

Supporting Information

HFIP-promoted strain-release rearrangement/*N*-functionalization of azabicyclo[1.1.0]butanes with *para*-quinone methides for the synthesis of azetidines

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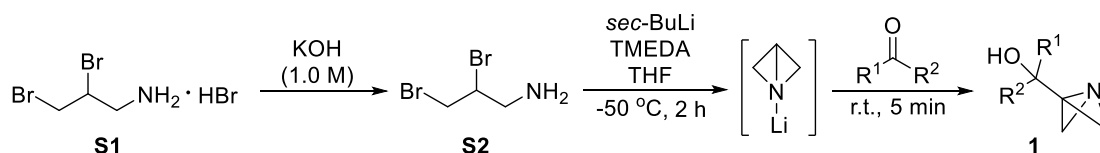
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General information ^1H and ^{13}C NMR spectra were recorded on a Bruker Avance 400 instrument at 400 (^1H NMR), 100 (^{13}C NMR) as well as 376 MHz (^{19}F NMR). Tetramethylsilane (TMS), CDCl_3 (7.26 ppm for ^1H NMR, 77.0 ppm for ^{13}C NMR), $\text{DMSO-}d_6$ (2.50 ppm for ^1H NMR, 39.5 ppm for ^{13}C NMR) or $\text{THF-}d_8$ (1.72 ppm and 3.58 ppm for ^1H NMR, 67.2 ppm and 25.3 ppm for ^{13}C NMR) were used as references. Data for ^1H NMR were reported as follows: chemical shift (ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, dt = doublet of triplets, m = multiplet), coupling constants (Hz), and integration. Data for ^{13}C NMR were reported as ppm. High-resolution mass spectra (HRMS) analyses were performed by the electrospray ionization (ESI) method on a Waters SYNAPT G2-Si mass spectrometer with a Q-TOF analyzer. Melting points were determined using a X-4 digital micro melting point apparatus. Thin-layer chromatography (TLC) was performed, and visualization of the compounds was accomplished with UV light (254 nm). Flash column chromatography was performed on basic aluminum oxide or silica gel (200–300 mesh). Known ABBs **1**¹ and *p*-QMs **2**² were prepared according to literature procedures. Other purchased reagents and solvents were used without further purification, if not stated otherwise.

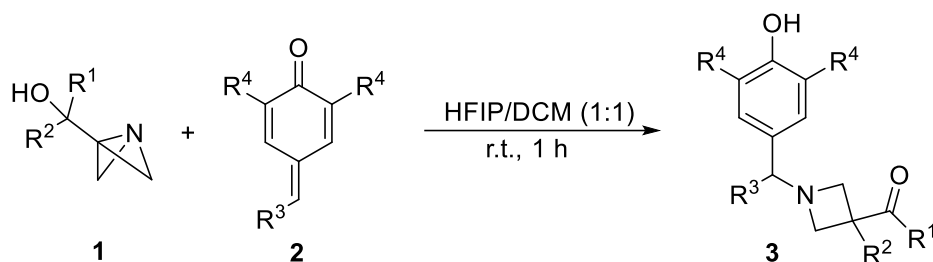
General procedure for preparation of ABBs **1**



2,3-Dibromopropan-1-amine hydrobromide **7** (1.5 g, 5.0 mmol, 1.0 eq.) was added to a 50 mL separatory funnel containing aq. KOH (1.0 M, 10.0 mL, 10.0 mmol, 2.0 eq.) and DCM (10.0 mL). After separation of the organic layer, the aqueous layer was further extracted with DCM (3 × 10 mL). The combined organic phase was dried over anhydrous Na_2SO_4 , filtered into a 100 mL round-bottom flask, and concentrated under reduced pressure to afford the free amine **8**. The flask was evacuated and refilled with argon using a balloon. Anhydrous THF (12.5 mL) was added to dissolve the residue, followed by the addition of anhydrous TMEDA (1.9 g, 16.0 mmol, 3.2 eq.). The reaction mixture was cooled to -50 °C. *Sec*-butyl lithium (1.3 M in cyclohexane, 12.3 mL, 16.0 mmol, 3.2 eq.) was added dropwise, then the solution was stirred at the same temperature. After 2 h, respective carbonyl derivative (10.0 mmol, 2.0 eq.) in anhydrous THF (5.0 mL) was

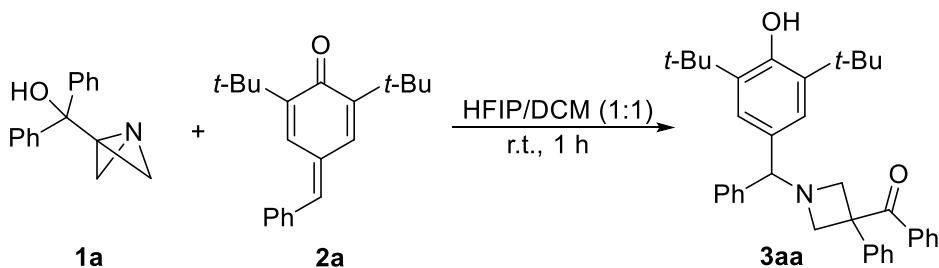
added and the resulting solution was stirred for 5 minutes out of the cooling bath. The reaction mixture was quenched with water and extracted with ethyl acetate (3 × 10 mL). The combined organic layer was collected, dried over anhydrous Na₂SO₄, filtered and evaporated under reduced pressure. The residue was purified using silica gel column chromatography to give the ABBs **1**.

General procedure for preparation of products **3**



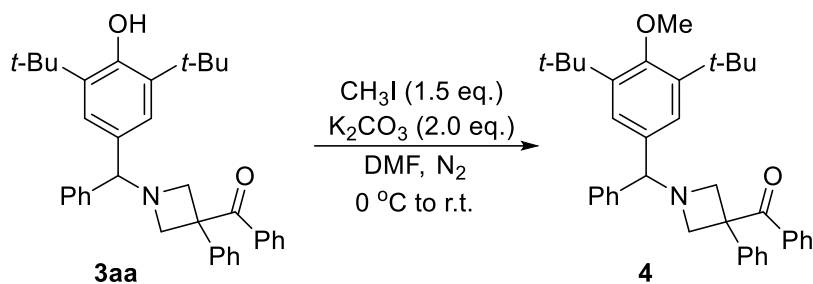
p-QMs **2** (0.24 mmol, 1.2 eq.) was added to a mixture of ABBs **1** (0.2 mmol, 1.0 eq.) and HFIP/DCM (1.0 mL/1.0 mL). The resulting mixture was stirred for 1 h at room temperature. Then, the solvent was removed in vacuo on a rotary evaporator and the residue was purified by basic aluminum oxide column chromatography to give the pure products **3**.

Procedure for scale-up synthesis of compound **3aa**



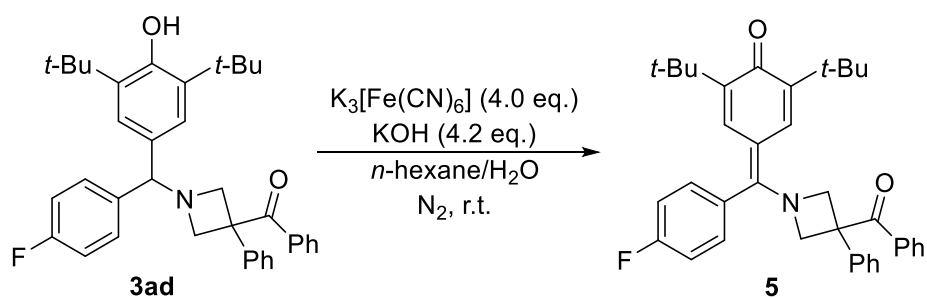
p-QM **2a** (1.8 g, 6.0 mmol, 1.2 eq.) was added to a mixture of ABB **1a** (1.2 g, 5.0 mmol, 1.0 eq.) and HFIP/DCM (25.0 mL/25.0 mL). The resulting mixture was stirred for 1 h at room temperature. After evaporating the solvent under reduced pressure, the residue was purified by basic aluminum oxide column chromatography (PE/EA = 20/1) to give the pure products **3aa** (2.1 g, 80% yield) as a white solid.

Procedure for preparation of compound **4**



Under nitrogen atmosphere, anhydrous DMF (2.0 mL) was added to the mixture of **3aa** (106.3 mg, 0.2 mmol, 1.0 eq.) and K_2CO_3 (55.3 mg, 0.4 mmol, 2.0 eq.). The mixture was stirred at 0 °C for 0.5 h. Then, MeI (42.6 mg, 0.3 mmol, 1.5 eq.) was added to the reaction mixture. The resulting mixture was stirred for 12 h at room temperature. After the completion of the reaction which was indicated by TLC, saturated aqueous NH_4Cl was added to the reaction mixture, and the aqueous layer was extracted with ethyl acetate (3×2 mL). The combined organic layer was dried over anhydrous Na_2SO_4 , filtered and concentrated under the reduced pressure. The residue was purified by silica gel column chromatography (PE/EA = 25/1) to afford the product **4** (101.5 mg, 93% yield) as a white solid.

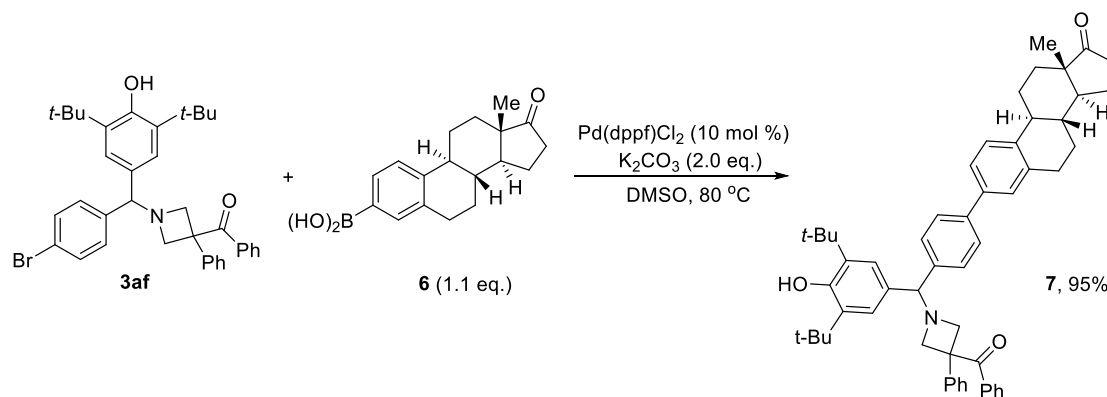
Procedure for preparation of compound 5



To a solution of **3ad** (109.9 mg, 0.2 mmol, 1.0 eq.) in hexane (2.0 mL) was added a solution of $K_3[Fe(CN)_6]$ (263.4 mg, 0.8 mmol, 4.0 eq.) and KOH (47.1 mg, 0.84 mmol, 4.2 eq.) in water (2.0 mL) under nitrogen atmosphere. The reaction mixture was vigorously stirred at room temperature for 12 h. The aqueous layer was separated and extracted with ethyl acetate (3×2 mL). The combined organic layer was dried over anhydrous Na_2SO_4 , filtered and concentrated under the

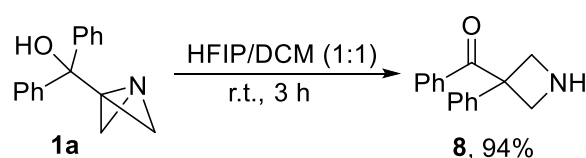
reduced pressure. The residue was purified by silica gel column chromatography (PE/EA = 3/1) to afford the product **5** (97.5 mg, 89% yield) as a yellow solid.

Procedure for preparation of compound **7**

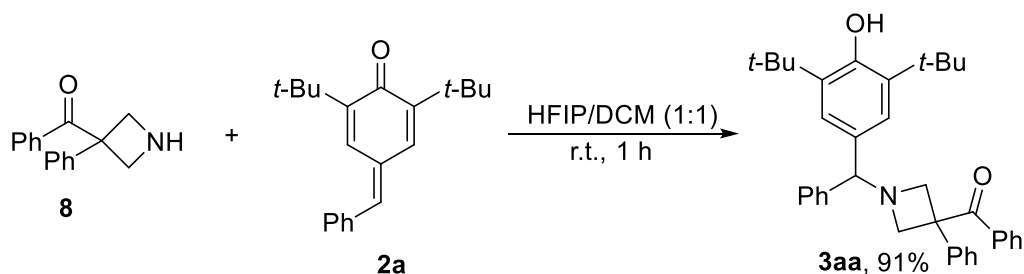


An oven-dried 25 mL Schlenk tube was charged with boronic acid **6** (65.7 mg, 0.22 mmol, 1.1 eq.), Pd(dppf)Cl₂ (14.6 mg, 0.02 mmol, 10 mol %), and K₂CO₃ (55.3 mg, 0.4 mmol, 2.0 eq.). After the flask was degassed and refilled with Ar for three times to ensure the complete exclusion of air, **3af** (122.1 mg, 0.2 mmol, 1.0 eq.) and anhydrous DMSO (2.0 mL) were added sequentially. The resulting mixture was heated in an oil bath at 80 °C for 2 h, quenched with 5.0 mL of H₂O and extracted with ethyl acetate (10 mL × 3). The combined organic layer was washed sequentially with water (20 mL) and saturated NaCl solution (20 mL), dried with anhydrous Na₂SO₄, filtered and concentrated under the reduced pressure. The residue was purified by silica gel column chromatography (PE/EA = 4:1) to give the pure product **7** (149.0 mg, 95% yield) as a yellow solid.

Control Experiments

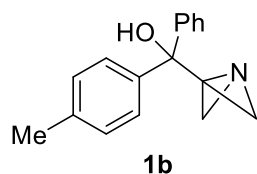


A mixture of ABB **1a** (71.2 mg, 0.3 mmol) and HFIP/DCM (1.0 mL/1.0 mL) was stirred for 3 h at room temperature. The residue was purified by silica gel column chromatography (DCM/MeOH = 10/1) to give product **8** (66.9 mg, 94% yield).

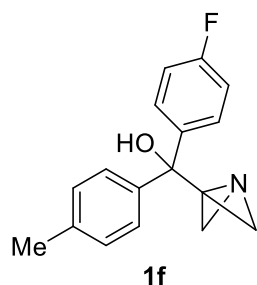


p-QM **2a** (70.7 mg, 0.24 mmol, 1.2 eq.) was added to a mixture of **8** (47.5 mg, 0.2 mmol, 1.0 eq.) and HFIP/DCM (1.0 mL/1.0 mL). The resulting mixture was stirred for 1 h at room temperature. After evaporating the solvent under reduced pressure, the residue was purified by basic aluminum oxide column chromatography (PE/EA = 20/1) to give the pure products **3aa** (96.8 mg, 91% yield) as a white solid.

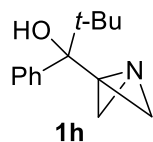
Characterization data



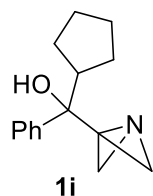
(1-Azabicyclo[1.1.0]butan-3-yl)(phenyl)(p-tolyl)methanol (1b). White solid; 377.0 mg, 30% yield, PE/EA = 3/1 as the eluent; mp = 92–93 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.49 (d, $J = 7.2$ Hz, 2H), 7.38–7.31 (m, 4H), 7.28–7.25 (m, 1H), 7.13 (d, $J = 8.0$ Hz, 2H), 3.02 (s, 1H), 2.33–2.28 (m, 5H), 1.47 (s, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 144.3, 141.3, 137.0, 128.7, 128.0, 127.3, 126.7, 126.6, 75.4, 54.7, 54.6, 38.1, 21.0; **HRMS (ESI)** m/z calcd for $[\text{M} + \text{H}]^+$ $\text{C}_{17}\text{H}_{18}\text{NO}$ 252.1383, found 252.1391.



