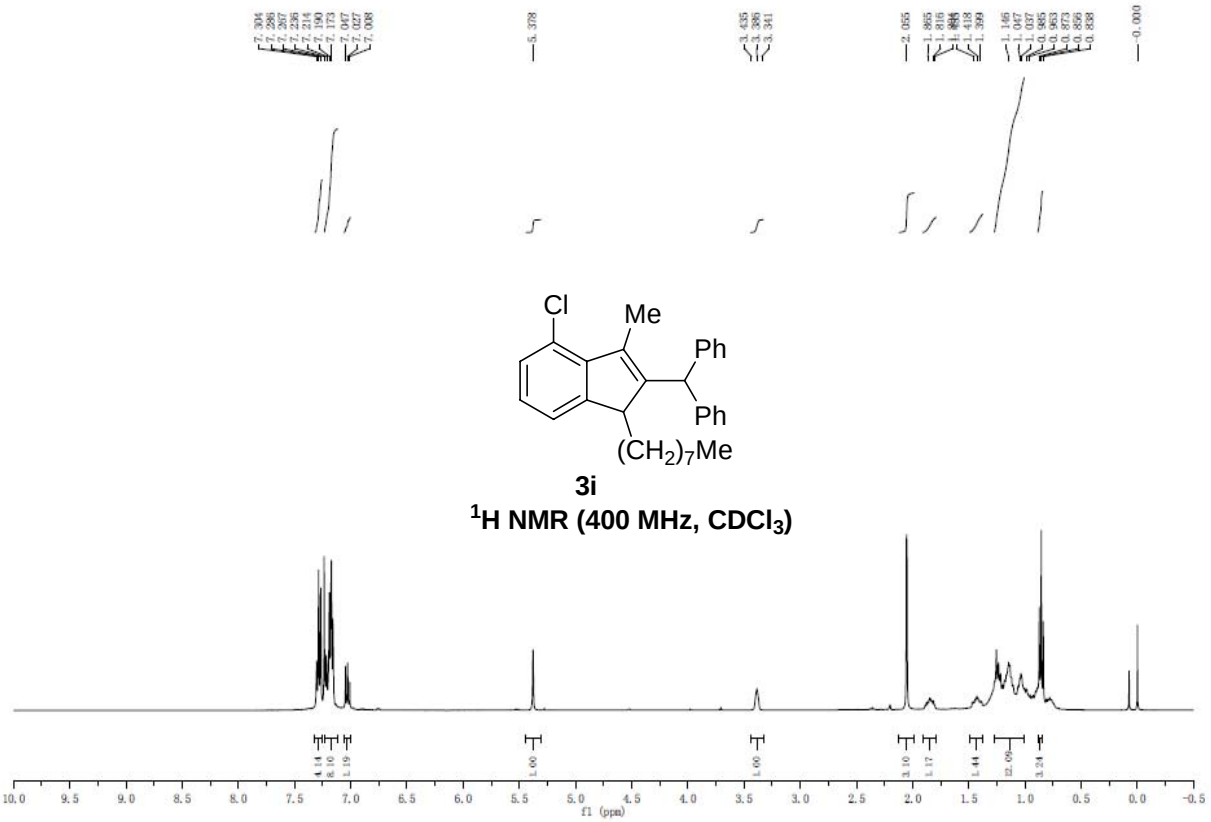
3h

^{1}H NMR (400 MHz, $CDCl_3$)



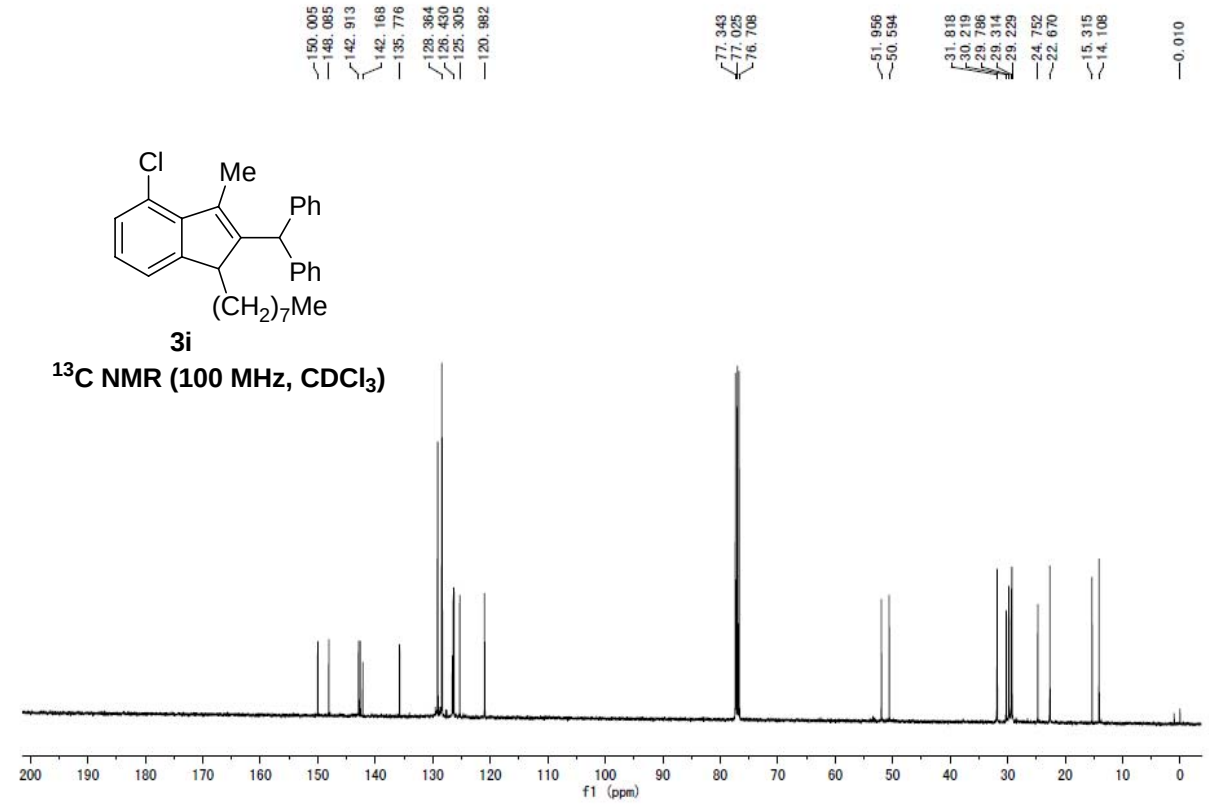
3h

^{13}C NMR (100 MHz, $CDCl_3$)



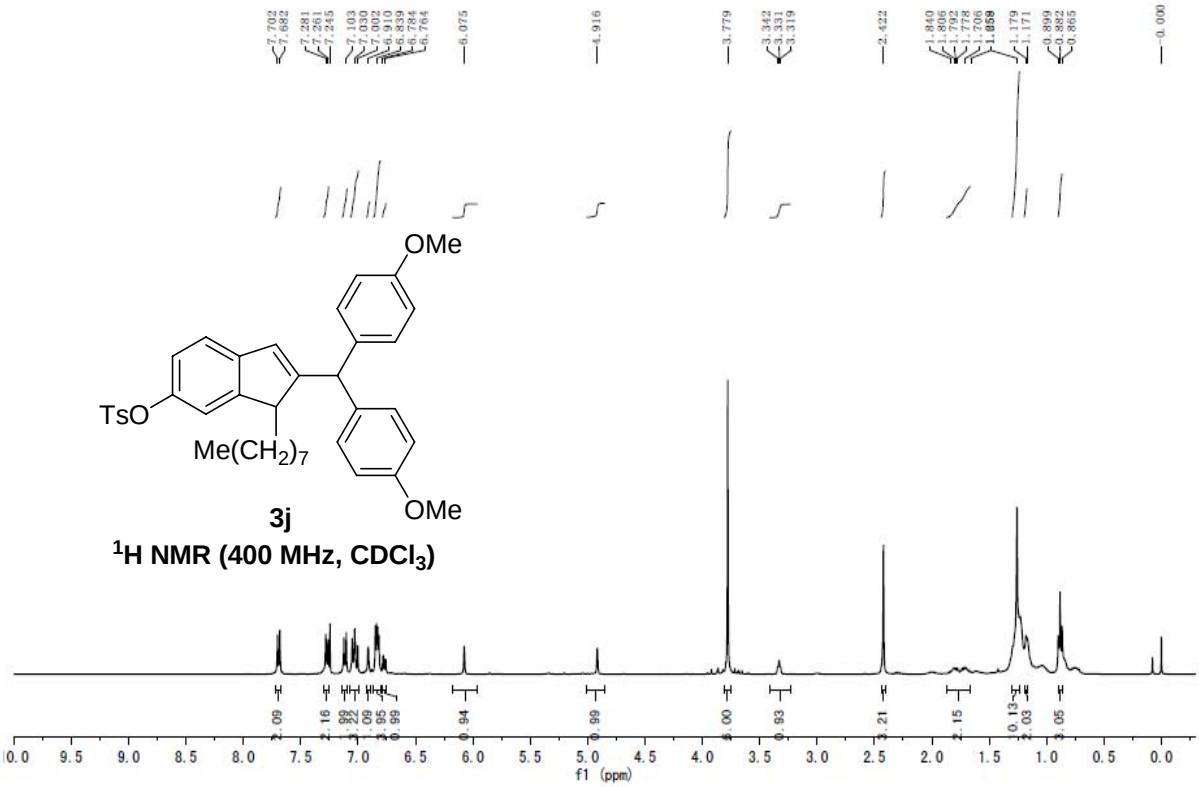
3i

^{1}H NMR (400 MHz, $CDCl_3$)