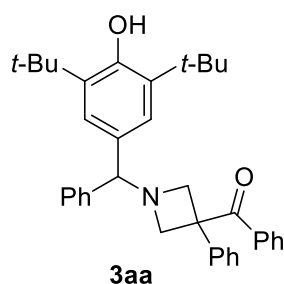
(1-Azabicyclo[1.1.0]butan-3-yl)(4-fluorophenyl)(p-tolyl)methanol (1f). White solid; 484.8 mg, 36% yield, PE/EA = 3/1 as the eluent; mp = 97–98 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.48–7.43 (m, 2H), 7.35 (d, $J = 8.4$ Hz, 2H), 7.14 (d, $J = 8.0$ Hz, 2H), 7.02–6.97 (m, 2H), 3.25 (s, 1H), 2.34–2.27 (m, 5H), 1.46 (t, $J = 2.8$ Hz, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 161.9 (d, $J = 245.0$ Hz), 141.2, 140.3 (d, $J = 4.0$ Hz), 137.2, 128.8, 128.5 (d, $J = 8.0$ Hz), 126.6, 114.7 (d, $J = 21.0$ Hz), 75.1, 54.7, 54.4, 38.0, 21.0; $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ -115.16 – -115.23 (m, 1F); **HRMS (ESI)** m/z calcd for $[\text{M} + \text{H}]^+$ $\text{C}_{17}\text{H}_{17}\text{FNO}$ 270.1289, found 270.1294.



1-(1-Azabicyclo[1.1.0]butan-3-yl)-2,2-dimethyl-1-phenylpropan-1-ol (1h). White solid; 434.6 mg, 40% yield, PE/EA = 8/1 as the eluent; mp = 124–125 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.50 (d, $J = 7.2$ Hz, 2H), 7.31–7.22 (m, 3H), 2.72 (dd, $J = 6.8, 2.8$ Hz, 1H), 2.43 (dd, $J = 6.8, 2.8$ Hz, 1H), 2.26 (s, 1H), 1.48 (d, $J = 2.8$ Hz, 1H), 1.43 (d, $J = 2.4$ Hz, 1H), 1.03 (s, 9H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 141.1, 128.1, 126.8, 126.7, 77.2, 58.14, 58.10, 53.66, 39.3, 34.8, 26.3; **HRMS (ESI)** m/z calcd for $[\text{M} + \text{H}]^+$ $\text{C}_{14}\text{H}_{20}\text{NO}$ 218.1539, found 218.1545.

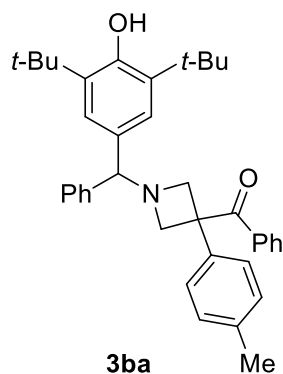


1-(1-Azabicyclo[1.1.0]butan-3-yl)(cyclopentyl)(phenyl)methanol (1i). White solid; 435.7 mg, 38% yield, PE/EA = 5/1 as the eluent; mp = 81–82 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.48 (d, $J = 7.6$ Hz, 2H), 7.33 (t, $J = 7.2$ Hz, 2H), 7.24 (t, $J = 7.2$ Hz, 1H), 2.65–2.56 (m, 2H), 2.44 (s, 1H), 2.13 (dd, $J = 6.8, 2.8$ Hz, 1H), 1.82–1.65 (m, 2H), 1.63–1.46 (m, 3H), 1.44–1.25 (m, 4H), 1.19 (d, $J = 2.4$ Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 143.7, 127.6, 126.7, 125.9, 74.0, 53.8, 53.6, 48.6, 37.6, 27.7, 26.4, 26.1, 25.2; **HRMS (ESI)** m/z calcd for $[\text{M} + \text{H}]^+$ $\text{C}_{15}\text{H}_{20}\text{NO}$ 230.1539, found 230.1544.

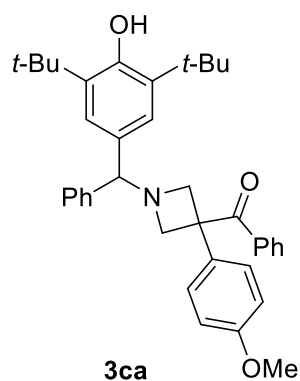


1-((3,5-Di-tert-butyl-4-hydroxyphenyl)(phenyl)methyl)-3-phenylazetidin-3-yl(phenyl)metha

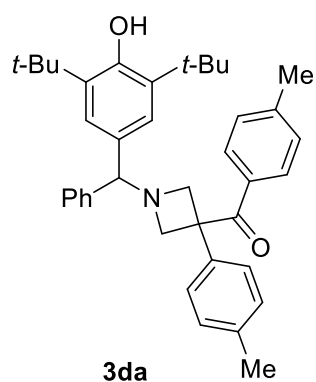
none (3aa). White solid; 88.3 mg, 83% yield, PE/EA = 20/1 as the eluent; mp = 103–104 °C; ^1H NMR (400 MHz, DMSO- d_6) δ 7.68 (d, J = 7.6 Hz, 2H), 7.54–7.50 (m, 3H), 7.40–7.37 (m, 6H), 7.26 (t, J = 7.6 Hz, 3H), 7.19–7.14 (m, 3H), 6.82 (s, 1H), 4.38 (s, 1H), 3.72 (dd, J = 19.2, 7.6 Hz, 2H), 3.61 (d, J = 7.6 Hz, 1H), 3.52 (d, J = 7.2 Hz, 1H), 1.33 (s, 18H); ^{13}C NMR (100 MHz, CDCl $_3$) δ 199.9, 152.7, 142.7, 142.0, 135.6, 134.1, 132.7, 132.4, 129.5, 129.0, 128.4, 127.5, 126.93, 126.87, 126.1, 123.8, 77.7, 62.7, 62.6, 51.8, 34.3, 30.3; **HRMS (ESI)** m/z calcd for $[\text{M} + \text{H}]^+$ C $_{37}$ H $_{42}$ NO $_2$ 532.3210, found 532.3220.



(1-((3,5-Di-tert-butyl-4-hydroxyphenyl)(phenyl)methyl)-3-(*p*-tolyl)azetidin-3-yl)(phenyl)methanone (3ba). White solid; 72.0 mg, 66% yield, PE/EA = 20/1 as the eluent; mp = 163–164 °C; ^1H NMR (400 MHz, DMSO- d_6) δ 7.67 (d, J = 7.6 Hz, 2H), 7.51 (t, J = 7.2 Hz, 1H), 7.42–7.37 (m, 6H), 7.26 (t, J = 7.2 Hz, 2H), 7.18–7.14 (m, 5H), 6.83 (s, 1H), 4.36 (s, 1H), 3.70 (dd, J = 22.8, 7.2 Hz, 2H), 3.58 (d, J = 7.2 Hz, 1H), 3.48 (d, J = 7.2 Hz, 1H), 2.26 (s, 3H), 1.33 (s, 18H); ^{13}C NMR (100 MHz, DMSO- d_6) δ 199.0, 152.8, 142.8, 139.1, 138.4, 136.2, 133.3, 133.1, 132.9, 129.6, 129.2, 128.7, 128.4, 127.1, 126.8, 125.9, 123.0, 76.4, 62.2, 61.9, 51.2, 34.5, 30.4, 20.5; **HRMS (ESI)** m/z calcd for $[\text{M} + \text{H}]^+$ C $_{38}$ H $_{44}$ NO $_2$ 546.3367, found 546.3374.

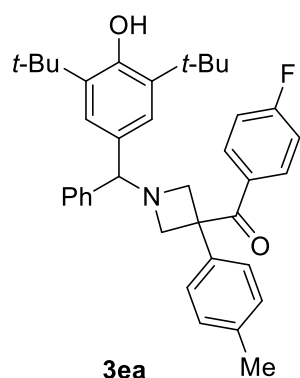


(1-((3,5-Di-*tert*-butyl-4-hydroxyphenyl)(phenyl)methyl)-3-(4-methoxyphenyl)azetidin-3-yl)(p-phenyl)methanone. White solid; 78.6 mg, 70% yield, PE/EA = 10/1 as the eluent; mp = 79–80 °C; $^1\text{H NMR}$ (400 MHz, DMSO- d_6) δ 7.68 (d, J = 7.6 Hz, 2H), 7.52 (t, J = 7.6 Hz, 1H), 7.44–7.37 (m, 6H), 7.26 (t, J = 7.2 Hz, 2H), 7.18–7.14 (m, 3H), 6.94 (d, J = 8.8 Hz, 2H), 6.83 (s, 1H), 4.36 (s, 1H), 3.72–3.65 (m, 5H), 3.58 (d, J = 7.2 Hz, 1H), 3.49 (d, J = 6.8 Hz, 1H), 1.33 (s, 18H); $^{13}\text{C NMR}$ (100 MHz, DMSO- d_6) δ 199.1, 158.1, 152.8, 142.8, 139.1, 133.3, 133.2, 133.1, 132.9, 129.2, 128.7, 128.4, 127.3, 127.1, 126.8, 123.1, 114.3, 76.4, 62.3, 62.1, 55.0, 50.9, 34.5, 30.4; **HRMS (ESI)** m/z calcd for $[\text{M} + \text{H}]^+$ $\text{C}_{38}\text{H}_{44}\text{NO}_3$ 562.3316, found 562.3322.

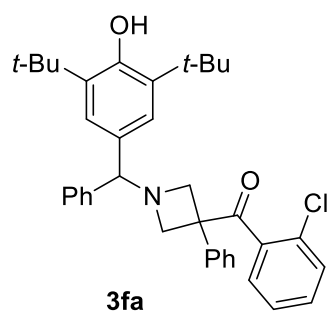


(1-((3,5-Di-*tert*-butyl-4-hydroxyphenyl)(phenyl)methyl)-3-(*p*-tolyl)azetidin-3-yl)(*p*-tolyl)methanone (3da). White solid; 80.6 mg, 72% yield, PE/EA = 20/1 as the eluent; mp = 144–145 °C; $^1\text{H NMR}$ (400 MHz, DMSO- d_6) δ 7.56 (d, J = 8.4 Hz, 2H), 7.40–7.37 (m, 4H), 7.26 (t, J = 7.6 Hz, 2H), 7.18–7.14 (m, 7H), 6.83 (s, 1H), 4.35 (s, 1H), 3.68 (dd, J = 22.4, 7.2 Hz, 2H), 3.56 (d, J = 7.6

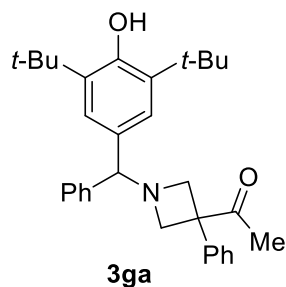
Hz, 1H), 3.47 (d, $J = 7.2$ Hz, 1H), 2.26 (d, $J = 4.4$ Hz, 6H), 1.34 (s, 18H); ^{13}C NMR (100 MHz, DMSO- d_6) δ 198.6, 152.8, 143.5, 142.8, 139.1, 138.6, 136.1, 132.9, 130.8, 129.5, 129.3, 129.2, 128.4, 127.1, 126.8, 125.9, 123.0, 76.4, 62.3, 62.0, 51.1, 34.5, 30.4, 21.1, 20.5; HRMS (ESI) m/z calcd for $[\text{M} + \text{H}]^+$ $\text{C}_{39}\text{H}_{46}\text{NO}_2$ 560.3523, found 560.3531.



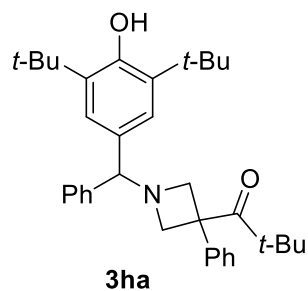
(1-((3,5-Di-*tert*-butyl-4-hydroxyphenyl)(phenyl)methyl)-3-(*p*-tolyl)azetidin-3-yl)(4-fluorophenyl)methanone (3ea). White solid; 102.6 mg, 91% yield, PE/EA = 20/1 as the eluent; mp = 101–102 °C; ^1H NMR (400 MHz, DMSO- d_6) δ 7.77–7.73 (m, 2H), 7.39 (t, $J = 8.0$ Hz, 4H), 7.28–7.14 (m, 9H), 6.83 (s, 1H), 4.37 (s, 1H), 3.70 (dd, $J = 20.8, 7.2$ Hz, 2H), 3.58 (d, $J = 7.2$ Hz, 1H), 3.47 (d, $J = 7.2$ Hz, 1H), 2.26 (s, 3H), 1.34 (s, 18H); ^{13}C NMR (100 MHz, DMSO- d_6) δ 197.5, 164.7 (d, $J = 251.0$ Hz), 152.8, 142.8, 139.1, 138.2, 136.3, 132.9, 132.3 (d, $J = 9.0$ Hz), 130.0 (d, $J = 2.0$ Hz), 129.6, 128.4, 127.1, 126.9, 125.9, 123.0, 115.8 (d, $J = 22.0$ Hz), 76.3, 62.2, 61.8, 51.1, 34.5, 30.4, 20.5; ^{19}F NMR (376 MHz, DMSO- d_6) δ -105.39 – -105.47 (m, 1F); HRMS (ESI) m/z calcd for $[\text{M} + \text{H}]^+$ $\text{C}_{38}\text{H}_{43}\text{FNO}_2$ 564.3272, found 564.3282.



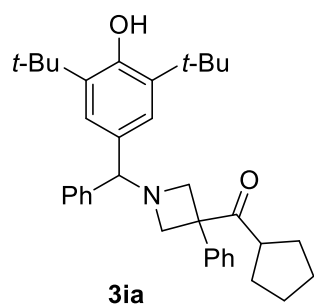
(2-Chlorophenyl)(1-((3,5-di-*tert*-butyl-4-hydroxyphenyl)(phenyl)methyl)-3-phenylazetidin-3-yl)methanone (3fa). White solid; 98.5 mg, 87% yield, PE/EA = 20/1 as the eluent; mp = 159–160 °C; $^1\text{H NMR}$ (400 MHz, DMSO- d_6) δ 7.51 (d, J = 8.0 Hz, 1H), 7.46–7.42 (m, 1H), 7.35–7.23 (m, 10H), 7.16–7.10 (m, 4H), 6.81 (s, 1H), 4.32 (s, 1H), 3.78 (d, J = 6.8 Hz, 2H), 3.40 (dd, J = 16.4, 7.2 Hz, 2H), 1.32 (s, 18H); $^{13}\text{C NMR}$ (100 MHz, DMSO- d_6) δ 202.8, 152.8, 142.8, 140.0, 139.1, 137.4, 132.8, 131.4, 130.1, 129.6, 128.6, 128.4, 127.8, 127.1, 126.9, 126.8, 126.6, 123.0, 76.4, 62.2, 61.9, 54.0, 34.5, 30.4; **HRMS (ESI)** m/z calcd for $[\text{M} + \text{H}]^+$ $\text{C}_{37}\text{H}_{41}\text{ClNO}_2$ 566.2820, found 566.2826.