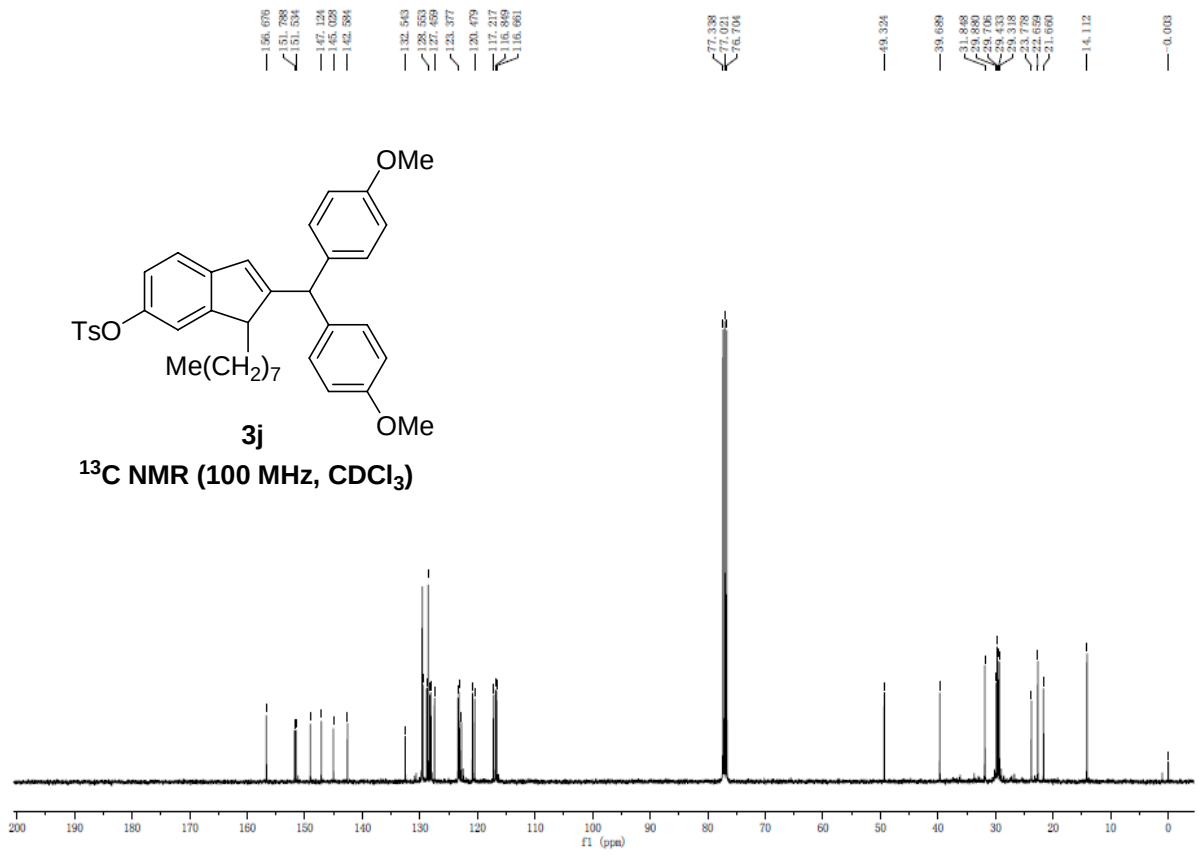
3i

^{13}C NMR (100 MHz, $CDCl_3$)



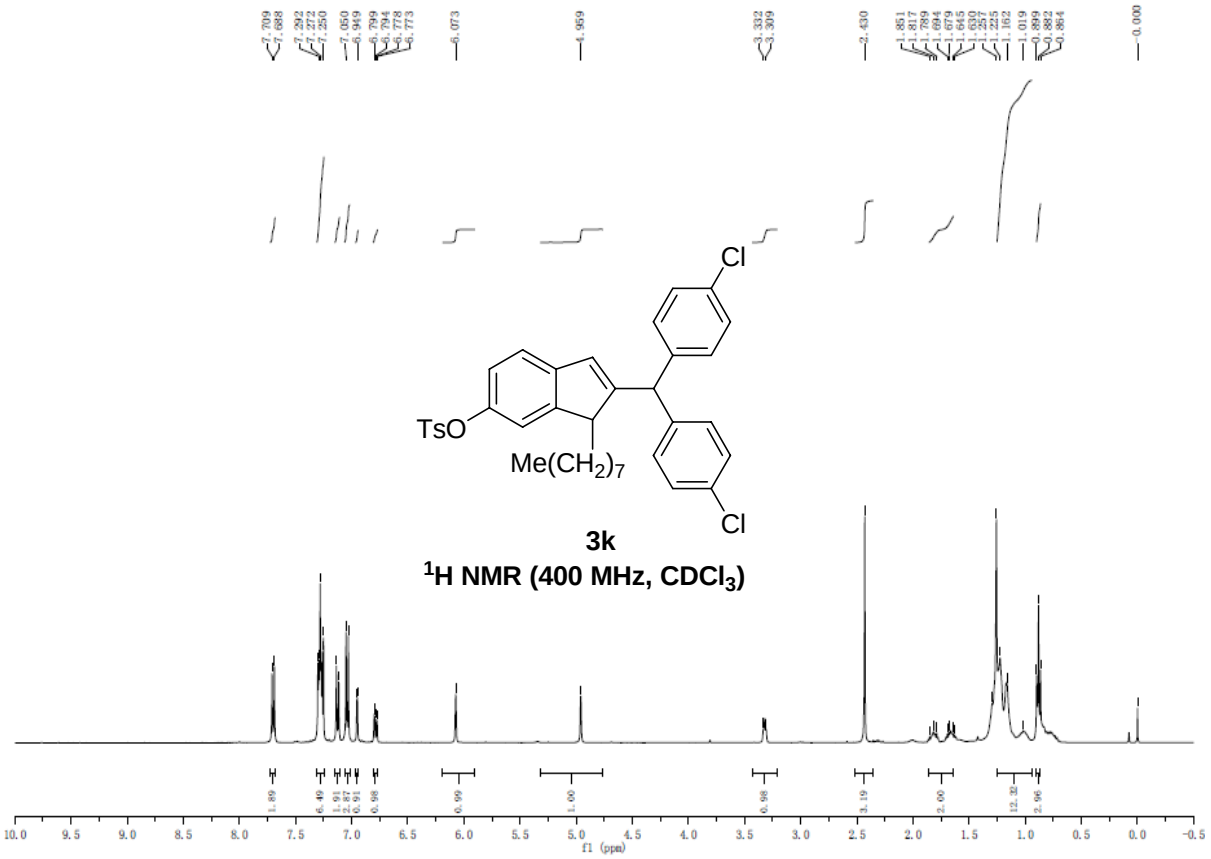
3j

^{1}H NMR (400 MHz, $CDCl_3$)



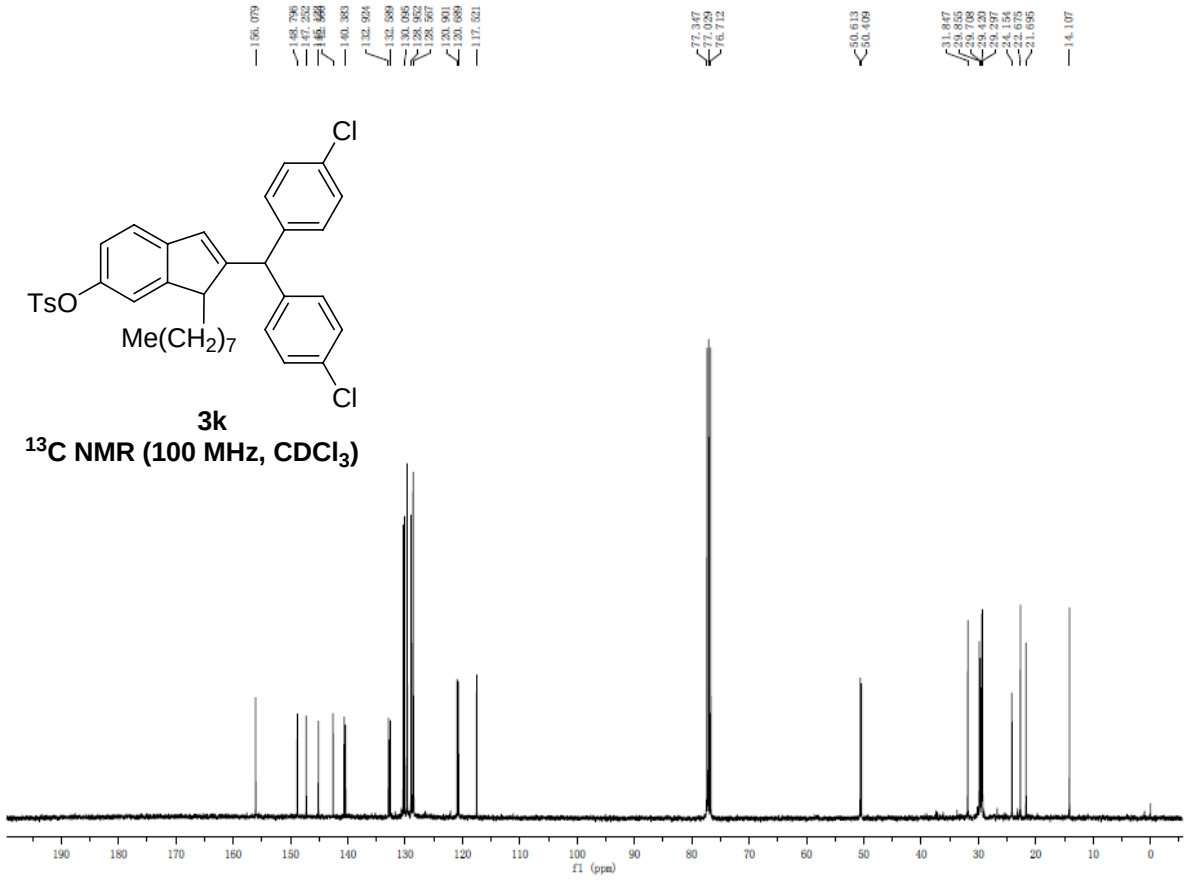
3j

^{13}C NMR (100 MHz, $CDCl_3$)