1-((3,5-Di-*tert*-butyl-4-hydroxyphenyl)(phenyl)methyl)-3-phenylazetidin-3-ylethan-1-one (3ga). White solid; 64.8 mg, 69% yield, PE/EA = 10/1 as the eluent; mp = 173–174 °C; $^1\text{H NMR}$ (400 MHz, DMSO- d_6) δ 7.38–7.33 (m, 4H), 7.29–7.24 (m, 5H), 7.18–7.15 (m, 3H), 6.82 (s, 1H), 4.30 (s, 1H), 3.79 (d, J = 7.2 Hz, 1H), 3.69 (d, J = 7.2 Hz, 1H), 3.35 (d, J = 7.6 Hz, 1H), 3.24 (d, J = 7.2 Hz, 1H), 2.07 (s, 3H), 1.34 (s, 18H); $^{13}\text{C NMR}$ (100 MHz, DMSO- d_6) δ 206.9, 152.8, 142.9, 140.7, 139.1, 132.9, 128.7, 128.4, 127.2, 127.0, 126.9, 126.5, 123.1, 76.7, 61.1, 60.9, 53.9, 34.5, 30.4, 24.8; **HRMS (ESI)** m/z calcd for $[\text{M} + \text{H}]^+$ $\text{C}_{32}\text{H}_{40}\text{NO}_2$ 470.3054, found 470.3060.

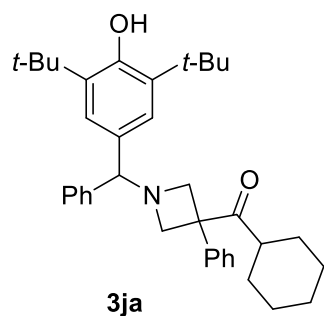


1-(1-((3,5-Di-*tert*-butyl-4-hydroxyphenyl)(phenyl)methyl)-3-phenylazetidin-3-yl)-2,2-dimethylpropan-1-one (3ha). White solid; 65.5 mg, 64% yield, PE/EA = 20/1 as the eluent; mp = 143–144 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.39–7.36 (m, 6H), 7.31–7.26 (m, 2H), 7.25–7.14 (m, 4H), 5.06 (s, 1H), 4.22 (s, 1H), 3.63 (dd, $J = 13.2, 7.2$ Hz, 2H), 3.55 (d, $J = 7.6$ Hz, 1H), 3.44 (d, $J = 7.6$ Hz, 1H), 1.40 (s, 18H), 0.99 (s, 9H); $^{13}\text{C NMR}$ (100 MHz, $\text{THF-}d_8$) δ 213.4, 153.8, 143.9, 141.5, 137.7, 133.7, 129.2, 128.8, 128.0, 127.3, 127.2, 127.1, 124.2, 78.5, 62.3, 62.2, 54.7, 44.9, 35.0, 30.5, 28.6; **HRMS (ESI)** m/z calcd for $[\text{M} + \text{H}]^+$ $\text{C}_{35}\text{H}_{46}\text{NO}_2$ 512.3523, found 512.3529.

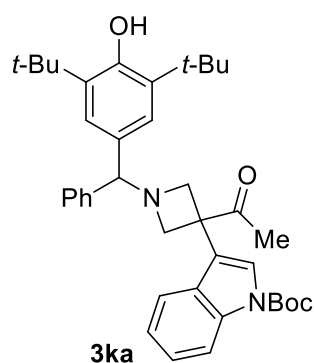


Cyclopentyl(1-((3,5-di-*tert*-butyl-4-hydroxyphenyl)(phenyl)methyl)-3-phenylazetidin-3-yl)methanone (3ia). White solid; 78.6 mg, 75% yield, PE/EA = 20/1 as the eluent; mp = 155–156 °C; $^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ 7.37–7.31 (m, 4H), 7.29–7.21 (m, 5H), 7.18–7.14 (m, 3H), 6.84 (s, 1H), 4.28 (s, 1H), 3.80 (d, $J = 7.2$ Hz, 1H), 3.72 (d, $J = 6.8$ Hz, 1H), 3.25 (t, $J = 7.2$ Hz, 2H), 3.18–3.10 (m, 1H), 1.57–1.50 (m, 4H), 1.48–1.40 (m, 4H), 1.34 (s, 18H); $^{13}\text{C NMR}$ (100 MHz, $\text{THF-}d_8$) δ 211.9, 153.8, 144.0, 141.7, 137.7, 133.7, 129.2, 128.8, 128.0, 127.3, 127.2, 124.3, 78.7, 61.8, 61.7, 55.1, 47.1, 35.0, 32.5, 32.2, 30.5, 27.0, 26.9; **HRMS (ESI)** m/z calcd for $[\text{M} + \text{H}]^+$

C₃₆H₄₆NO₂ 524.3523, found 524.3528.



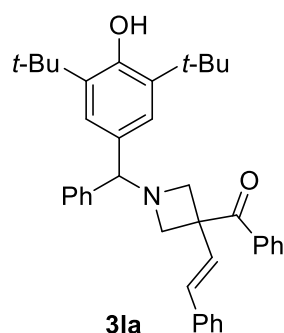
Cyclohexyl(1-((3,5-di-*tert*-butyl-4-hydroxyphenyl)(phenyl)methyl)-3-phenylazetidin-3-yl)metanone (3ja). White solid; 96.8 mg, 90% yield, PE/EA = 20/1 as the eluent; mp = 143–144 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 7.36–7.31 (m, 4H), 7.29–7.16 (m, 8H), 6.85 (s, 1H), 4.27 (s, 1H), 3.80 (d, *J* = 6.8 Hz, 1H), 3.71 (d, *J* = 6.8 Hz, 1H), 3.24 (t, *J* = 8.0 Hz, 2H), 2.83–2.78 (m, 1H), 1.60–1.50 (m, 3H), 1.44–1.34 (m, 19H), 1.27–1.08 (m, 6H); ¹³C NMR (100 MHz, THF-*d*₈) δ 210.9, 153.8, 144.0, 141.4, 137.7, 133.7, 129.1, 128.8, 127.9, 127.4, 127.2, 127.1, 124.2, 78.7, 61.8, 61.7, 55.1, 46.8, 35.0, 31.0, 30.7, 30.5, 26.5, 26.24, 26.19; **HRMS (ESI)** *m/z* calcd for [M + H]⁺ C₃₇H₄₈NO₂ 538.3680, found 538.3685.



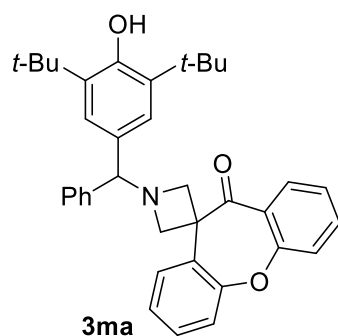
***Tert*-butyl**

3-(3-acetyl-1-((3,5-di-*tert*-butyl-4-hydroxyphenyl)(phenyl)methyl)azetidin-3-yl)-1*H*-indole-1-carboxylate (3ka). White solid; 81.6 mg, 67% yield, PE/EA = 10/1 as the eluent; mp = 180–181 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 8.07 (d, *J* = 8.4 Hz, 1H), 7.79 (s, 1H), 7.38 (d, *J* = 7.2 Hz, 2H), 7.33–7.21 (m, 4H), 7.18–7.13 (m, 4H), 6.83 (s, 1H), 4.35 (s, 1H), 3.85 (d, *J* = 7.2 Hz,

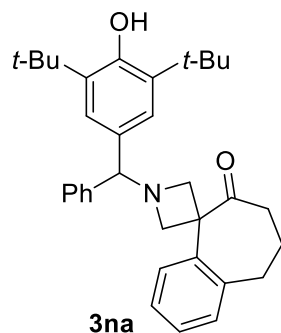
1H), 3.72 (d, $J = 7.2$ Hz, 1H), 3.47 (d, $J = 7.2$ Hz, 1H), 3.27 (d, $J = 7.2$ Hz, 1H), 2.12 (s, 3H), 1.64 (s, 9H), 1.35 (s, 18H); ^{13}C NMR (100 MHz, DMSO- d_6) δ 206.4, 152.8, 148.9, 142.8, 139.1, 135.0, 132.8, 128.4, 128.1, 127.1, 126.8, 124.6, 123.7, 123.0, 122.8, 120.4, 119.7, 115.0, 83.9, 76.5, 60.0, 59.9, 48.1, 34.5, 30.4, 27.7, 24.6; HRMS (ESI) m/z calcd for $[\text{M} + \text{H}]^+$ $\text{C}_{39}\text{H}_{49}\text{N}_2\text{O}_4$ 609.3687, found 609.3696.



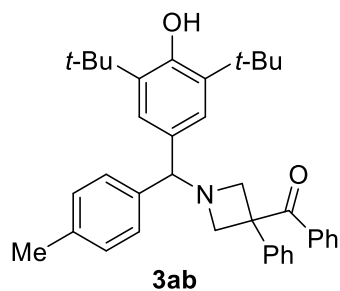
(E)-1-((3,5-Di-*tert*-butyl-4-hydroxyphenyl)(phenyl)methyl)-3-styrylazetid-3-yl(phenyl)ethanone (3la). White solid; 70.3 mg, 63% yield, PE/EA = 20/1 as the eluent; mp = 166–167 °C; ^1H NMR (400 MHz, DMSO- d_6) δ 7.81 (d, $J = 7.6$ Hz, 2H), 7.59 (t, $J = 7.6$ Hz, 1H), 7.50–7.43 (m, 6H), 7.33–7.21 (m, 5H), 7.18–7.15 (m, 3H), 6.95 (d, $J = 16.4$ Hz, 1H), 6.82 (s, 1H), 6.56 (d, $J = 16.4$ Hz, 1H), 4.34 (s, 1H), 3.57 (dd, $J = 11.6, 7.2$ Hz, 2H), 3.49 (dd, $J = 10.8, 7.2$ Hz, 2H), 1.34 (s, 18H); ^{13}C NMR (100 MHz, DMSO- d_6) δ 198.8, 152.8, 142.8, 139.0, 136.3, 133.3, 132.9, 131.4, 130.7, 129.1, 128.8, 128.6, 128.4, 127.8, 127.2, 126.8, 126.3, 123.1, 76.5, 62.0, 49.5, 34.5, 30.4; HRMS (ESI) m/z calcd for $[\text{M} + \text{H}]^+$ $\text{C}_{39}\text{H}_{44}\text{NO}_2$ 558.3367, found 558.3375.



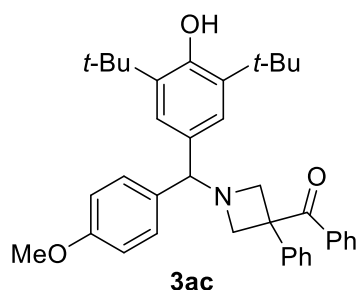
1-((3,5-Di-*tert*-butyl-4-hydroxyphenyl)(phenyl)methyl)-11'*H*-spiro[azetidine-3,10'-dibenzo[*b*,*f*]oxepin]-11'-one (3ma). White solid; 65.5 mg, 60% yield, PE/EA = 10/1 as the eluent; mp = 145–146 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 8.04 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.70–7.65 (m, 1H), 7.40–7.26 (m, 10H), 7.15–7.12 (m, 3H), 6.83 (s, 1H), 4.34 (s, 1H), 3.88 (d, *J* = 6.8 Hz, 1H), 3.71 (d, *J* = 7.2 Hz, 1H), 3.41 (t, *J* = 8.0 Hz, 2H), 1.31 (s, 18H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ 190.4, 158.5, 155.8, 152.8, 142.6, 139.0, 135.5, 132.7, 131.0, 130.7, 129.0, 128.4, 127.2, 127.1, 126.9, 126.6, 124.3, 123.10, 123.07, 121.1, 120.8, 76.5, 59.6, 59.1, 51.8, 34.5, 30.4; **HRMS (ESI)** *m/z* calcd for [M + H]⁺ C₃₇H₄₀NO₃ 546.3003, found 546.3011.



1-((3,5-Di-*tert*-butyl-4-hydroxyphenyl)(phenyl)methyl)-8',9'-dihydrospiro[azetidine-3,5'-benzo[7]annulen]-6'(7'*H*)-one (3na). White solid; 56.5 mg, 57% yield, PE/EA = 10/1 as the eluent; mp = 163–164 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 7.37 (d, *J* = 7.2 Hz, 2H), 7.29–7.13 (m, 9H), 6.82 (s, 1H), 4.31 (s, 1H), 3.90 (d, *J* = 7.2 Hz, 1H), 3.79 (d, *J* = 6.8 Hz, 1H), 3.33 (d, *J* = 7.6 Hz, 1H), 3.24 (d, *J* = 6.8 Hz, 1H), 2.78–2.69 (m, 4H), 1.86–1.75 (m, 2H), 1.35 (s, 18H); ¹³C NMR (100 MHz, THF-*d*₈) δ 208.5, 153.8, 144.0, 141.3, 139.9, 137.7, 133.6, 130.2, 128.8, 128.1, 127.9, 127.5, 127.2, 126.9, 124.3, 78.4, 62.6, 62.5, 55.0, 40.5, 35.0, 33.3, 30.5, 28.6; **HRMS (ESI)** *m/z* calcd for [M + H]⁺ C₃₄H₄₂NO₂ 496.3210, found 496.3216.



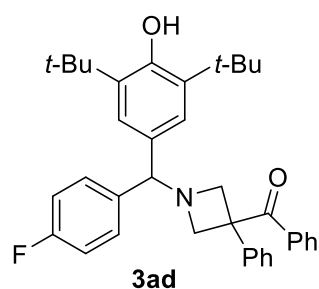
(1-((3,5-Di-*tert*-butyl-4-hydroxyphenyl)(*p*-tolyl)methyl)-3-phenylazetidin-3-yl)(phenyl)methanone (3ab). White solid; 88.4 mg, 81% yield, PE/EA = 20/1 as the eluent; mp = 181–182 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.66 (d, *J* = 7.6 Hz, 2H), 7.57 (d, *J* = 7.6 Hz, 2H), 7.42–7.28 (m, 7H), 7.26–7.23 (m, 3H), 7.07 (d, *J* = 8.0 Hz, 2H), 5.03 (s, 1H), 4.28 (s, 1H), 3.78–3.71 (m, 3H), 3.62 (d, *J* = 7.6 Hz, 1H), 2.28 (s, 3H), 1.40 (s, 18H); ¹³C NMR (100 MHz, CDCl₃) δ 200.0, 152.7, 142.1, 139.8, 136.4, 135.5, 134.2, 132.70, 132.68, 129.5, 129.1, 128.9, 128.3, 127.3, 126.9, 126.1, 123.7, 77.5, 62.69, 62.65, 51.7, 34.3, 30.3, 21.1; HRMS (ESI) *m/z* calcd for [M + H]⁺ C₃₈H₄₄NO₂ 546.3367, found 546.3372.



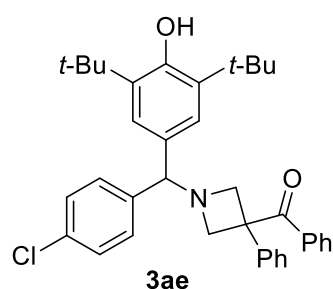
(1-((3,5-Di-*tert*-butyl-4-hydroxyphenyl)(4-methoxyphenyl)methyl)-3-phenylazetidin-3-yl)(phenyl)methanone (3ac). White solid; 94.4 mg, 84% yield, PE/EA = 10/1 as the eluent; mp = 169–170 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.65 (d, *J* = 7.2 Hz, 2H), 7.56 (d, *J* = 7.6 Hz, 2H), 7.42–7.28 (m, 7H), 7.26–7.24 (m, 1H), 7.20 (s, 2H), 6.79 (d, *J* = 8.4 Hz, 2H), 5.03 (s, 1H), 4.25 (s, 1H), 3.76–3.68 (m, 6H), 3.59 (d, *J* = 7.6 Hz, 1H), 1.40 (s, 18H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ 198.9, 158.1, 152.6, 141.4, 139.1, 134.7, 133.3, 133.20, 133.16, 129.2, 129.0, 128.7, 128.2, 127.0,

126.0, 122.9, 113.8, 75.6, 62.1, 62.0, 54.9, 51.5, 34.5, 30.4; **HRMS (ESI)** m/z calcd for $[M + H]^+$

$C_{38}H_{44}NO_3$ 562.3316, found 562.3315.