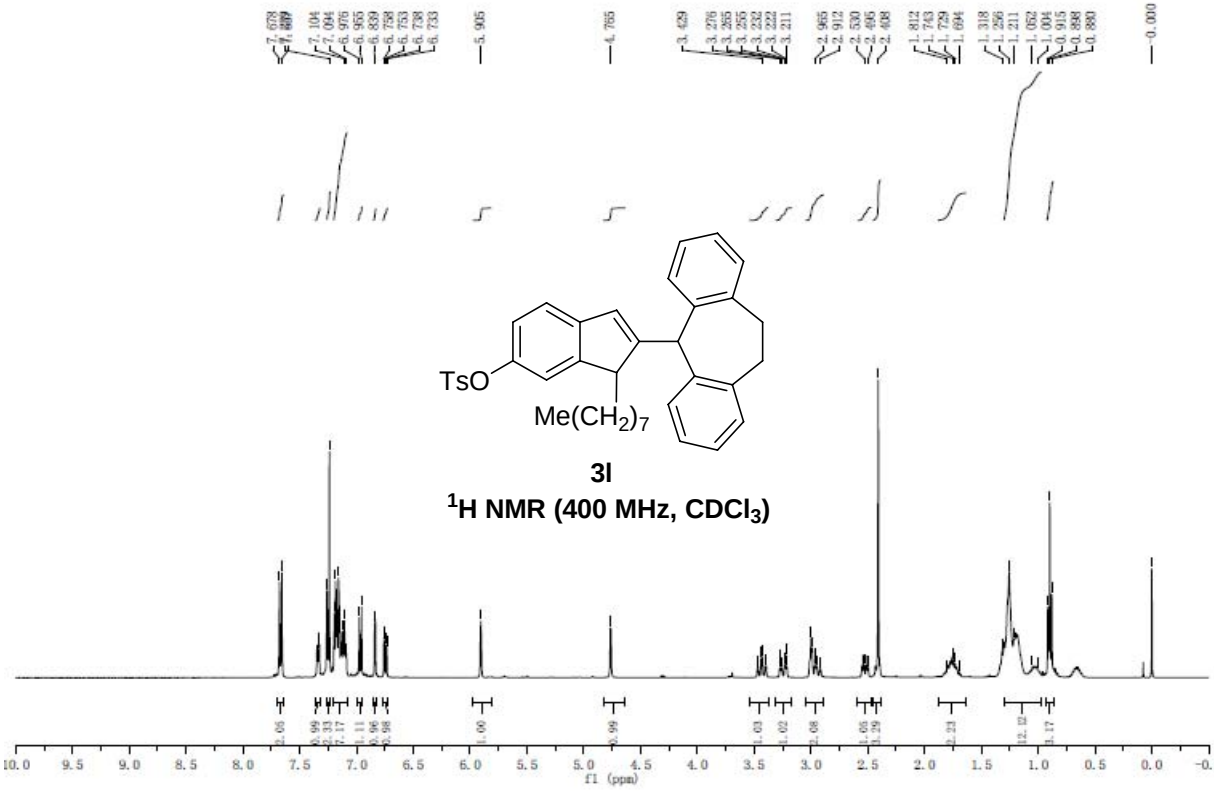
3k

^{1}H NMR (400 MHz, $CDCl_3$)



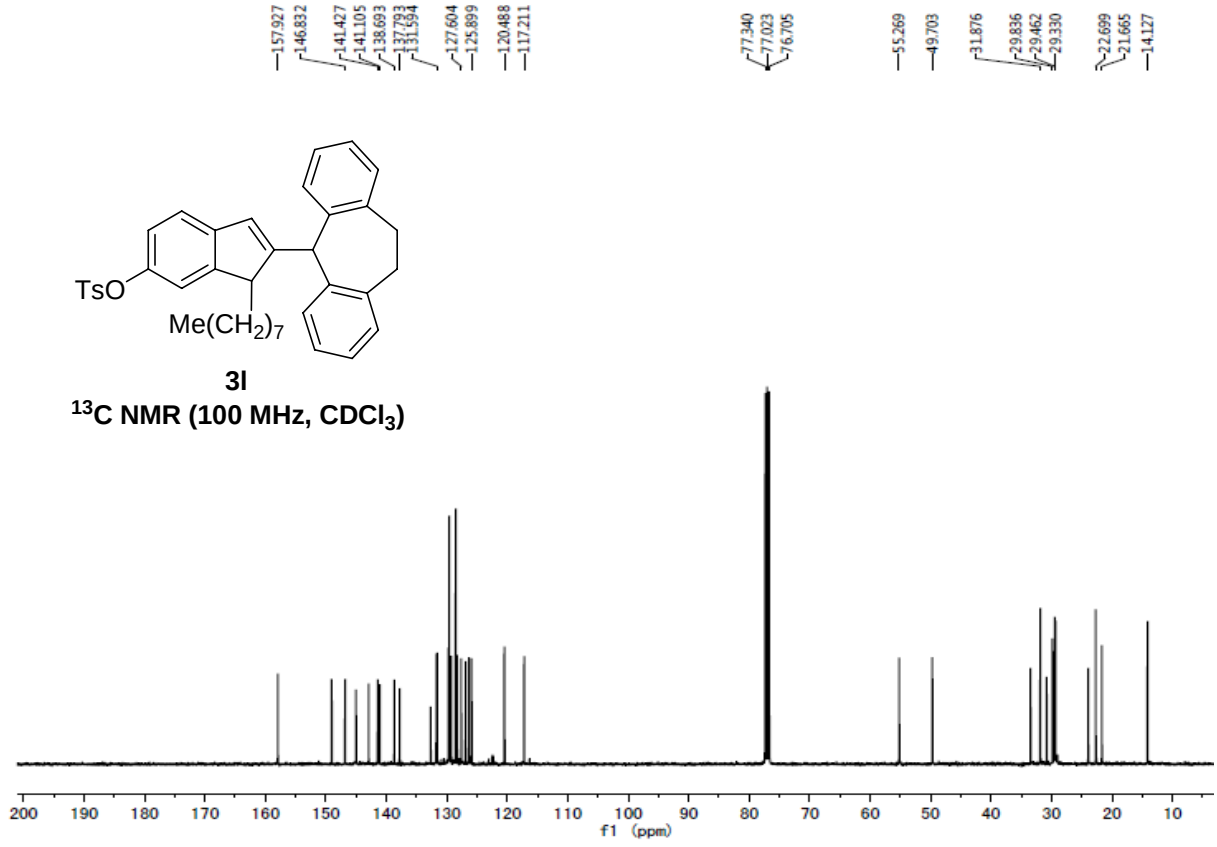
3k

^{13}C NMR (100 MHz, $CDCl_3$)



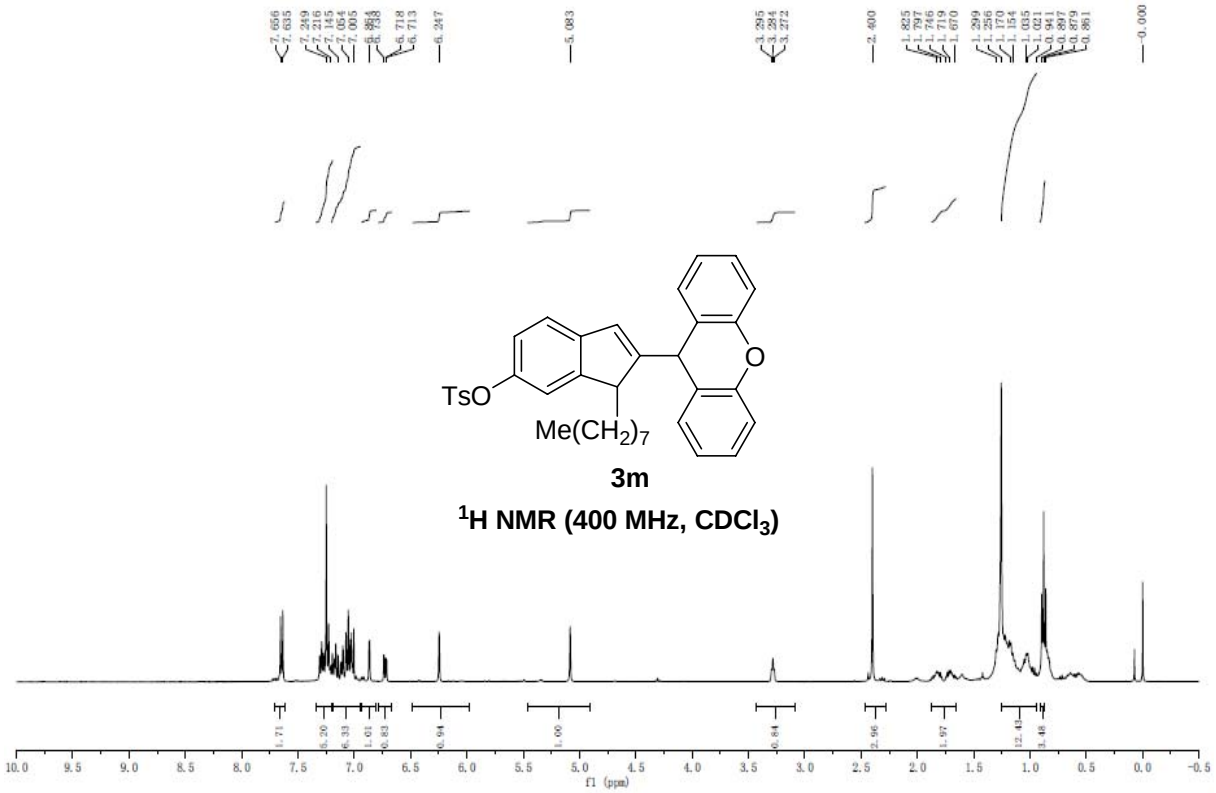
3l

^{1}H NMR (400 MHz, $CDCl_3$)



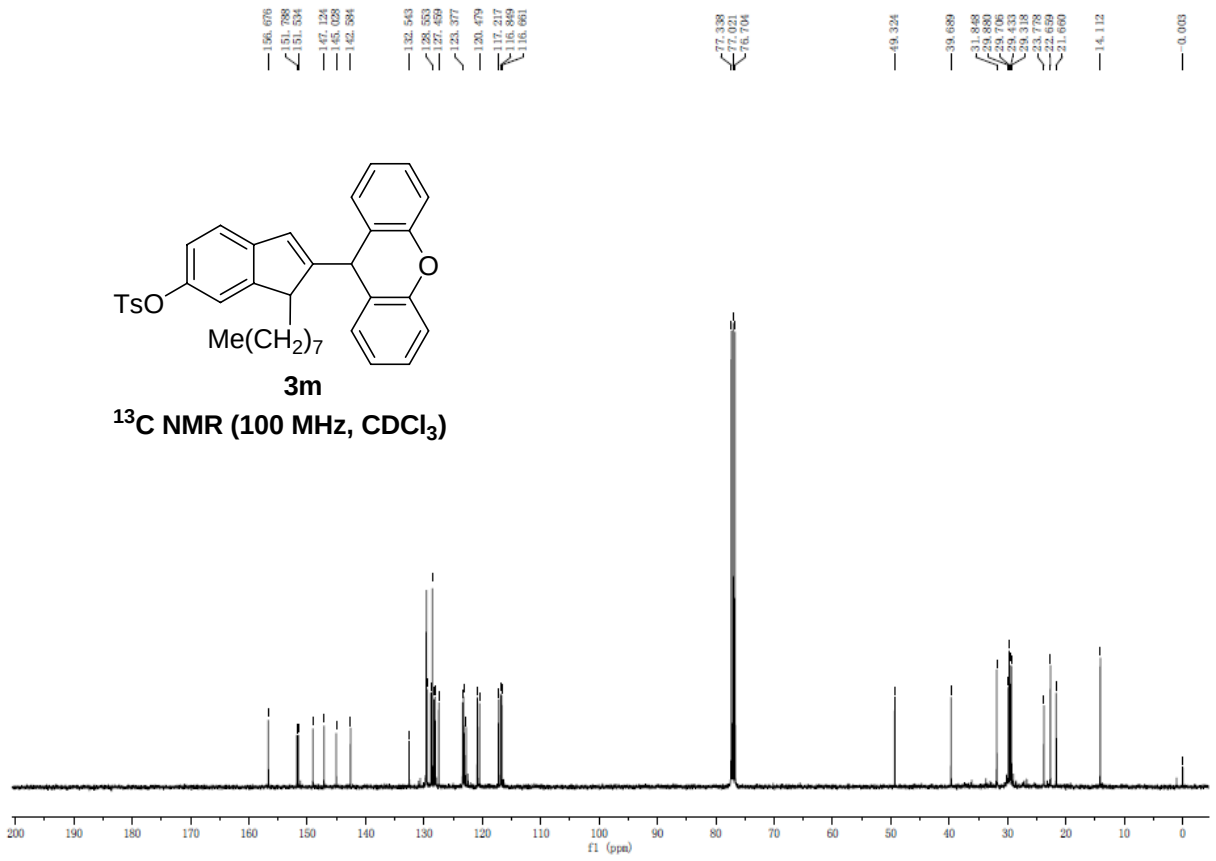
3l

^{13}C NMR (100 MHz, $CDCl_3$)



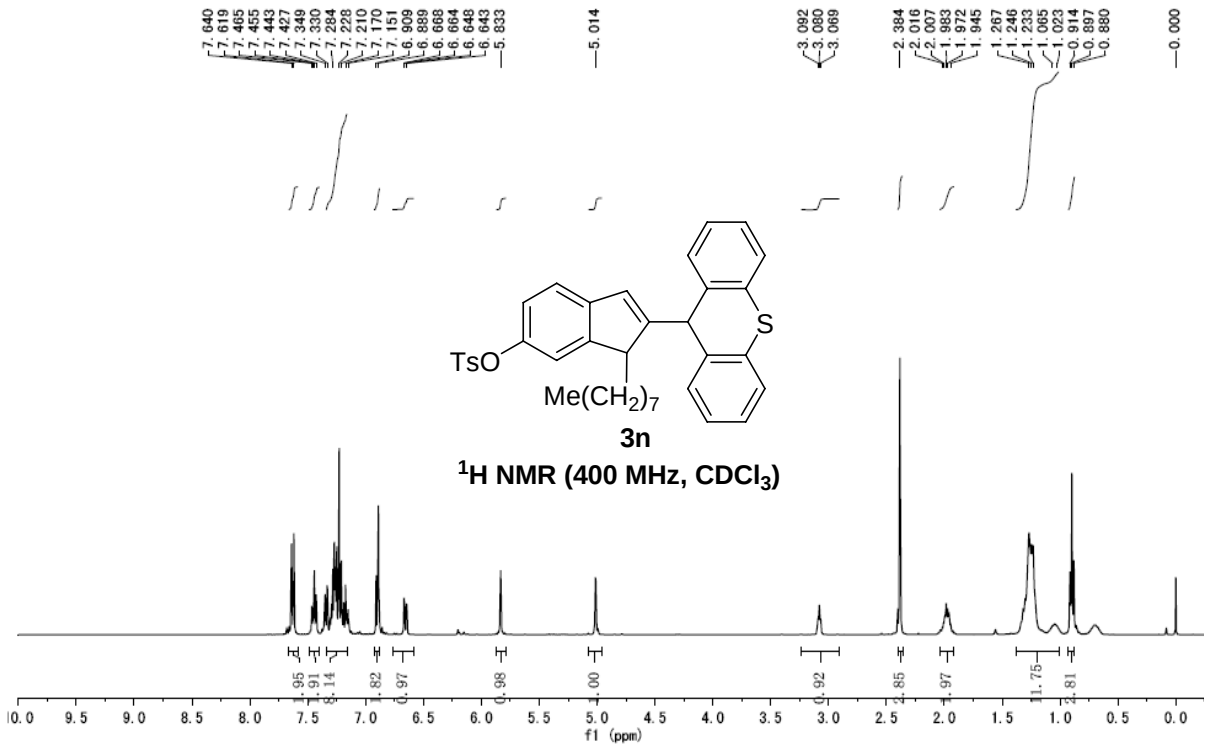
3m

^{1}H NMR (400 MHz, $CDCl_3$)



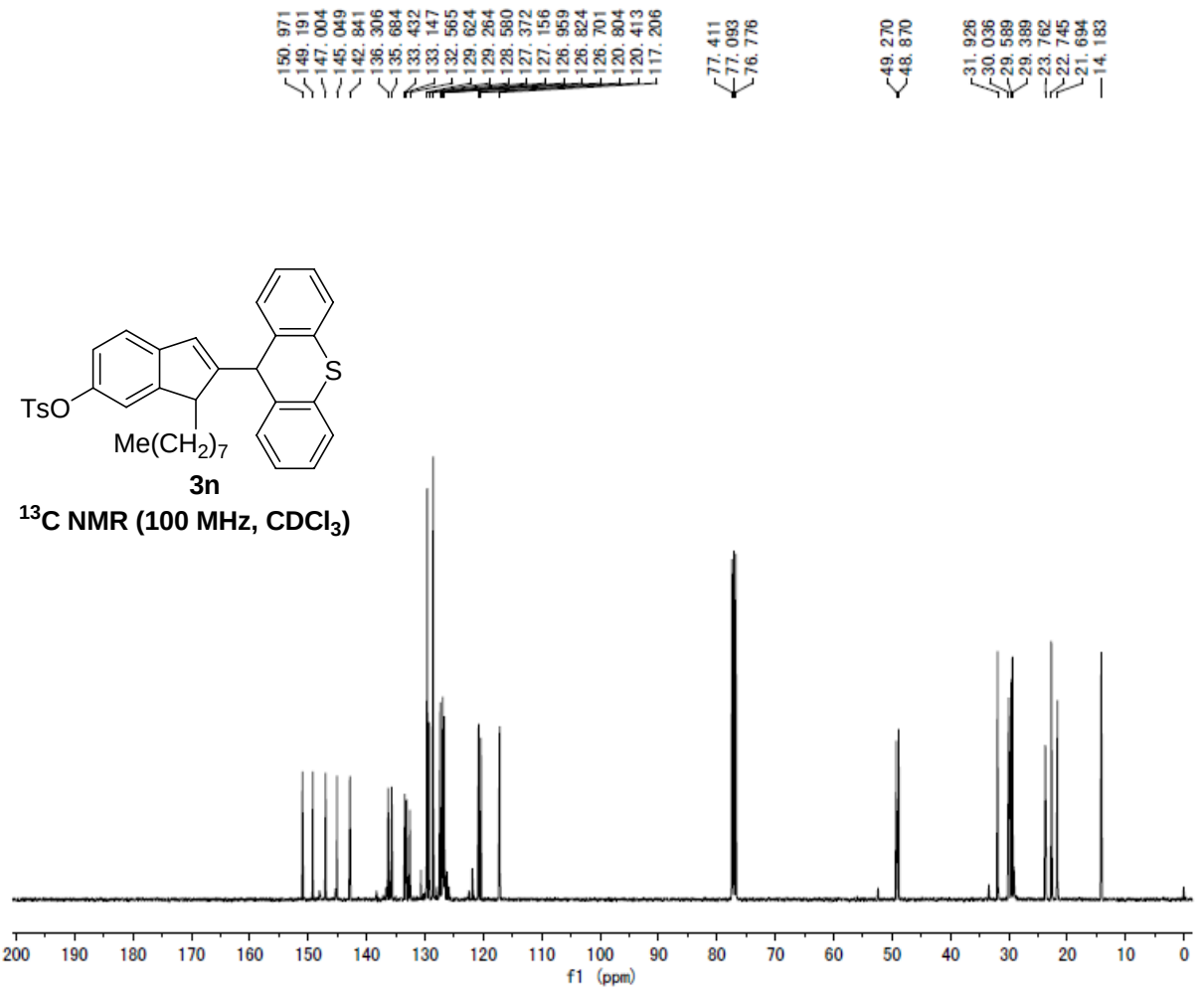
3m

^{13}C NMR (100 MHz, $CDCl_3$)