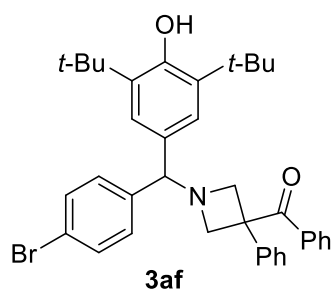


(1-((3,5-Di-*tert*-butyl-4-hydroxyphenyl)(4-fluorophenyl)methyl)-3-phenylazetidin-3-yl)(phenyl)methanone (3ad). White solid; 85.6 mg, 78% yield, PE/EA = 20/1 as the eluent; mp = 144–145 °C; 1H NMR (400 MHz, DMSO- d_6) δ 7.69 (d, J = 7.6 Hz, 2H), 7.54–7.50 (m, 3H), 7.43–7.36 (m, 6H), 7.28–7.24 (m, 1H), 7.17 (s, 2H), 7.09 (t, J = 8.4 Hz, 2H), 6.83 (s, 1H), 4.41 (s, 1H), 3.73 (dd, J = 16.0, 7.6 Hz, 2H), 3.62 (d, J = 7.6 Hz, 1H), 3.52 (d, J = 7.2 Hz, 1H), 1.34 (s, 18H); ^{13}C NMR (100 MHz, DMSO- d_6) δ 198.8, 161.1 (d, J = 241.0 Hz), 152.8, 141.3, 139.2, 138.9 (d, J = 3.0 Hz), 133.3, 133.1, 132.7, 129.2, 129.0, 128.9, 128.7, 127.0, 126.0, 123.0, 115.1 (d, J = 21.0 Hz), 75.3, 62.1, 61.8, 51.5, 34.50, 30.4; ^{19}F NMR (376 MHz, DMSO- d_6) δ -115.93 – -116.01 (m, 1F); **HRMS (ESI)** m/z calcd for $[M + H]^+$ $C_{37}H_{41}FNO_2$ 550.3116, found 550.3123.

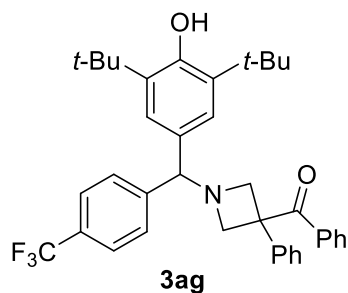


(1-((4-Chlorophenyl)(3,5-di-*tert*-butyl-4-hydroxyphenyl)methyl)-3-phenylazetidin-3-yl)(phenyl)methanone (3ae). White solid; 86.1 mg, 76% yield, PE/EA = 20/1 as the eluent; mp = 168–169 °C; 1H NMR (400 MHz, DMSO- d_6) δ 7.68 (d, J = 7.6 Hz, 2H), 7.53–7.49 (m, 3H), 7.41–7.36

(m, 6H), 7.31 (d, $J = 8.0$ Hz, 2H), 7.27–7.24 (m, 1H), 7.17 (s, 2H), 6.84 (s, 1H), 4.42 (s, 1H), 3.73 (dd, $J = 16.8, 7.2$ Hz, 2H), 3.62 (d, $J = 7.6$ Hz, 1H), 3.54 (d, $J = 7.2$ Hz, 1H), 1.33 (s, 18H); ^{13}C NMR (100 MHz, DMSO- d_6) δ 198.8, 152.9, 141.8, 141.3, 139.2, 133.3, 133.2, 132.4, 131.3, 129.2, 129.0, 128.9, 128.7, 128.4, 127.0, 126.1, 123.0, 75.3, 62.1, 61.8, 51.6, 34.5, 30.4; HRMS (ESI) m/z calcd for $[\text{M} + \text{H}]^+$ $\text{C}_{37}\text{H}_{41}\text{ClNO}_2$ 566.2820, found 566.2823.

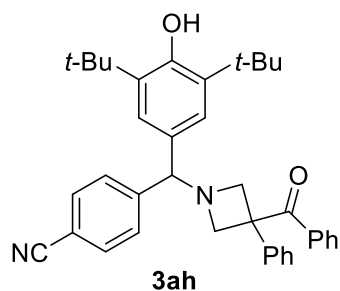


(1-((4-Bromophenyl)(3,5-di-*tert*-butyl-4-hydroxyphenyl)methyl)-3-phenylazetidin-3-yl)(phenyl)methanone (3af). White solid; 89.2 mg, 73% yield, PE/EA = 20/1 as the eluent; mp = 186–187 °C; ^1H NMR (400 MHz, DMSO- d_6) δ 7.68 (d, $J = 7.6$ Hz, 2H), 7.54–7.50 (m, 3H), 7.46 (d, $J = 8.4$ Hz, 2H), 7.40–7.34 (m, 6H), 7.28–7.24 (m, 1H), 7.16 (s, 2H), 6.84 (s, 1H), 4.41 (s, 1H), 3.73 (dd, $J = 16.4, 7.2$ Hz, 2H), 3.63 (d, $J = 7.6$ Hz, 1H), 3.55 (d, $J = 7.2$ Hz, 1H), 1.33 (s, 18H); ^{13}C NMR (100 MHz, DMSO- d_6) δ 198.8, 152.9, 142.2, 141.2, 139.2, 133.3, 133.1, 132.3, 131.3, 129.3, 129.2, 129.0, 128.7, 127.0, 126.0, 122.9, 119.8, 75.4, 62.0, 61.8, 51.5, 34.5, 30.4; HRMS (ESI) m/z calcd for $[\text{M} + \text{H}]^+$ $\text{C}_{37}\text{H}_{41}\text{BrNO}_2$ 610.2315, found 610.2322.



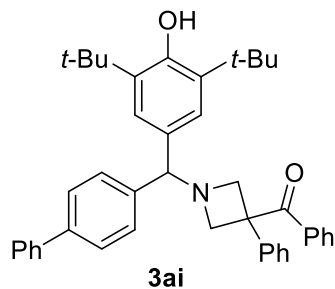
(1-((3,5-Di-*tert*-butyl-4-hydroxyphenyl)(4-(trifluoromethyl)phenyl)methyl)-3-phenylazetidin-

3-yl)(phenyl)methanone (3ag). White solid; 78.0 mg, 65% yield, PE/EA = 20/1 as the eluent; mp = 170–171 °C; $^1\text{H NMR}$ (400 MHz, DMSO- d_6) δ 7.69 (d, J = 7.6 Hz, 2H), 7.65–7.60 (m, 4H), 7.54–7.50 (m, 3H), 7.38 (t, J = 7.6 Hz, 4H), 7.28–7.24 (m, 1H), 7.20 (s, 2H), 6.87 (s, 1H), 4.54 (s, 1H), 3.75 (dd, J = 16.4, 7.6 Hz, 2H), 3.66 (d, J = 7.6 Hz, 1H), 3.58 (d, J = 7.2 Hz, 1H), 1.33 (s, 18H); $^{13}\text{C NMR}$ (100 MHz, DMSO- d_6) δ 198.7, 153.0, 147.4, 141.2, 139.3, 133.2, 133.1, 132.0, 129.2, 128.9, 128.7, 127.8, 127.5 (q, J = 32.0 Hz), 127.0, 126.0, 125.3 (q, J = 4.0 Hz), 124.2 (q, J = 270.0 Hz), 123.1, 75.6, 62.0, 61.7, 51.6, 34.5, 30.3; $^{19}\text{F NMR}$ (376 MHz, DMSO- d_6) δ -60.84 (s, 3F); **HRMS (ESI)** m/z calcd for $[\text{M} + \text{H}]^+$ $\text{C}_{38}\text{H}_{41}\text{F}_3\text{NO}_2$ 600.3084, found 600.3091.

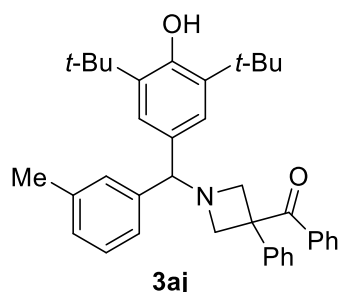


4-((3-Benzoyl-3-phenylazetidin-1-yl)(3,5-di-*tert*-butyl-4-hydroxyphenyl)methyl)benzonitrile

(3ah). White solid; 83.5 mg, 75% yield, PE/EA = 10/1 as the eluent; mp = 184–185 °C; $^1\text{H NMR}$ (400 MHz, DMSO- d_6) δ 7.72–7.67 (m, 4H), 7.59 (d, J = 8.4 Hz, 2H), 7.54–7.50 (m, 3H), 7.38 (t, J = 7.6 Hz, 4H), 7.28–7.25 (m, 1H), 7.18 (s, 2H), 6.87 (s, 1H), 4.54 (s, 1H), 3.74 (dd, J = 15.6, 7.6 Hz, 2H), 3.66 (d, J = 7.6 Hz, 1H), 3.57 (d, J = 7.2 Hz, 1H), 1.33 (s, 18H); $^{13}\text{C NMR}$ (100 MHz, DMSO- d_6) δ 198.7, 153.1, 148.4, 141.2, 139.3, 133.20, 133.17, 132.4, 131.7, 129.2, 129.0, 128.7, 128.0, 127.0, 126.0, 123.1, 118.8, 109.6, 75.4, 62.0, 61.6, 51.6, 34.5, 30.3; **HRMS (ESI)** m/z calcd for $[\text{M} + \text{H}]^+$ $\text{C}_{38}\text{H}_{41}\text{N}_2\text{O}_2$ 557.3163, found 557.3164.

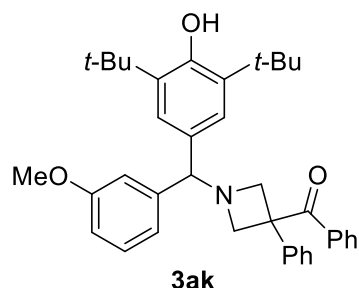


(1-([1,1'-Biphenyl]-4-yl(3,5-di-tert-butyl-4-hydroxyphenyl)methyl)-3-phenylazetidin-3-yl)(phenyl)methanone (3ai). White solid; 97.3 mg, 80% yield, PE/EA = 20/1 as the eluent; mp = 172–173 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 7.70 (d, *J* = 7.6 Hz, 2H), 7.60–7.37 (m, 15H), 7.34–7.25 (m, 4H), 6.84 (s, 1H), 4.44 (s, 1H), 3.77 (dd, *J* = 14.0, 7.2 Hz, 2H), 3.65 (d, *J* = 7.6 Hz, 1H), 3.57 (d, *J* = 6.8 Hz, 1H), 1.35 (s, 18H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ 198.9, 152.8, 142.0, 141.4, 139.9, 139.2, 138.8, 133.3, 133.2, 132.8, 129.2, 129.0, 128.8, 128.7, 127.7, 127.2, 127.0, 126.8, 126.5, 126.1, 123.0, 75.9, 62.2, 62.0, 51.6, 34.5, 30.4; **HRMS (ESI)** *m/z* calcd for [M + H]⁺ C₄₃H₄₆NO₂ 608.3523, found 608.3527.

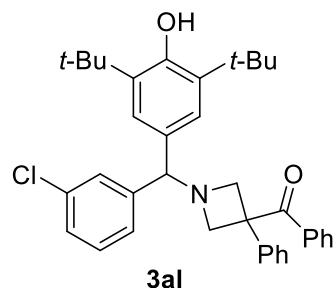


(1-((3,5-Di-tert-butyl-4-hydroxyphenyl)(*m*-tolyl)methyl)-3-phenylazetidin-3-yl)(phenyl)methanone (3aj). White solid; 71.0 mg, 65% yield, PE/EA = 20/1 as the eluent; mp = 145–146 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 7.68 (d, *J* = 7.6 Hz, 2H), 7.53–7.50 (m, 3H), 7.38 (t, *J* = 7.6 Hz, 4H), 7.28–7.24 (m, 1H), 7.20–7.13 (m, 5H), 6.97 (d, *J* = 6.8 Hz, 1H), 6.80 (s, 1H), 4.32 (s, 1H), 3.72 (dd, *J* = 18.4, 7.6 Hz, 2H), 3.60 (d, *J* = 7.2 Hz, 1H), 3.51 (d, *J* = 7.2 Hz, 1H), 2.24 (s, 3H), 1.34 (s, 18H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ 198.9, 152.7, 142.6, 141.3, 139.0, 137.3, 133.3,

133.1, 132.8, 129.2, 128.9, 128.7, 128.3, 127.8, 127.5, 127.0, 126.0, 124.2, 123.1, 76.5, 62.2, 61.9, 51.5, 34.5, 30.4, 21.1; **HRMS (ESI)** m/z calcd for $[M + H]^+$ $C_{38}H_{44}NO_2$ 546.3367, found 546.3372.

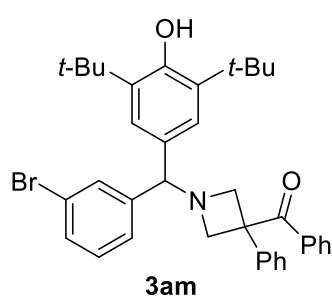


(1-((3,5-Di-tert-butyl-4-hydroxyphenyl)(3-methoxyphenyl)methyl)-3-phenylazetidin-3-yl)(phenyl)methanone (3ak). White solid; 70.8 mg, 63% yield, PE/EA = 10/1 as the eluent; mp = 130–131 °C; **¹H NMR** (400 MHz, DMSO- d_6) δ 7.68 (d, J = 8.0 Hz, 2H), 7.54–7.50 (m, 3H), 7.39 (t, J = 7.6 Hz, 4H), 7.28–7.24 (m, 1H), 7.20–7.16 (m, 3H), 6.98–6.94 (m, 2H), 6.80 (s, 1H), 6.74 (d, J = 8.0 Hz, 1H), 4.34 (s, 1H), 3.72–3.69 (m, 5H), 3.62 (d, J = 7.6 Hz, 1H), 3.55 (d, J = 7.2 Hz, 1H), 1.34 (s, 18H); **¹³C NMR** (100 MHz, DMSO- d_6) δ 198.9, 159.2, 152.7, 144.3, 141.3, 139.0, 133.3, 133.1, 132.7, 129.4, 129.2, 128.9, 128.7, 127.0, 126.0, 123.0, 119.3, 113.0, 111.7, 76.1, 62.1, 61.9, 54.8, 51.6, 34.5, 30.4; **HRMS (ESI)** m/z calcd for $[M + H]^+$ $C_{38}H_{44}NO_3$ 562.3316, found 562.3324.

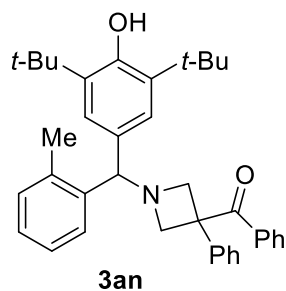


(1-((3-Chlorophenyl)(3,5-di-tert-butyl-4-hydroxyphenyl)methyl)-3-phenylazetidin-3-yl)(phenyl)methanone (3al). White solid; 79.3 mg, 70% yield, PE/EA = 20/1 as the eluent; mp = 155–

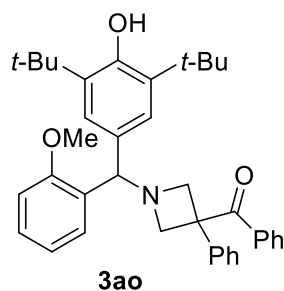
156 °C; $^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ 7.69 (d, $J = 7.5$ Hz, 2H), 7.54–7.50 (m, 3H), 7.39 (t, $J = 7.6$ Hz, 6H), 7.33–7.24 (m, 3H), 7.18 (s, 2H), 6.87 (s, 1H), 4.44 (s, 1H), 3.74 (t, $J = 7.6$ Hz, 2H), 3.63 (d, $J = 7.6$ Hz, 1H), 3.56 (d, $J = 7.2$ Hz, 1H), 1.34 (s, 18H); $^{13}\text{C NMR}$ (100 MHz, $\text{DMSO-}d_6$) δ 198.8, 152.9, 145.3, 141.2, 139.2, 133.21, 133.15, 132.9, 132.1, 130.4, 129.2, 129.0, 128.7, 127.0, 126.9, 126.0, 125.8, 123.1, 75.3, 62.0, 61.7, 51.5, 34.5, 30.4; **HRMS (ESI)** m/z calcd for $[\text{M} + \text{H}]^+$ $\text{C}_{37}\text{H}_{41}\text{ClNO}_2$ 566.2820, found 566.2827.



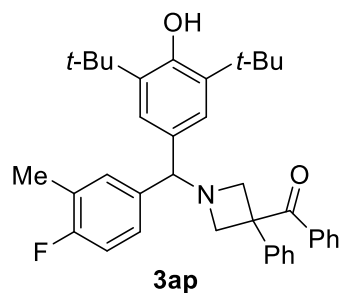
(1-((3-Bromophenyl)(3,5-di-*tert*-butyl-4-hydroxyphenyl)methyl)-3-phenylazetidin-3-yl)(phenyl)methanone (3am). White solid; 90.4 mg, 74% yield, PE/EA = 20/1 as the eluent; mp = 150–151 °C; $^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ 7.69 (d, $J = 7.4$ Hz, 2H), 7.55–7.50 (m, 4H), 7.43–7.36 (m, 6H), 7.28–7.23 (m, 2H), 7.17 (s, 2H), 6.88 (s, 1H), 4.43 (s, 1H), 3.74 (t, $J = 7.6$ Hz, 2H), 3.63 (m, 6H), 7.28–7.23 (m, 2H), 7.17 (s, 2H), 6.88 (s, 1H), 4.43 (s, 1H), 3.74 (t, $J = 7.6$ Hz, 2H), 3.63 (d, $J = 7.2$ Hz, 1H), 3.55 (d, $J = 7.2$ Hz, 1H), 1.34 (s, 18H); $^{13}\text{C NMR}$ (100 MHz, $\text{DMSO-}d_6$) δ 198.8, 153.0, 145.6, 141.2, 139.2, 133.19, 133.16, 132.1, 130.7, 129.79, 129.75, 129.2, 129.0, 128.7, 127.0, 126.2, 126.0, 123.1, 121.6, 75.3, 62.0, 61.8, 51.5, 34.5, 30.4; **HRMS (ESI)** m/z calcd for $[\text{M} + \text{H}]^+$ $\text{C}_{37}\text{H}_{41}\text{BrNO}_2$ 610.2315, found 610.2319.



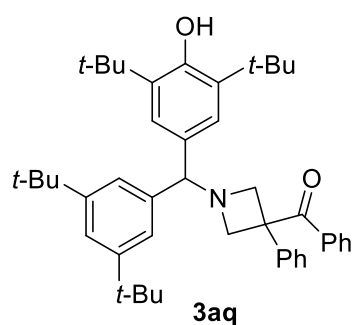
(1-((3,5-Di-*tert*-butyl-4-hydroxyphenyl)(*o*-tolyl)methyl)-3-phenylazetid-3-yl)(phenyl)methanone (3an). White solid; 80.8 mg, 74% yield, PE/EA = 20/1 as the eluent; mp = 161–162 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 7.68 (d, *J* = 7.2 Hz, 2H), 7.61 (d, *J* = 8.0 Hz, 1H), 7.54–7.50 (m, 3H), 7.39 (t, *J* = 7.6 Hz, 4H), 7.28–7.25 (m, 1H), 7.21–7.16 (m, 3H), 7.06 (d, *J* = 4.0 Hz, 2H), 6.85 (s, 1H), 4.59 (s, 1H), 3.71 (d, *J* = 7.2 Hz, 1H), 3.60 (q, *J* = 7.2 Hz, 3H), 2.32 (s, 3H), 1.32 (s, 18H); ¹³C NMR (100 MHz, THF-*d*₈) δ 199.2, 153.8, 143.1, 141.5, 137.5, 136.1, 135.0, 133.2, 132.3, 131.0, 130.1, 129.5, 128.9, 127.4, 126.8, 126.7, 126.6, 125.2, 74.1, 63.4, 62.8, 52.4, 35.0, 30.5, 19.9; **HRMS (ESI)** *m/z* calcd for [M + H]⁺ C₃₈H₄₄NO₂ 546.3367, found 546.3373.



(1-((3,5-Di-*tert*-butyl-4-hydroxyphenyl)(2-methoxyphenyl)methyl)-3-phenylazetid-3-yl)(phenyl)methanone (3ao). White solid; 100.0 mg, 89% yield, PE/EA = 10/1 as the eluent; mp = 175–176 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 7.68 (d, *J* = 7.6 Hz, 2H), 7.55–7.50 (m, 3H), 7.46 (d, *J* = 7.2 Hz, 1H), 7.41–7.37 (m, 4H), 7.29–7.23 (m, 3H), 7.15–7.11 (m, 1H), 6.92–6.88 (m, 2H), 6.78 (s, 1H), 4.76 (s, 1H), 3.78 (s, 3H), 3.70 (d, *J* = 7.6 Hz, 1H), 3.64–3.57 (m, 3H), 1.33 (s, 18H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ 198.9, 156.4, 152.6, 141.5, 138.7, 133.3, 133.2, 132.5, 130.5, 129.2, 129.0, 128.7, 127.5, 127.0, 126.5, 126.0, 123.3, 120.7, 111.0, 68.4, 62.4, 62.0, 55.3, 51.5, 34.5, 30.4; **HRMS (ESI)** *m/z* calcd for [M + H]⁺ C₃₈H₄₄NO₃ 562.3316, found 562.3322.

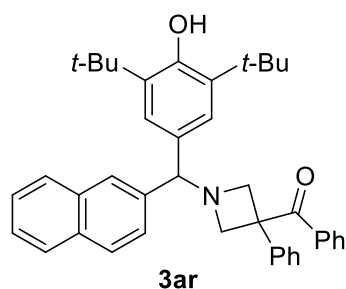


(1-((3,5-Di-*tert*-butyl-4-hydroxyphenyl)(4-fluoro-3-methylphenyl)methyl)-3-phenylazetidin-3-yl)(phenyl)methanone (3ap). White solid; 76.7 mg, 68% yield, PE/EA = 20/1 as the eluent; mp = 146–147 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 7.68 (d, *J* = 7.2 Hz, 2H), 7.53–7.50 (m, 3H), 7.40–7.36 (m, 4H), 7.28–7.21 (m, 3H), 7.16 (s, 2H), 7.02 (t, *J* = 8.8 Hz, 1H), 6.81 (s, 1H), 4.34 (s, 1H), 3.72 (dd, *J* = 16.4, 7.6 Hz, 2H), 3.60 (d, *J* = 7.2 Hz, 1H), 3.52 (d, *J* = 7.6 Hz, 1H), 2.17 (s, 3H), 1.34 (s, 18H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ 198.8, 159.6 (d, *J* = 240.0 Hz), 152.8, 141.3, 139.1, 138.5 (d, *J* = 3.0 Hz), 133.3, 133.1, 132.7, 130.2 (d, *J* = 4.0 Hz), 129.2, 128.9, 128.7, 126.9, 126.1, 126.0, 123.9 (d, *J* = 17.0 Hz), 123.0, 114.8 (d, *J* = 22.0 Hz), 75.6, 62.1, 61.9, 51.5, 34.5, 30.4, 14.2 (d, *J* = 2.0 Hz); ¹⁹F NMR (376 MHz, DMSO-*d*₆) δ -120.26 – -120.32 (m, 1F); **HRMS (ESI)** *m/z* calcd for [M + H]⁺ C₃₈H₄₃F₂NO₂ 564.3272, found 564.3281.

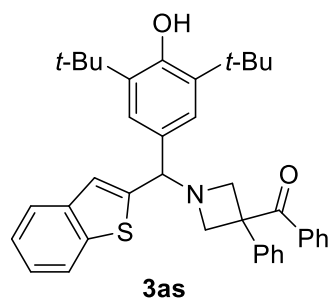


(1-((3,5-Di-*tert*-butyl-4-hydroxyphenyl)(3,5-di-*tert*-butylphenyl)methyl)-3-phenylazetidin-3-yl)(phenyl)methanone (3aq). White solid; 78.6 mg, 61% yield, PE/EA = 20/1 as the eluent; mp = 202–203 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 7.68 (d, *J* = 7.6 Hz, 2H), 7.55–7.50 (m, 3H), 7.38 (t, *J* = 7.6 Hz, 4H), 7.28–7.25 (m, 3H), 7.22 (s, 2H), 7.17 (s, 1H), 6.80 (s, 1H), 4.35 (s, 1H), 3.67–

3.58 (m, 4H), 1.33 (s, 18H), 1.23 (s, 18H); ^{13}C NMR (100 MHz, THF-*d*₈) δ 199.5, 153.7, 150.9, 143.4, 143.1, 137.6, 135.2, 134.1, 133.1, 130.0, 129.4, 128.9, 127.4, 126.7, 124.2, 122.1, 121.0, 78.7, 63.4, 63.3, 52.5, 35.2, 35.0, 31.7, 30.5; **HRMS (ESI)** m/z calcd for $[\text{M} + \text{H}]^+$ C₄₅H₅₈NO₂ 644.4462, found 644.4463.

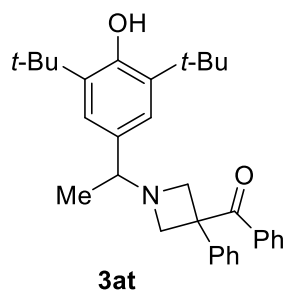


(1-((3,5-Di-*tert*-butyl-4-hydroxyphenyl)(naphthalen-2-yl)methyl)-3-phenylazetidin-3-yl)(phenyl)methanone (3ar). White solid; 91.9 mg, 79% yield, PE/EA = 20/1 as the eluent; mp = 147–148 °C; ^1H NMR (400 MHz, DMSO-*d*₆) δ 7.92–7.89 (m, 2H), δ 7.82–7.79 (m, 2H), 7.70 (d, J = 7.2 Hz, 2H), 7.56–7.36 (m, 10H), 7.29–7.25 (m, 3H), 6.83 (s, 1H), 4.56 (s, 1H), 3.80 (dd, J = 18.4, 7.6 Hz, 2H), 3.68 (d, J = 7.6 Hz, 1H), 3.56 (d, J = 7.2 Hz, 1H), 1.34 (s, 18H); ^{13}C NMR (100 MHz, DMSO-*d*₆) δ 198.8, 152.8, 141.3, 140.3, 139.1, 133.24, 133.16, 133.0, 132.6, 132.3, 129.2, 129.0, 128.7, 128.1, 127.7, 127.4, 127.0, 126.1, 126.0, 125.7, 125.54, 125.50, 123.2, 76.5, 62.2, 62.0, 51.6, 34.5, 30.4; **HRMS (ESI)** m/z calcd for $[\text{M} + \text{H}]^+$ C₄₁H₄₄NO₂ 582.3367, found 582.3376.



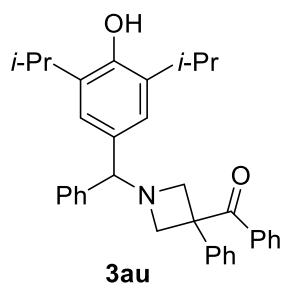
(1-(Benzo[*b*]thiophen-2-yl(3,5-di-*tert*-butyl-4-hydroxyphenyl)methyl)-3-phenylazetidin-3-yl)(

phenyl)methanone (3as). White solid; 77.6 mg, 66% yield, PE/EA = 20/1 as the eluent; mp = 178–179 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 7.85 (d, *J* = 8.0 Hz, 1H), 7.74–7.68 (m, 3H), 7.55–7.50 (m, 3H), 7.41–7.37 (m, 5H), 7.31–7.20 (m, 5H), 6.92 (s, 1H), 4.83 (s, 1H), 3.84 (d, *J* = 7.2 Hz, 1H), 3.77 (d, *J* = 7.2 Hz, 1H), 3.72–3.69 (m, 2H), 1.35 (s, 18H); ¹³C NMR (100 MHz, THF-*d*₈) δ 199.2, 154.2, 149.6, 143.1, 140.7, 140.5, 137.9, 134.9, 133.2, 132.4, 130.1, 129.5, 129.0, 127.5, 126.7, 124.6, 124.4, 124.2, 123.7, 122.8, 120.8, 74.0, 63.4, 63.0, 52.2, 35.1, 30.5; **HRMS (ESI)** *m/z* calcd for [M + H]⁺ C₃₉H₄₂NO₂S 588.2931, found 588.2927.



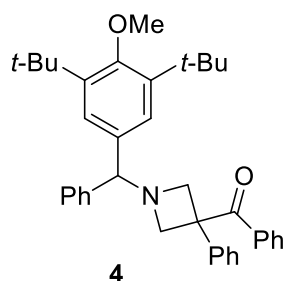
(1-(1-(3,5-Di-*tert*-butyl-4-hydroxyphenyl)ethyl)-3-phenylazetidin-3-yl)(phenyl)methanone

(3at). White solid; 66.7 mg, 71% yield, PE/EA = 8/1 as the eluent; mp = 125–126 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 7.68 (d, *J* = 7.6 Hz, 2H), 7.54–7.50 (m, 3H), 7.41–7.35 (m, 4H), 7.27–7.23 (m, 1H), 7.02 (s, 2H), 6.76 (s, 1H), 3.75–3.67 (m, 3H), 3.55 (d, *J* = 7.2 Hz, 1H), 3.26–3.22 (m, 1H), 1.36 (s, 18H), 1.06 (d, *J* = 6.1 Hz, 3H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ 199.0, 152.6, 141.2, 138.9, 134.1, 133.4, 133.1, 129.2, 128.9, 128.7, 126.9, 126.1, 122.7, 67.1, 62.0, 61.6, 51.4, 34.5, 30.4, 21.6; **HRMS (ESI)** *m/z* calcd for [M + H]⁺ C₃₂H₄₀NO₂ 470.3054, found 470.3062.