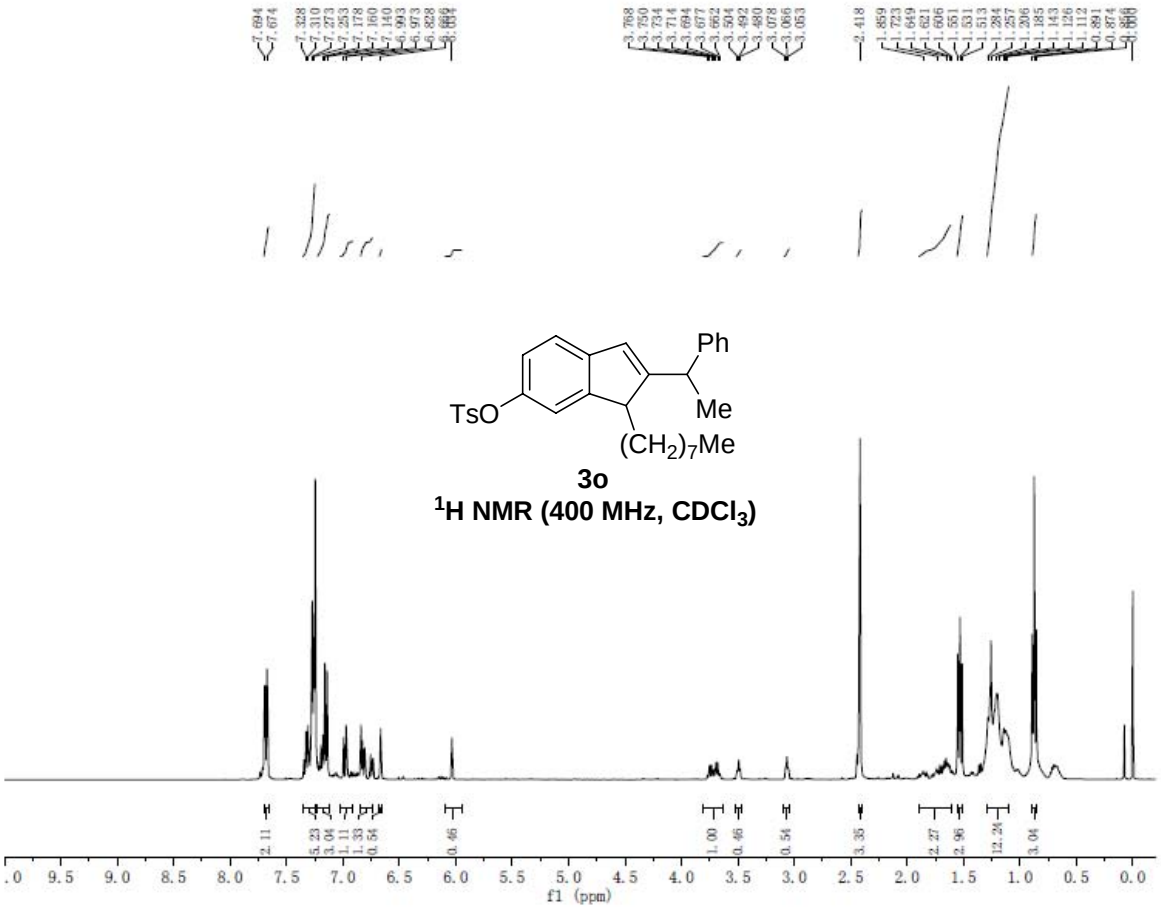
3n

^{1}H NMR (400 MHz, $CDCl_3$)



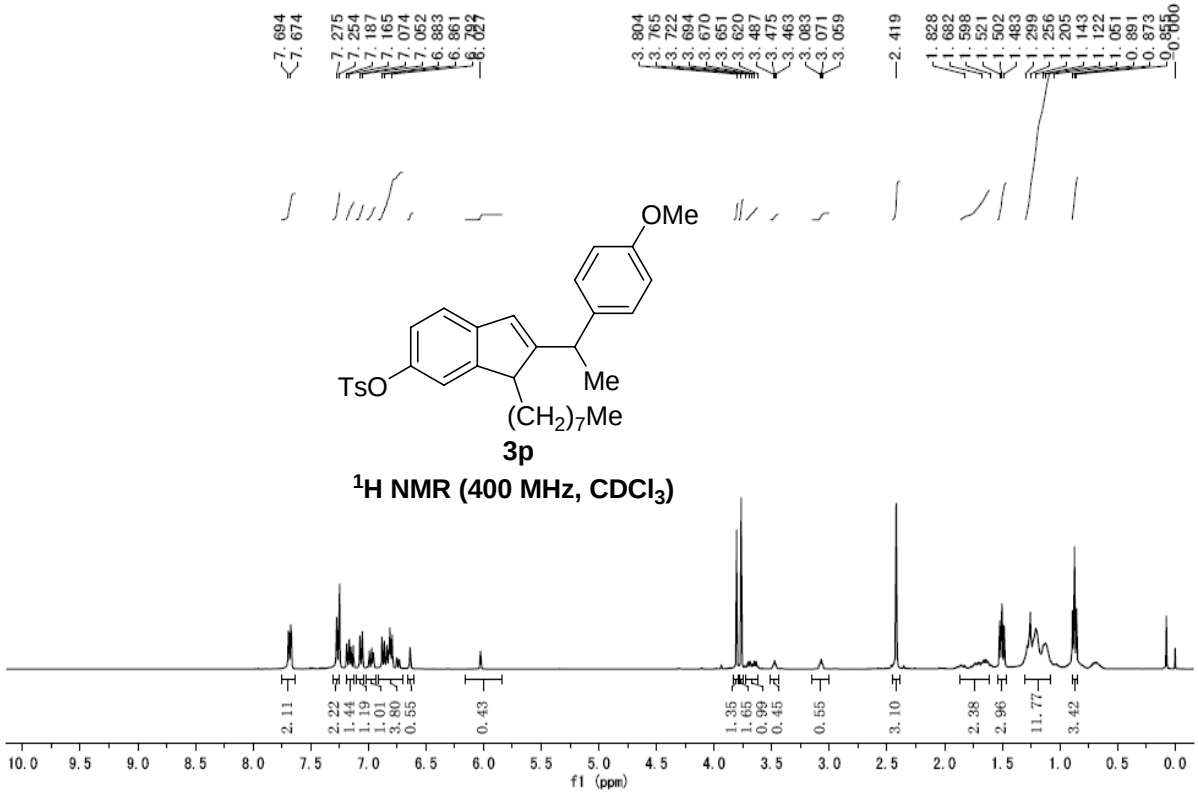
3n

^{13}C NMR (100 MHz, $CDCl_3$)