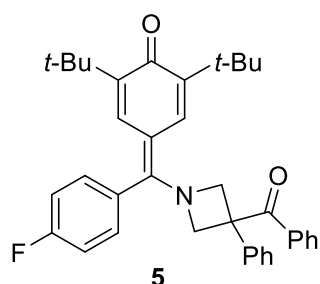


(1-((4-Hydroxy-3,5-diisopropylphenyl)(phenyl)methyl)-3-phenylazetidin-3-yl)(phenyl)metha

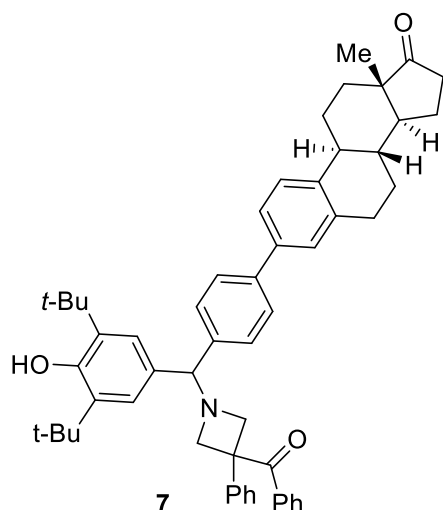
none (3au). White solid; 77.6 mg, 77% yield, PE/EA = 20/1 as the eluent; mp = 123–124 °C; ^1H NMR (400 MHz, DMSO- d_6) δ 7.93 (s, 1H), 7.68 (d, J = 7.6 Hz, 2H), 7.54–7.50 (m, 3H), 7.41–7.37 (m, 6H), 7.26 (t, J = 7.2 Hz, 3H), 7.17–7.14 (m, 1H), 7.04 (s, 2H), 4.37 (s, 1H), 3.70 (d, J = 7.2 Hz, 2H), 3.63 (d, J = 7.2 Hz, 1H), 3.56 (d, J = 7.2 Hz, 1H), 3.27–3.17 (m, 2H), 1.11–1.09 (m, 12H); ^{13}C NMR (100 MHz, DMSO- d_6) δ 198.9, 149.7, 142.8, 141.3, 135.0, 133.3, 133.2, 132.9, 129.2, 129.0, 128.7, 128.4, 127.1, 127.0, 126.8, 126.0, 121.6, 76.3, 62.2, 62.0, 51.5, 26.2, 23.03, 22.98; **HRMS (ESI)** m/z calcd for $[\text{M} + \text{H}]^+$ $\text{C}_{35}\text{H}_{38}\text{NO}_2$ 504.2897, found 504.2905.



(1-((3,5-Di-tert-butyl-4-methoxyphenyl)(phenyl)methyl)-3-phenylazetidin-3-yl)(phenyl)methanone (4). White solid; 101.5 mg, 93% yield, PE/EA = 25/1 as the eluent; mp = 70–71 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.66 (d, J = 7.6 Hz, 2H), 7.56 (d, J = 8.0 Hz, 2H), 7.43–7.35 (m, 5H), 7.31–7.26 (m, 5H), 7.26–7.24 (m, 2H), 7.19–7.15 (m, 1H), 4.31 (s, 1H), 3.78–3.70 (m, 3H), 3.62 (s, 3H), 3.61 (d, J = 6.4 Hz, 1H), 1.38 (s, 18H); ^{13}C NMR (100 MHz, CDCl_3) δ 199.9, 158.3, 143.2, 142.4, 142.0, 135.5, 134.1, 132.7, 129.5, 129.0, 128.4, 128.3, 127.5, 127.0, 126.9, 126.1, 125.3, 77.6, 64.0, 62.7, 62.6, 51.8, 35.7, 32.1; **HRMS (ESI)** m/z calcd for $[\text{M} + \text{H}]^+$ $\text{C}_{38}\text{H}_{44}\text{NO}_2$ 546.3367, found 546.3376.

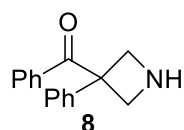


4-((3-Benzoyl-3-phenylazetididin-1-yl)(4-fluorophenyl)methylene)-2,6-di-*tert*-butylcyclohexa-2,5-dien-1-one (5). Yellow solid; 97.5 mg, 89% yield, PE/EA = 3/1 as the eluent; mp = 211–212 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 7.69 (d, *J* = 7.6 Hz, 2H), 7.59–7.38 (m, 12H), 7.33–7.30 (m, 1H), 6.42 (s, 1H), 5.57 (s, 1H), 5.13 (s, 1H), 4.65 (s, 1H), 4.53 (s, 1H), 1.31 (s, 9H), 1.03 (s, 9H); ¹³C NMR (100 MHz, DMSO-*d*₆) δ 196.7, 180.7, 162.5 (d, *J* = 245.0 Hz), 160.1, 141.6, 140.0, 139.4, 133.7, 132.4, 131.5, 131.0 (d, *J* = 9.0 Hz), 129.4, 129.3, 129.2 (d, *J* = 3.0 Hz), 128.9, 127.8, 126.1, 125.5, 115.8 (d, *J* = 21.0 Hz), 107.0, 51.9, 35.0, 34.4, 29.3, 29.1; ¹⁹F NMR (376 MHz, DMSO-*d*₆) δ -111.32 – -111.37 (m, 1F); HRMS (ESI) *m/z* calcd for [M + H]⁺ C₃₇H₃₉FNO₂ 548.2959, found 548.2961.



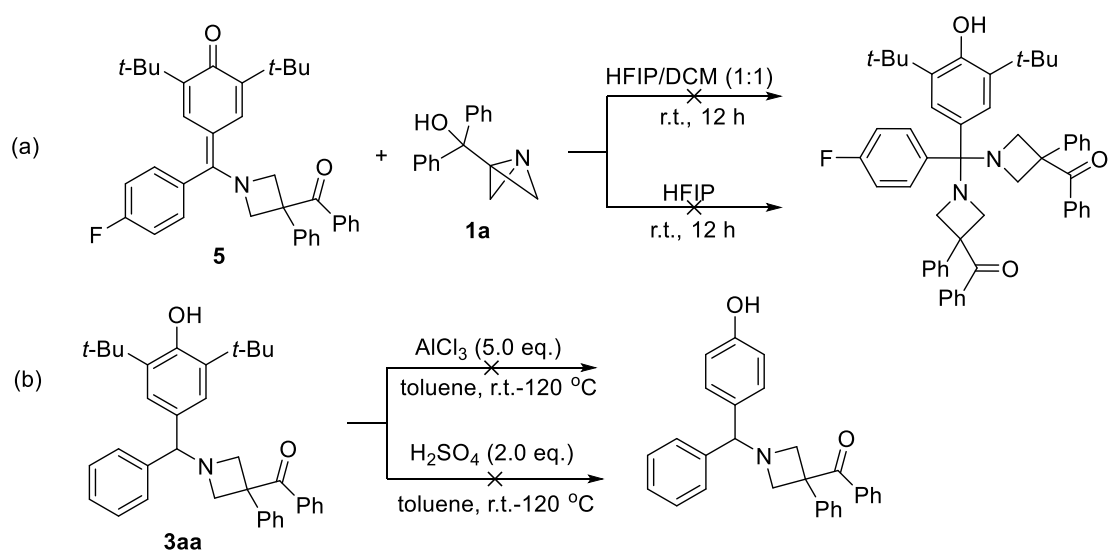
(8*R*,9*S*,13*S*,14*S*)-3-(4-((3-benzoyl-3-phenylazetididin-1-yl)(3,5-di-*tert*-butyl-4-hydroxyphenyl)methyl)phenyl)-13-methyl-6,7,8,9,11,12,13,14,15,16-decahydro-17*H*-cyclopenta[*a*]phenanthren-17-one (7). Yellow solid; 149.0 mg, 95% yield, PE/EA = 4/1 as the eluent; mp = 131–132 °C; ¹H NMR (400 MHz, DMSO-*d*₆) δ 7.70 (d, *J* = 7.2 Hz, 2H), 7.56–7.50 (m, 5H), 7.44 (d, *J* = 8.0 Hz, 2H), 7.39 (t, *J* = 7.6 Hz, 4H), 7.33 (s, 2H), 7.28–7.25 (m, 2H), 7.22 (s, 2H), 6.84 (s, 1H), 4.42 (s, 1H), 3.77 (dd, *J* = 16.8, 7.6 Hz, 2H), 3.64 (d, *J* = 7.6 Hz, 1H), 3.56 (d, *J* = 7.2 Hz, 1H), 2.90–2.87 (m, 2H), 2.47–2.38 (m, 2H), 2.30–2.23 (m, 1H), 2.11–1.94 (m, 4H), 1.79–1.76 (m, 1H), 1.59–1.49

(m, 5H), 1.35 (s, 18H), 0.83 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 199.9, 152.8, 142.0, 141.5, 139.6, 138.6, 136.7, 135.6, 134.1, 132.7, 132.4, 129.5, 129.0, 128.4, 127.7, 127.6, 127.0, 126.9, 126.1, 125.7, 124.4, 123.8, 77.4, 62.7, 62.6, 51.7, 50.5, 48.0, 44.3, 38.1, 35.8, 34.3, 31.6, 30.3, 29.5, 26.5, 25.7, 21.6, 13.8; HRMS (ESI) m/z calcd for $[\text{M} + \text{H}]^+$ $\text{C}_{55}\text{H}_{62}\text{NO}_3$ 784.4724, found 784.4720.



Phenyl(3-phenylazetididin-3-yl)methanone (8). Colorless liquid; 66.9 mg, 94% yield, DCM/MeOH = 10/1 as the eluent; ^1H NMR (400 MHz, CDCl_3) δ 7.66 (d, $J = 7.6$ Hz, 2H), 7.50–7.43 (m, 3H), 7.39–7.31 (m, 4H), 7.27–7.24 (m, 1H), 4.48 (d, $J = 8.4$ Hz, 2H), 4.03 (d, $J = 8.4$ Hz, 2H), 2.85 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 199.5, 141.2, 133.6, 132.9, 129.5, 129.2, 128.4, 127.2, 125.6, 56.8, 56.2. Physical and spectral properties of this material were identical to those previously reported in literature.³

Unsuccessful transformations



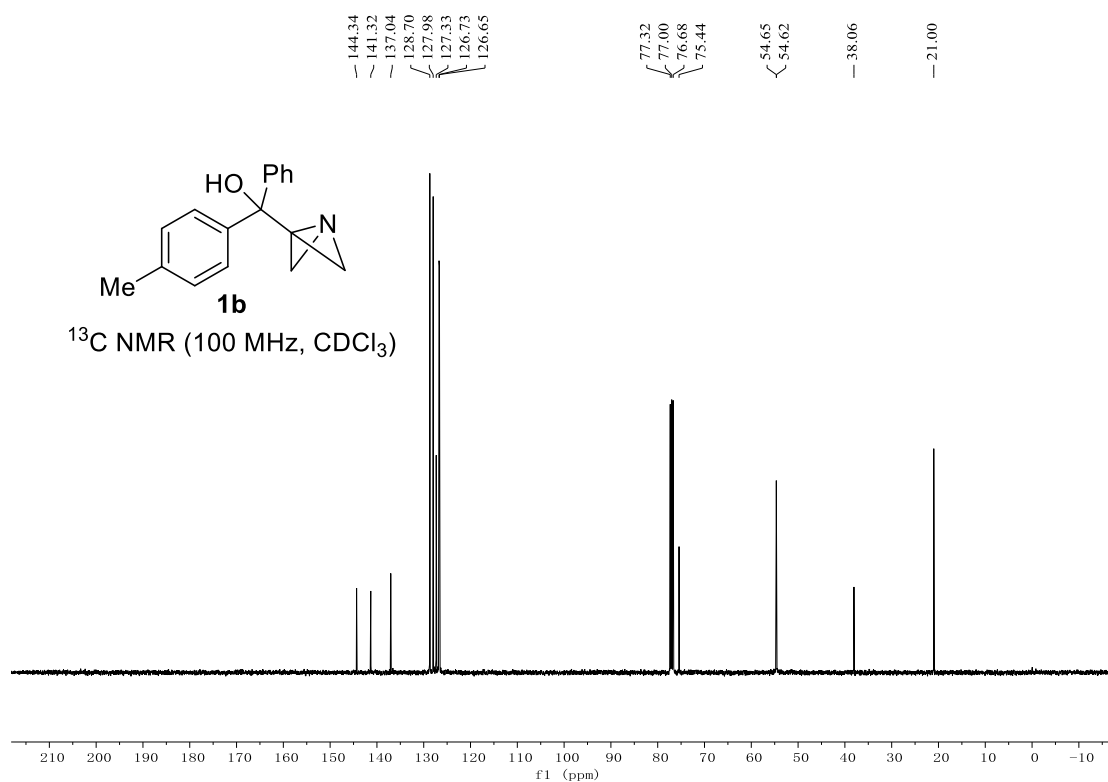
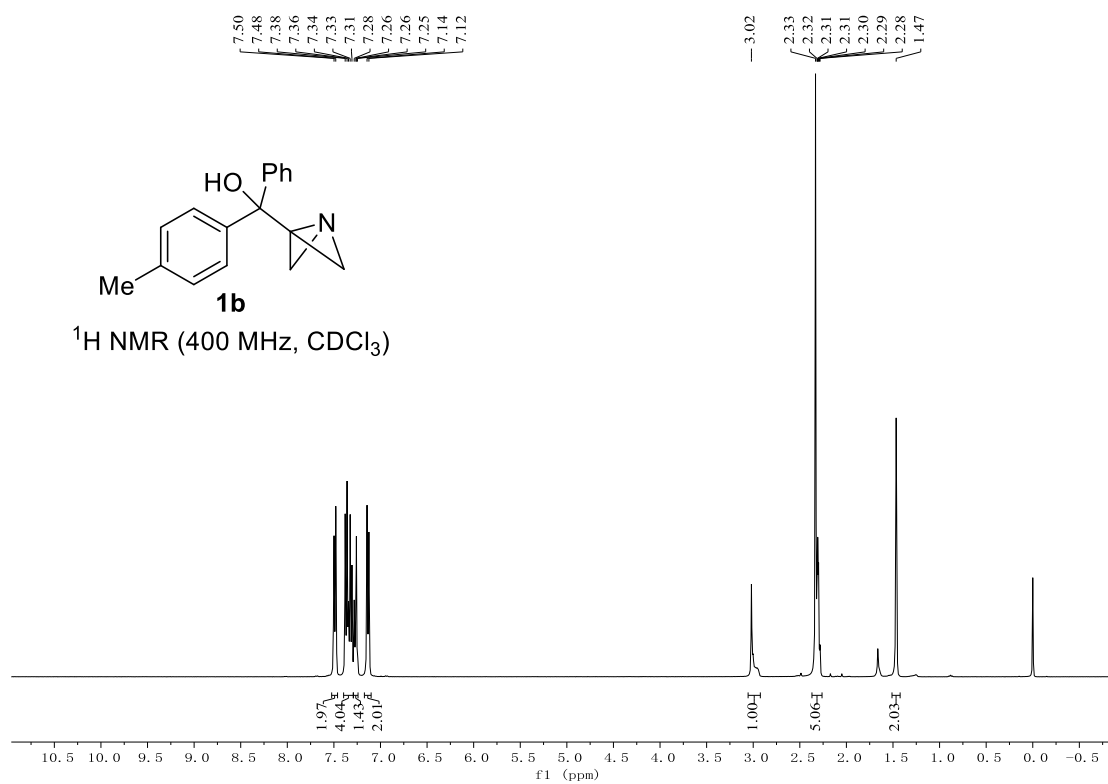
References

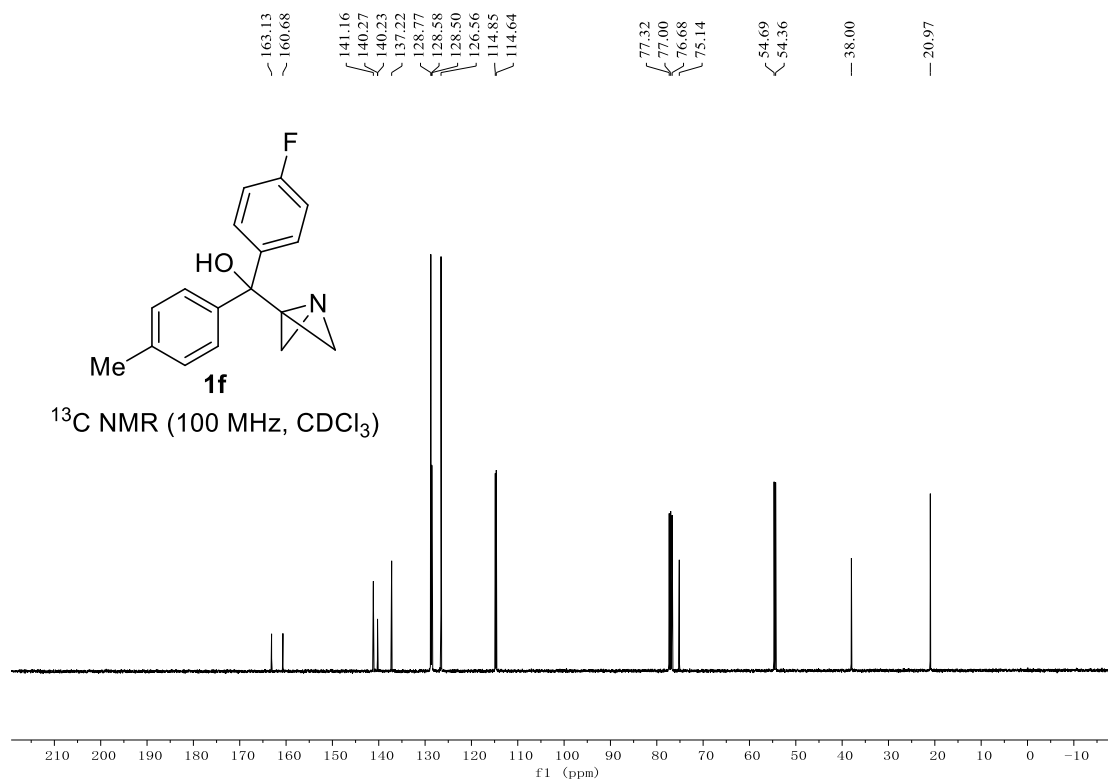
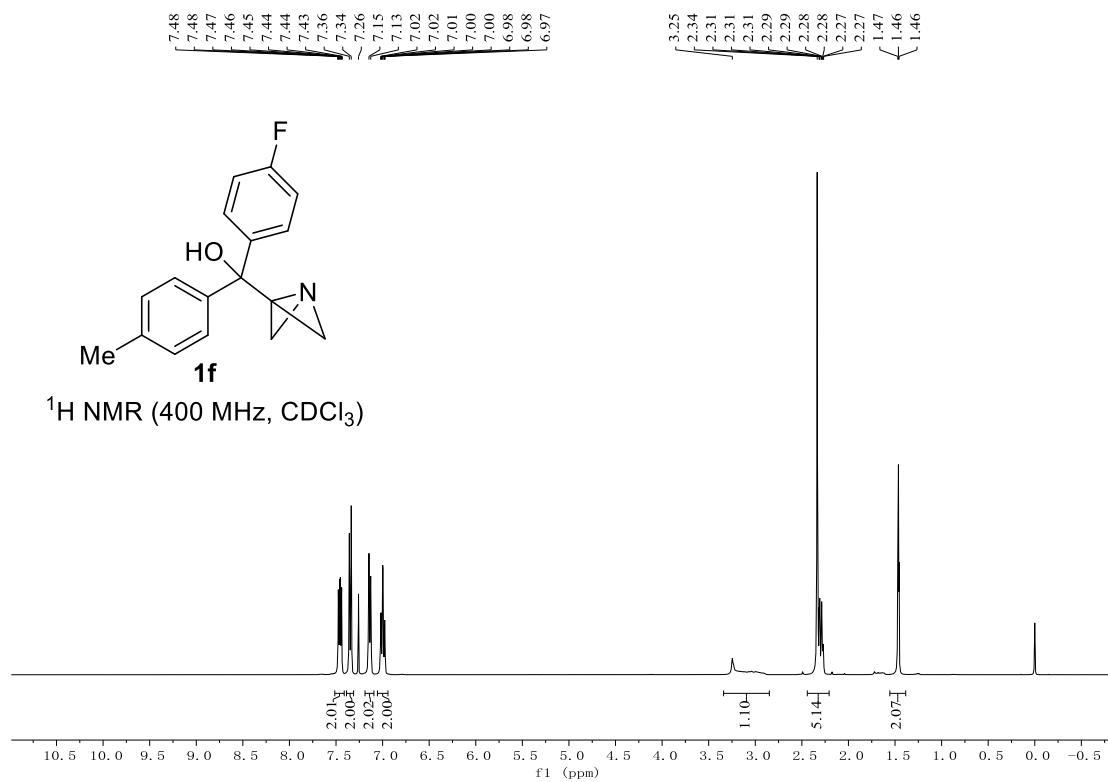
(1) (a) P. Musci, T. V. Keutz, F. Belaj, L. Degennaro, D. Cantillo, C. O. Kappe and R. Luisi, *Angew. Chem., Int. Ed.*, 2021, **60**, 6395–6399; (b) C. H. U. Gregson, A. Noble and V. K. Aggarwal, *Angew. Chem., Int. Ed.*, 2021, **60**, 7360–7365; (c) V. Jaiswal, S. Mondal, B. Singh, V. P. Singh and J. Saha, *Angew. Chem., Int. Ed.*, 2023, **62**, e202304471; (d) B. Singh, P. Sasmal, A. Taites, S. Hazra and J. Saha, *Org. Lett.*, 2024, **26**, 9558–9563.

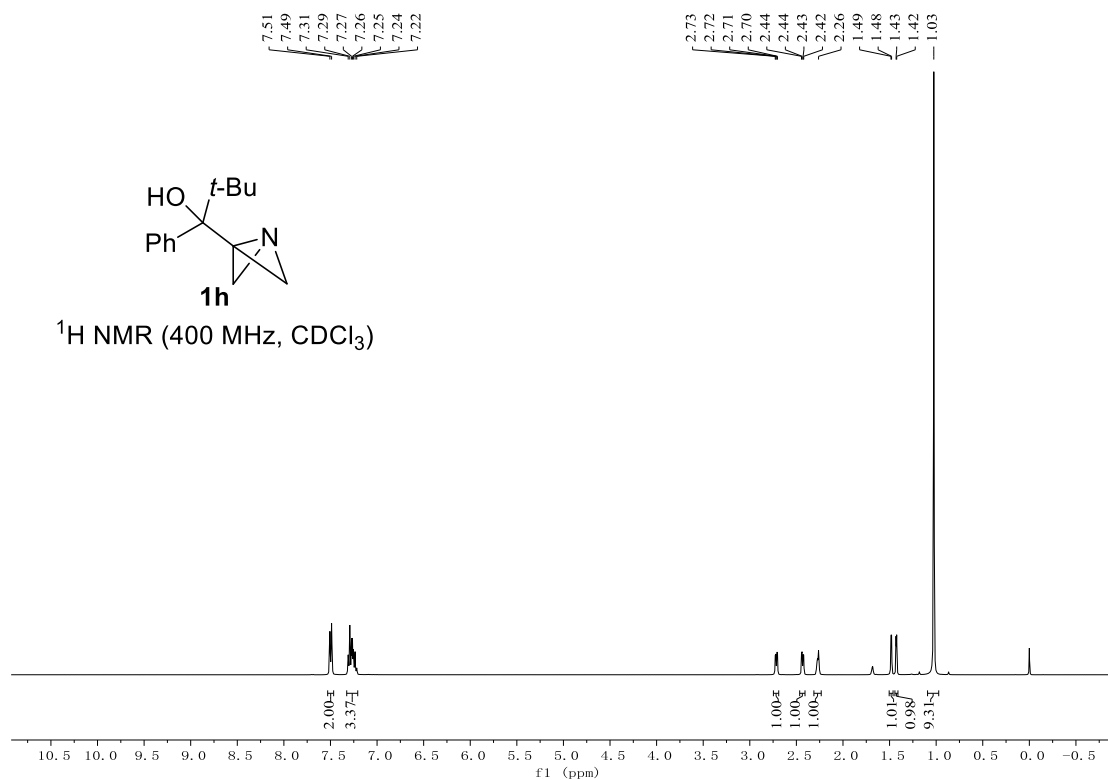
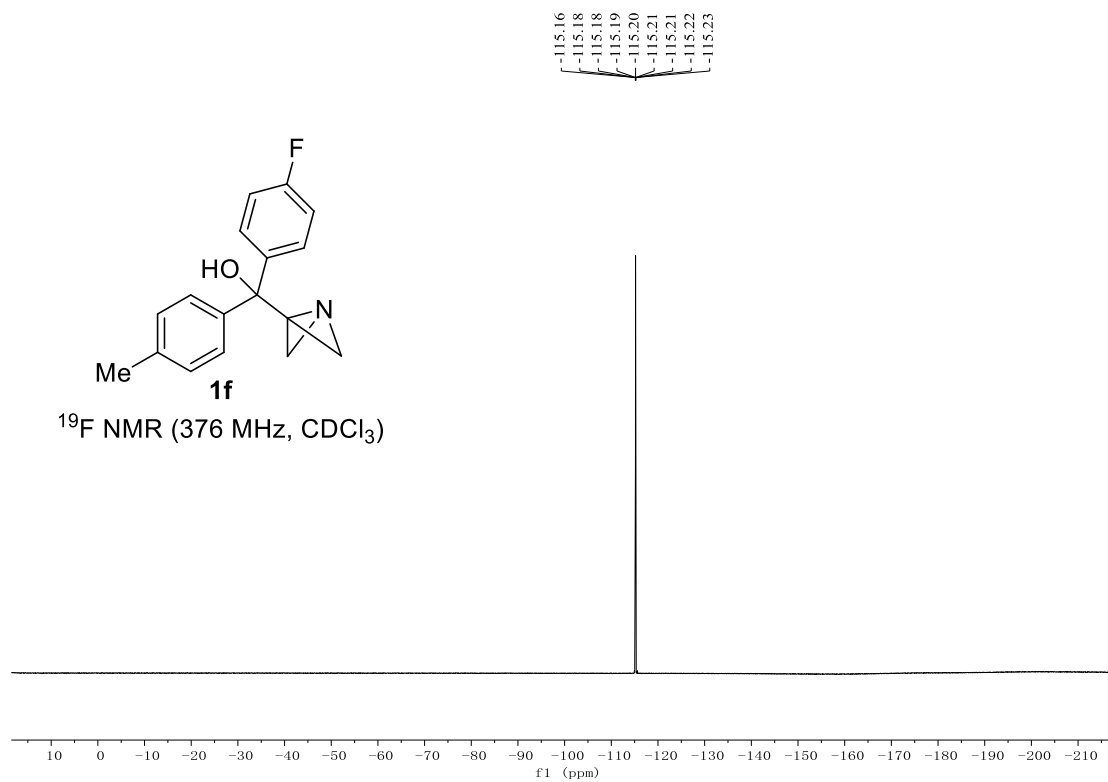
(2) (a) Z. Yuan, W. Wei, A. Lin and H. Yao, *Org. Lett.*, 2016, **18**, 3370–3373; (b) X. Li, G. Gao, S. He and Q. Song, *Org. Chem. Front.*, 2021, **8**, 4543–4548; (c) J. Zhang, M. Sun, K. Gao, H. Wu, J. Li, Z. Wang and J. Yang, *J. Org. Chem.*, 2024, **89**, 2847–2857; (d) Y.-T. Mei, H. Zhang, Y. Jiang, Y.-J. Gu, J.-L. Deng, D. Yang, L.-H. Jing and M.-S. Shi, *Chem. Commun.*, 2024, **60**, 8589–8592.

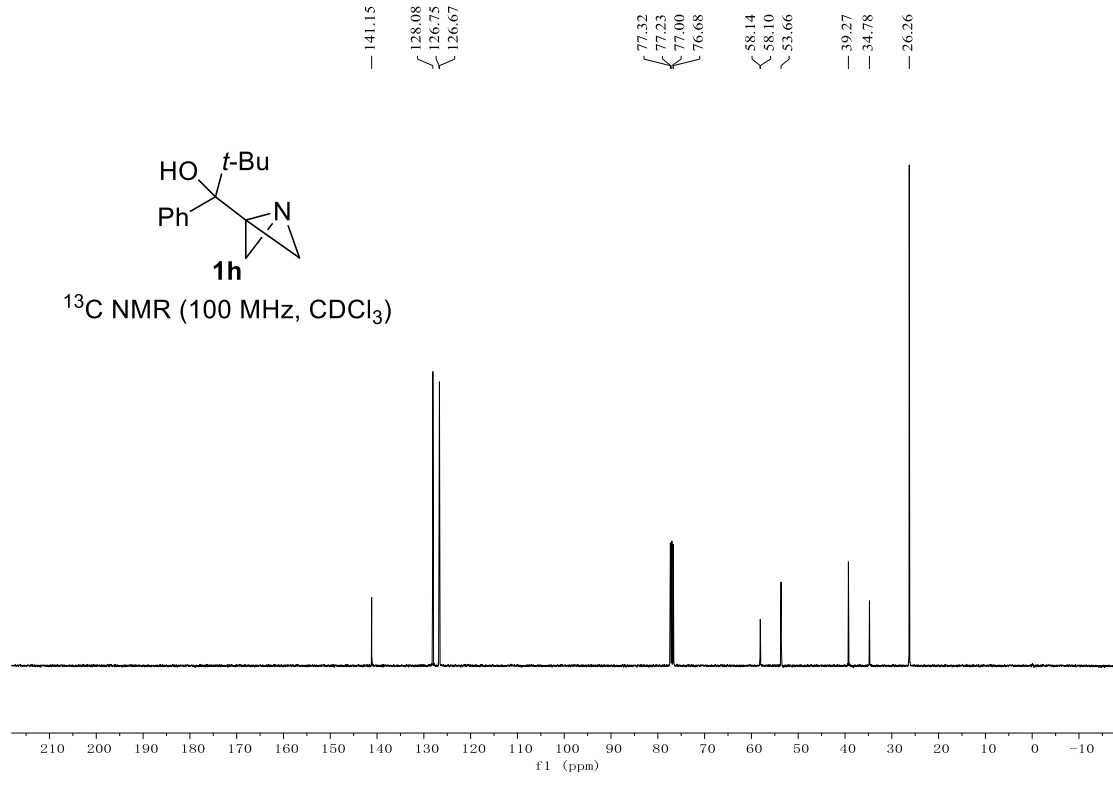
(3) V. Jaiswal, S. Mondal, B. Singh, V. P. Singh and J. Saha, *Angew. Chem., Int. Ed.*, 2023, **62**, e202304471.