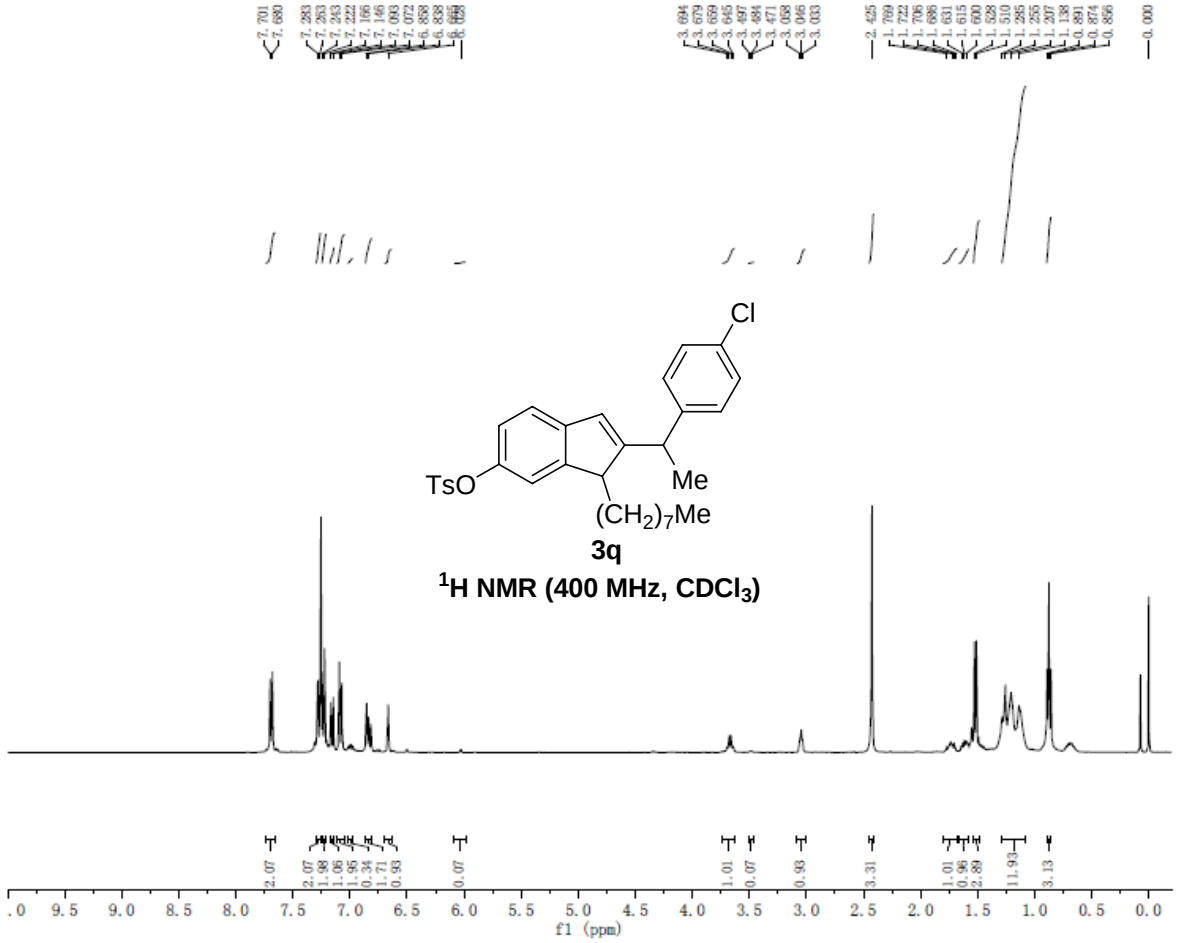
3o

^{1}H NMR (400 MHz, $CDCl_3$)



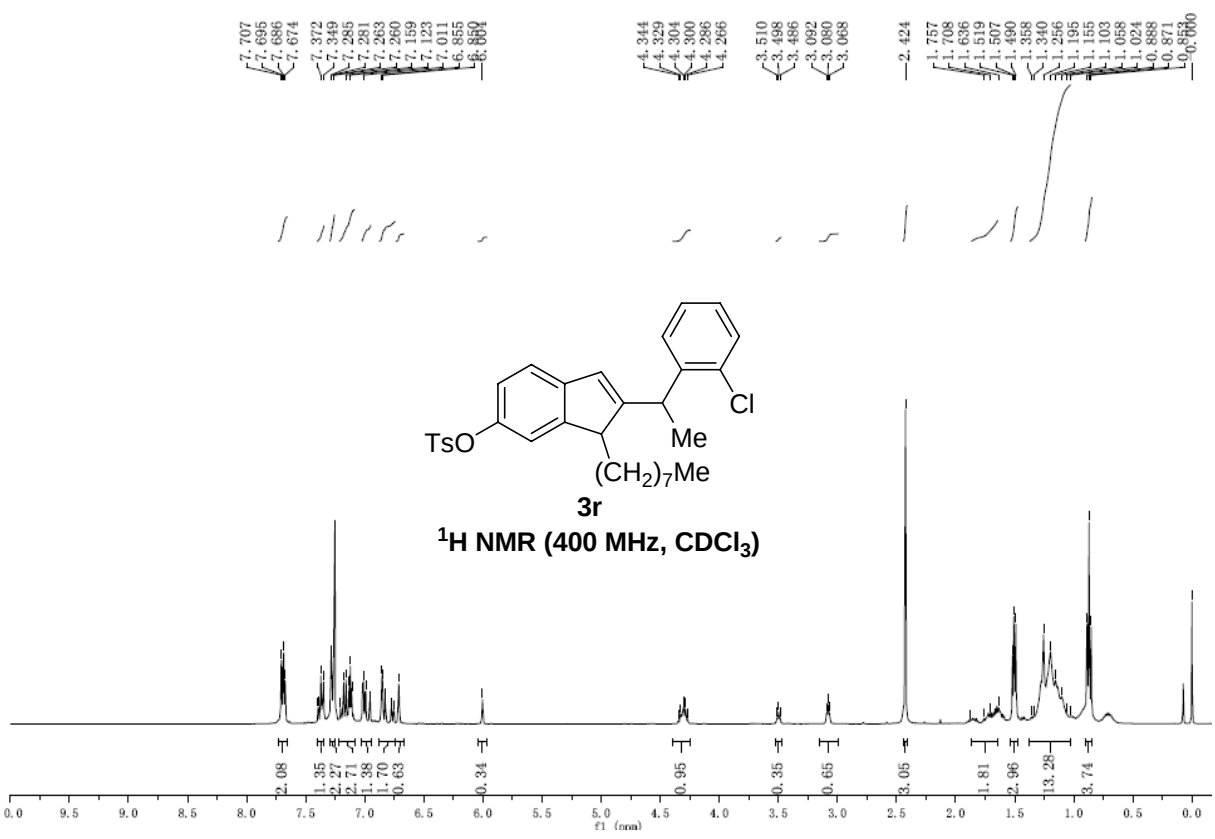
3p

^{1}H NMR (400 MHz, $CDCl_3$)



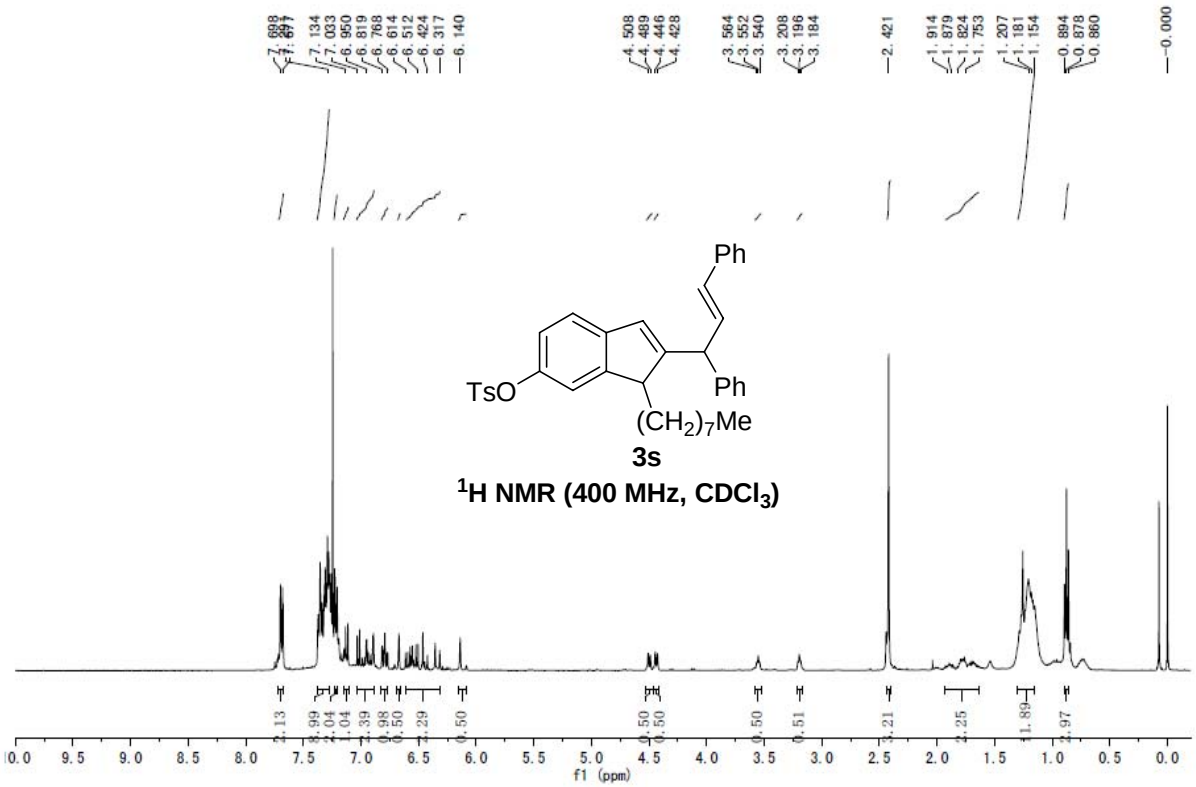
3q

^{1}H NMR (400 MHz, $CDCl_3$)



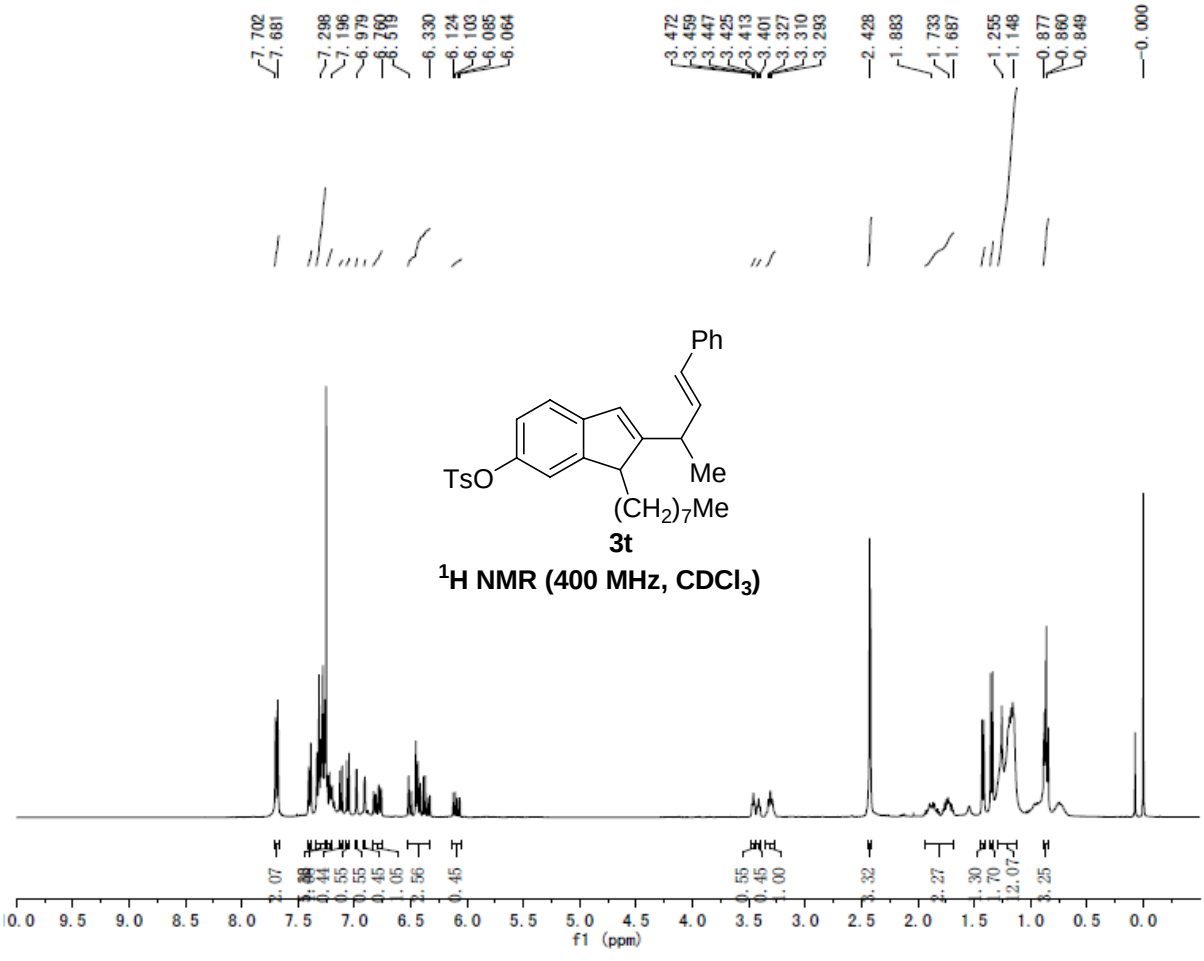
3r

^{1}H NMR (400 MHz, $CDCl_3$)



3s

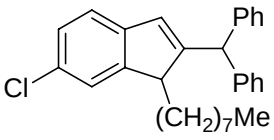
^{1}H NMR (400 MHz, $CDCl_3$)



3t

^{1}H NMR (400 MHz, $CDCl_3$)

The crystal of compound **3c** was obtained by leaving alone its solution in toluene at room temperature in the open air for several days. The crystal data of compound **3c** have been deposited in CCDC with number 889428.



3c

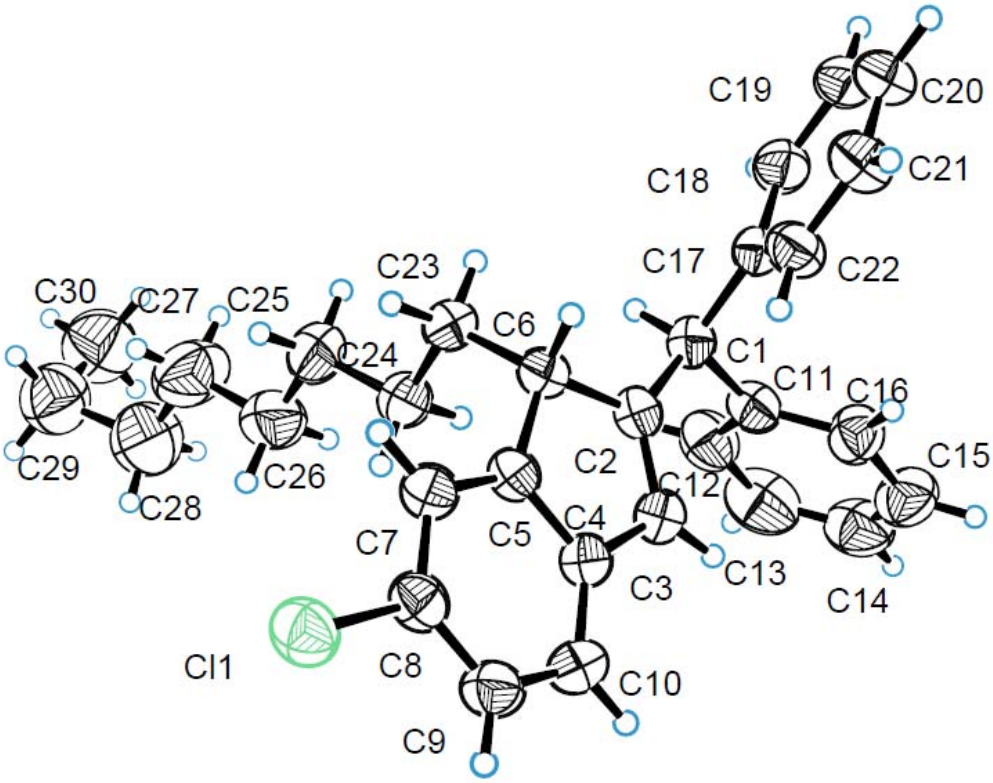


Table 1 Crystal data and structure refinement for 110321

Identification code	110321
Empirical formula	$C_{33}H_{40}Cl$
Formula weight	429.01
Temperature	291(2)
Crystal system	Triclinic
Space group	P-1
a/Å, b/Å, c/Å	8.1616 (4), 9.0676 (4), 17.2790 (8)
α/°, β/°, γ/°,	88.400 (4), 88.014 (4), 77.311 (4)
Volume/Å^3	1246.50 (10)
Z	2
ρ_{calc}mg/mm^3	1.143
m/mm^{-1}	1.437
F(000)	460
Crystal size	0.40 × 0.30 × 0.30
Theta range for data collection	5.00 to 69.58°
Index ranges	-9 ≤ h ≤ 9, -11 ≤ k ≤ 8, -20 ≤ l ≤ 18
Reflections collected	10255
Independent reflections	4574[R (int) = 0.0216]
Data/restraints/parameters	4574/51/287
Goodness-of-fit on F^2	1.053
Final R indexes [I>2σ (I)]	R_1 = 0.0665, wR_2 = 0.1331
Final R indexes [all data]	R_1 = 0.0795, wR_2 = 0.1403
Largest diff. peak/hole	0.341/-0.491

Table 2 Atomic Coordinates (Å×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 110321. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
Cl1	7440.2(11)	7087.1(9)	373(5)	81.5(3)

C6	4230(3)	3052(3)	1641.5(17)	60.9(7)
C2	5079(3)	1532(3)	1992.2(16)	59.7(7)
C23	2973(4)	4075(3)	2180.4(19)	68.5(8)
C1	4086(4)	451(3)	2348.7(17)	61.4(7)
C5	5712(3)	3700(3)	1373.3(16)	58.1(6)
C3	6750(4)	1347(3)	1930.6(18)	65.1(7)
C10	8753(4)	2986(4)	1356.2(19)	73.2(8)
C4	7205(3)	2661(3)	1549.9(17)	61.1(7)
C17	3122(3)	-228(3)	1757.4(17)	59(7)
C24	3687(4)	4409(4)	2935(2)	79.1(9)
C11	5191(4)	-754(3)	2845.7(18)	66(7)
C7	5768(4)	5063(3)	1007.8(16)	62.2(7)
C9	8809(4)	4359(4)	995.2(19)	71(8)
C18	1793(4)	-867(3)	2016(2)	72.5(8)
C22	3531(4)	-272(3)	984.6(18)	70.5(8)
C8	7327(4)	5369(3)	826(17)	64.3(7)
C20	1356(4)	-1551(4)	726(2)	84.1(10)
C16	6126(4)	-2070(4)	2533(2)	78.7(9)
C19	925(4)	-1525(4)	1496(2)	82.6(10)
C15	7206(5)	-3119(4)	2980(3)	95.7(11)
C21	2656(4)	-930(4)	467(2)	81.9(9)
C25	2426(5)	5455(4)	3441(2)	98.5(11)
	5350(5)	-530(4)	3620(2)	90.4(10)

C12				
C14	7355(6)	-2879(5)	3739(3)	108.6(14)
C26	3056(5)	5851(5)	4187(3)	118.3(14)
C13	6434(6)	-1592(6)	4069(2)	112.2(14)
C27	1789(6)	6859(7)	4709(3)	147(2)
C28'	2419(7)	7309(6)	5424(3)	148.2(15)
C29'	1409(14)	7640(16)	6201(6)	141.8(18)
C30'	-223(16)	8499(15)	5920(9)	162(2)
C28	2419(7)	7309(6)	5424(3)	148.2(15)
C29	1219(13)	8591(10)	5882(6)	141.8(18)
C30	288(16)	7713(14)	6386(7)	162(2)

Table 3 Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for 110321. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+...+2hka \times b \times U_{12}]$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Cl1	84.9(6)	78.1(5)	85.2(6)	7.1(4)	0.2(4)	-27.3(4)
C6	56.6(15)	59.1(16)	68.3(18)	-6.3(13)	-9.5(13)	-13.7(12)
C2	63.2(17)	53.1(15)	63.5(17)	-9.8(12)	-3.9(13)	-13(12)
C23	53.6(16)	62.1(17)	88(2)	-4.1(15)	-1.3(14)	-7.9(13)
C1	61.2(16)	56.9(16)	65.7(17)	-8.4(13)	2.2(13)	-12(13)
C5	56(15)	58.4(16)	61.1(16)	-9.7(13)	-5.5(12)	-13.5(12)
C3	57.7(16)	56(16)	79(2)	-5.3(14)	-3.6(14)	-6.7(12)
C10	53(16)	73(2)	91(2)	-2(17)	-3.7(15)	-7.8(14)
C4	55.7(16)	59.4(16)	67.6(18)	-9.6(13)	-3.6(13)	-9.8(12)
C17	52.6(15)	51.1(15)	72.9(18)	-3.9(13)	-2.1(13)	-9.6(12)
C24	68.7(19)	79(2)	89(2)	-22.8(18)	7.8(17)	-12.9(16)
C11	68.4(18)	67.6(18)	66.4(18)	-0.6(14)	-5.1(14)	-24.3(14)
C7	58.9(16)	62.2(17)	65.6(17)	-4.7(13)	-7.8(13)	-11.9(13)
C9	56.8(17)	78(2)	81(2)	-5.4(16)	2(15)	-20.2(15)
C18	64.3(18)	69.8(19)	84(2)	-3(16)	4.3(15)	-16(15)
C22	67.3(18)	74(19)	76(2)	-8.2(16)	0.3(15)	-27.7(15)
C8	67.2(18)	67.7(18)	60.9(17)	-8.3(14)	-1.9(14)	-20.2(14)
C20	74(2)	81(2)	103(3)	-11(2)	-20(2)	-25.4(17)
C16	83(2)	70(2)	80(2)	-0.1(17)	-9.8(17)	-10.2(16)
C19	58.8(18)	76(2)	118(3)	-6(2)	-2.5(19)	-25.4(16)
C15	86(3)	81(2)	117(3)	15(2)	-15(2)	-10.3(19)
C21	85(2)	87(2)	80(2)	-12.9(18)	-7.4(18)	-30(19)
C25	83(2)	87(2)	121(3)	-12(2)	6(2)	-6.6(19)
C12	106(3)	94(3)	75(2)	-4.8(19)	-11(2)	-29(2)
C14	99(3)	108(3)	124(4)	36(3)	-38(3)	-32(3)
C26	87(3)	132(4)	138(3)	-32(3)	7(2)	-27(2)
C13	136(4)	134(4)	77(3)	16(3)	-36(3)	-48(3)
C27	105(3)	178(5)	146(4)	-48(3)	2(3)	-2(3)
C28'	151(2)	151(2)	141.3(18)	-21.4(14)	-5.2(13)	-28.5(13)
C29'	145(2)	141(2)	139(2)	-10.9(13)	-3.5(14)	-27.9(14)
C30'	159(2)	164(3)	161(3)	0.8(14)	3.4(14)	-34.7(15)
C28	151(2)	151(2)	141.3(18)	-21.4(14)	-5.2(13)	-28.5(13)
C29	145(2)	141(2)	139(2)	-10.9(13)	-3.5(14)	-27.9(14)
C30	159(2)	164(3)	161(3)	0.8(14)	3.4(14)	-34.7(15)

Table 4 Bond Lengths for 110321.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Cl1	C8	1.743(3)	C11	C16	1.380(4)
C6	C5	1.511(4)	C7	C8	1.385(4)
C6	C2	1.520(4)	C9	C8	1.382(4)
C6	C23	1.531(4)	C18	C19	1.388(4)
C2	C3	1.338(4)	C22	C21	1.388(4)
C2	C1	1.509(4)	C20	C21	1.363(5)
C23	C24	1.511(4)	C20	C19	1.365(5)
C1	C11	1.521(4)	C16	C15	1.384(5)
C1	C17	1.532(4)	C15	C14	1.349(6)
C5	C7	1.381(4)	C25	C26	1.486(6)
C5	C4	1.404(4)	C12	C13	1.394(5)
C3	C4	1.458(4)	C14	C13	1.369(6)
C10	C9	1.386(4)	C26	C27	1.512(6)
C10	C4	1.387(4)	C27	C28'	1.456(7)
C17	C22	1.365(4)	C28'	C29'	1.551(8)
C17	C18	1.392(4)	C29'	C30'	1.478(8)
C24	C25	1.509(4)	C29	C30	1.467(8)
C11	C12	1.374(4)			

Table 5 Bond Angles for 110321.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C5	C6	C2	102.3(2)	C12	C11	C1	120.6(3)
C5	C6	C23	114.4(2)	C16	C11	C1	121.7(3)
C2	C6	C23	115.3(2)	C5	C7	C8	118.2(3)
C3	C2	C1	127.8(3)	C8	C9	C10	119.6(3)
C3	C2	C6	110.2(2)	C19	C18	C17	120.3(3)
C1	C2	C6	122.0(2)	C17	C22	C21	121.4(3)

C24	C23	C6	114.6(2)	C9	C8	C7	122
C2	C1	C11	111.0(2)	C9	C8	Cl1	118
C2	C1	C17	113.5(2)	C7	C8	Cl1	119
C11	C1	C17	112.3(2)	C21	C20	C19	119
C7	C5	C4	120.4(3)	C11	C16	C15	121
C7	C5	C6	130.5(3)	C20	C19	C18	120
C4	C5	C6	109.2(2)	C14	C15	C16	120
C2	C3	C4	110.6(3)	C20	C21	C22	120
C9	C10	C4	119.2(3)	C26	C25	C24	115
C10	C4	C5	120.5(3)	C11	C12	C13	120
C10	C4	C3	131.7(3)	C15	C14	C13	119
C5	C4	C3	107.8(2)	C25	C26	C27	116
C22	C17	C18	118.1(3)	C14	C13	C12	120
C22	C17	C1	122.9(3)	C28'	C27	C26	116
C18	C17	C1	119.0(3)	C27	C28'	C29'	126
C25	C24	C23	113.0(3)	C30'	C29'	C28'	100
C12	C11	C16	117.7(3)				

Table 6 Torsion Angles for 110321.

A	B	C	D	Angle/°
C5	C6	C2	C3	-0.3(3)
C23	C6	C2	C3	-125.0(3)
C5	C6	C2	C1	179.7(2)
C23	C6	C2	C1	55.0(3)
C5	C6	C23	C24	-62.0(3)
C2	C6	C23	C24	56.2(3)
C3	C2	C1	C11	15.7(4)
C6	C2	C1	C11	-164.2(2)
C3	C2	C1	C17	-111.9(3)
C6	C2	C1	C17	68.1(3)
C2	C6	C5	C7	-179.9(3)
C23	C6	C5	C7	-54.6(4)
C2	C6	C5	C4	0.3(3)
C23	C6	C5	C4	125.6(3)
C1	C2	C3	C4	-179.8(3)
C6	C2	C3	C4	0.1(3)
C9	C10	C4	C5	0.9(5)
C9	C10	C4	C3	-179.4(3)
C7	C5	C4	C10	-0.3(4)
C6	C5	C4	C10	179.5(3)
C7	C5	C4	C3	179.9(3)
C6	C5	C4	C3	-0.3(3)
C2	C3	C4	C10	-179.6(3)
C2	C3	C4	C5	0.1(3)
C2	C1	C17	C22	20.6(4)
C11	C1	C17	C22	-106.3(3)
C2	C1	C17	C18	-161.1(3)
C11	C1	C17	C18	72.0(3)
C6	C23	C24	C25	178.4(3)
C2	C1	C11	C12	90.4(3)
C17	C1	C11	C12	-141.4(3)
C2	C1	C11	C16	-86.5(3)
C17	C1	C11	C16	41.7(4)
C4	C5	C7	C8	-0.3(4)
C6	C5	C7	C8	180.0(3)
C4	C10	C9	C8	-0.8(5)
C22	C17	C18	C19	0.0(4)
C1	C17	C18	C19	-178.4(3)
C18	C17	C22	C21	0.1(5)
C1	C17	C22	C21	178.4(3)
C10	C9	C8	C7	0.3(5)
C10	C9	C8	Cl1	179.9(2)
C5	C7	C8	C9	0.3(4)
C5	C7	C8	Cl1	-179.4(2)
C12	C11	C16	C15	-0.9(5)
C1	C11	C16	C15	176.1(3)
C21	C20	C19	C18	0.3(5)
C17	C18	C19	C20	-0.2(5)
C11	C16	C15	C14	0.6(6)
C19	C20	C21	C22	-0.2(5)
C17	C22	C21	C20	0.0(5)
C23	C24	C25	C26	-179.0(3)
C16	C11	C12	C13	0.7(5)
C1	C11	C12	C13	-176.3(3)
C16	C15	C14	C13	0.0(6)
C24	C25	C26	C27	-178.2(4)
C15	C14	C13	C12	-0.2(7)
C11	C12	C13	C14	-0.1(6)
C25	C26	C27	C28'	-177.1(5)
C26	C27	C28'	C29'	-149.1(8)
C27	C28'	C29'	C30'	-41.5(14)

Table 7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for 110321.

Atom	*x*	*y*	*z*	U(eq)
H6	3635	2867	1183	73
H23B	2540	5024	1909	82
H23A	2035	3600	2296	82
H1	3240	1045	2700	74
H3	7515	497	2106	78
H10	9741	2291	1467	88
H24A	4088	3466	3217	95
H24B	4643	4863	2823	95
H7	4785	5756	887	75
H9	9837	4599	868	85
H18	1487	-853	2540	87
H22	4416	148	801	85
H20	766	-1989	379	101
H16	6029	-2255	2012	94
H19	41	-1952	1674	99
H15	7832	-3993	2756	115
H21	2958	-947	-57	98
H25B	2011	6383	3149	118
H25A	1481	4988	3552	118
H12	4726	341	3848	109
H14	8082	-3587	4037	130
H26B	3979	6346	4074	142
H26A	3506	4920	4471	142
H13	6532	-1427	4592	135
H27B	1297	7767	4417	176
H27A	894	6340	4844	176
H28A	2828	8214	5292	178
H28B	3401	6528	5543	178
H29A	1298	6715	6472	170
H29B	1920	8242	6536	170
H30A	-1001	8741	6351	242
H30B	-656	7898	5563	242
H30C	-73	9415	5664	242
H28C	3455	7634	5300	178
H28D	2695	6425	5763	178
H29C	1842	9162	6180	170
H29D	478	9275	5539	170
H30D	-531	8389	6695	242
H30E	1055	7051	6718	242
H30F	-269	7124	6074	242