Copies of ^1H , ^{13}C and ^{19}F NMR spectra

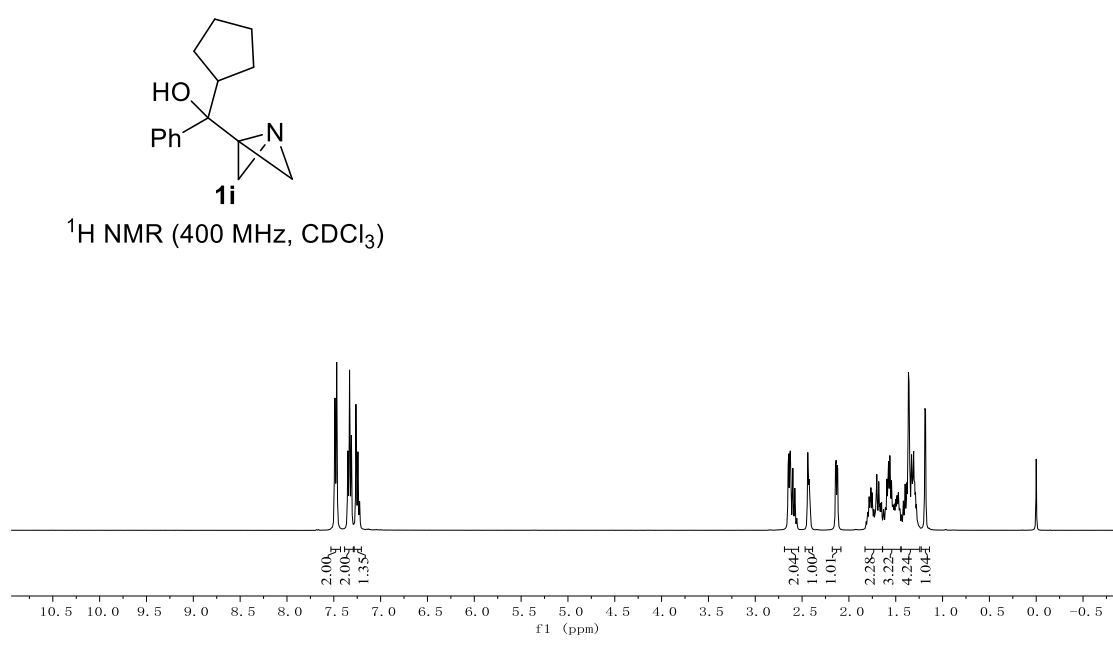


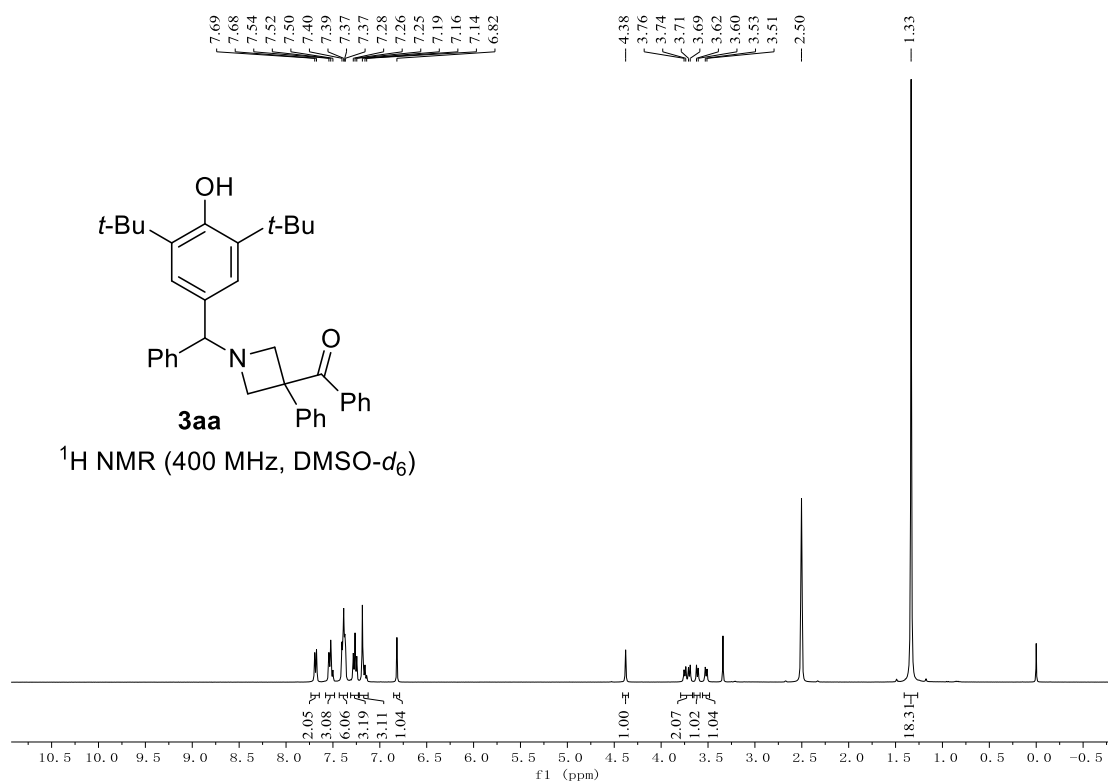
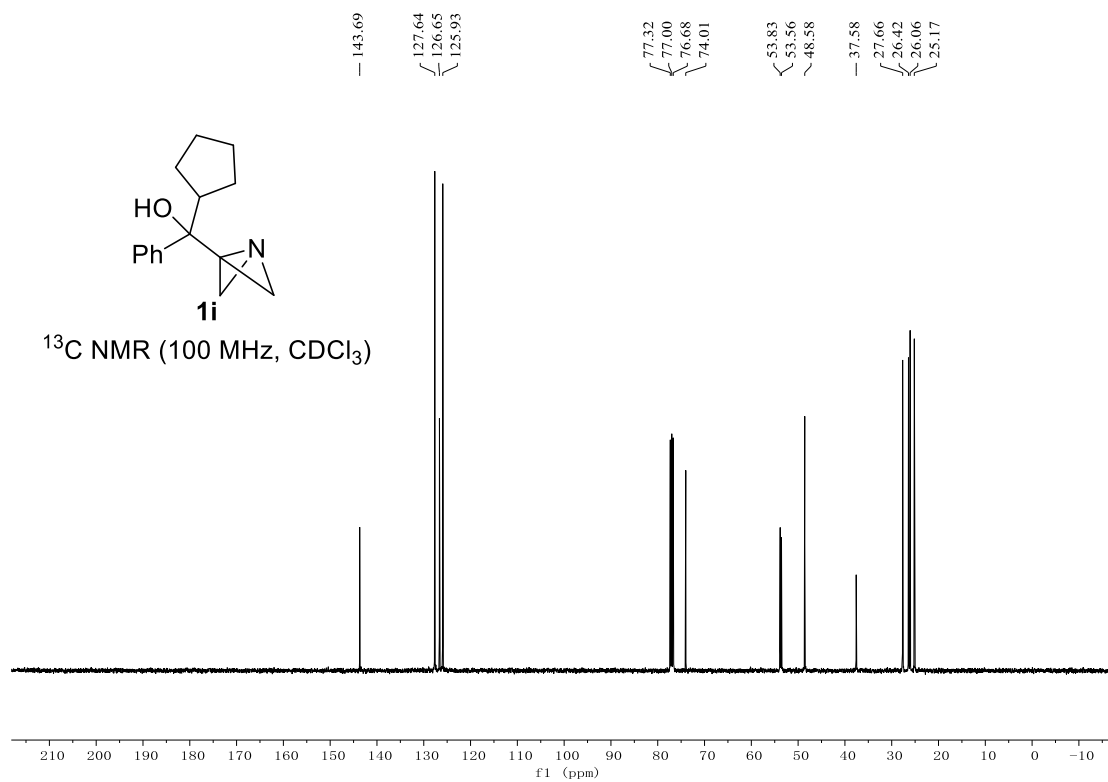


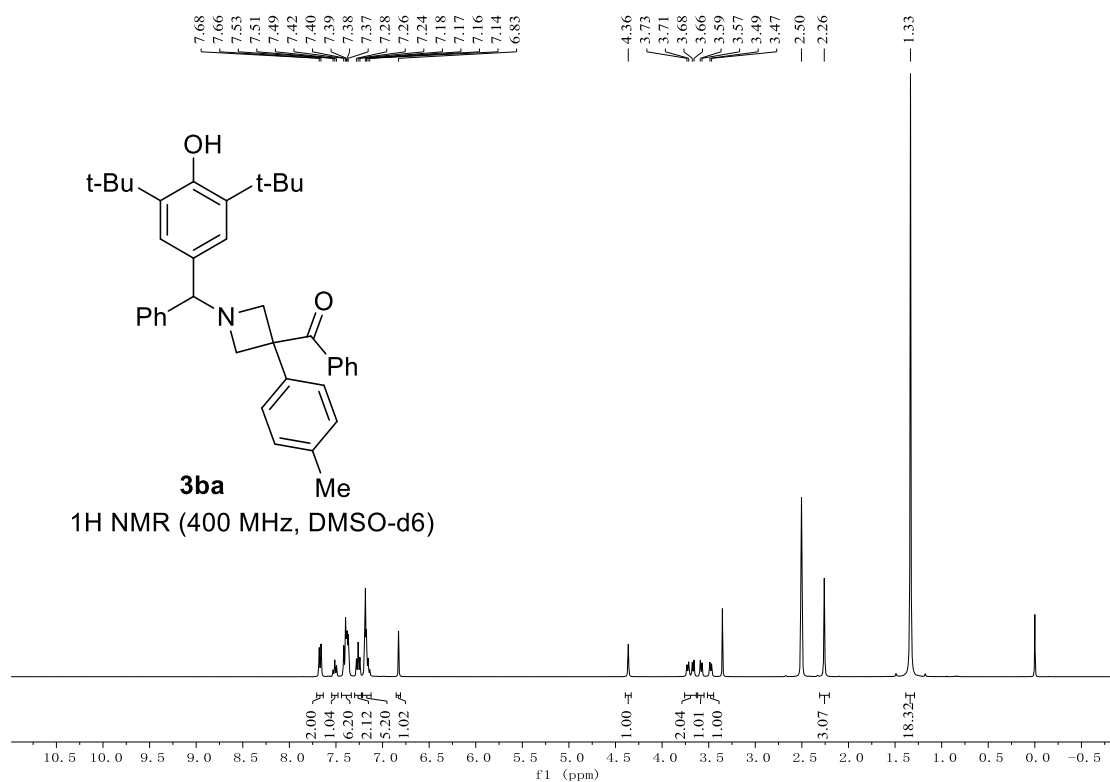
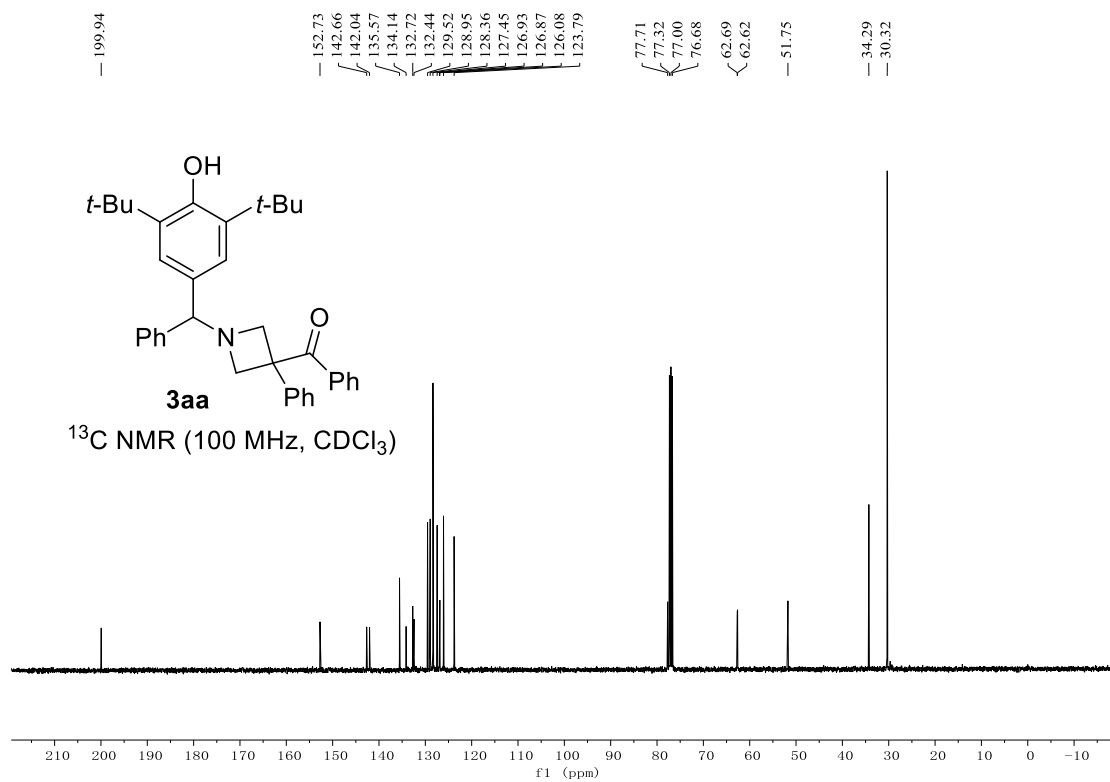


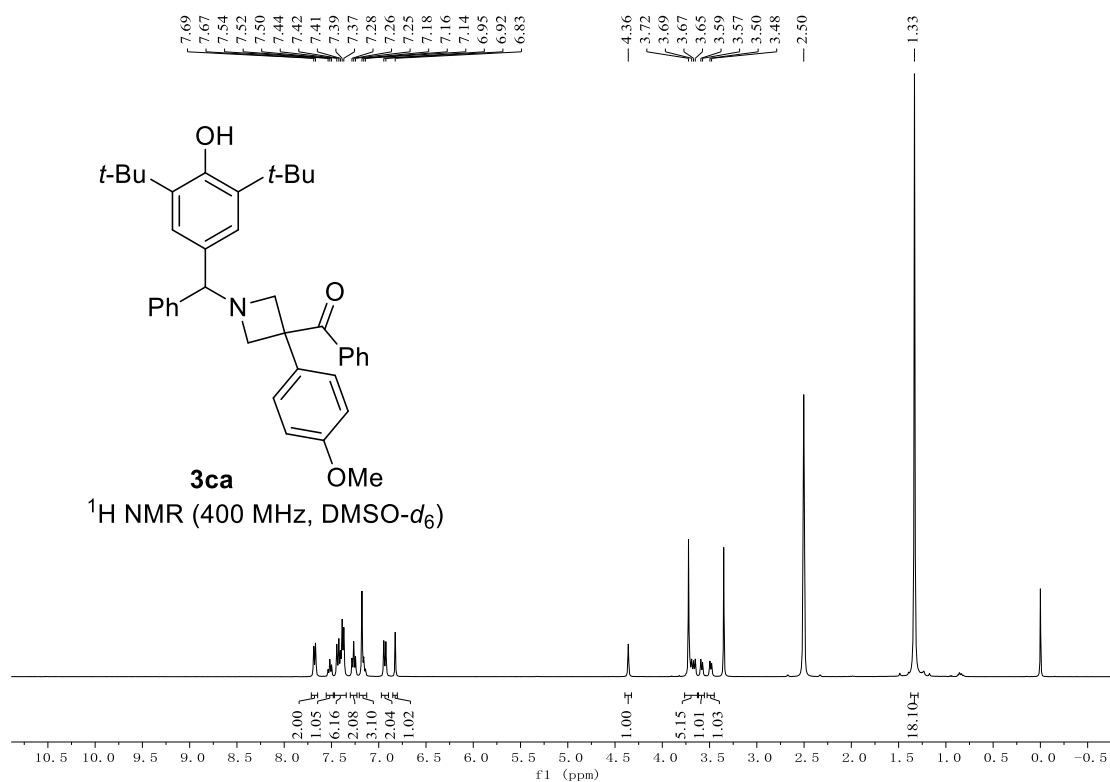
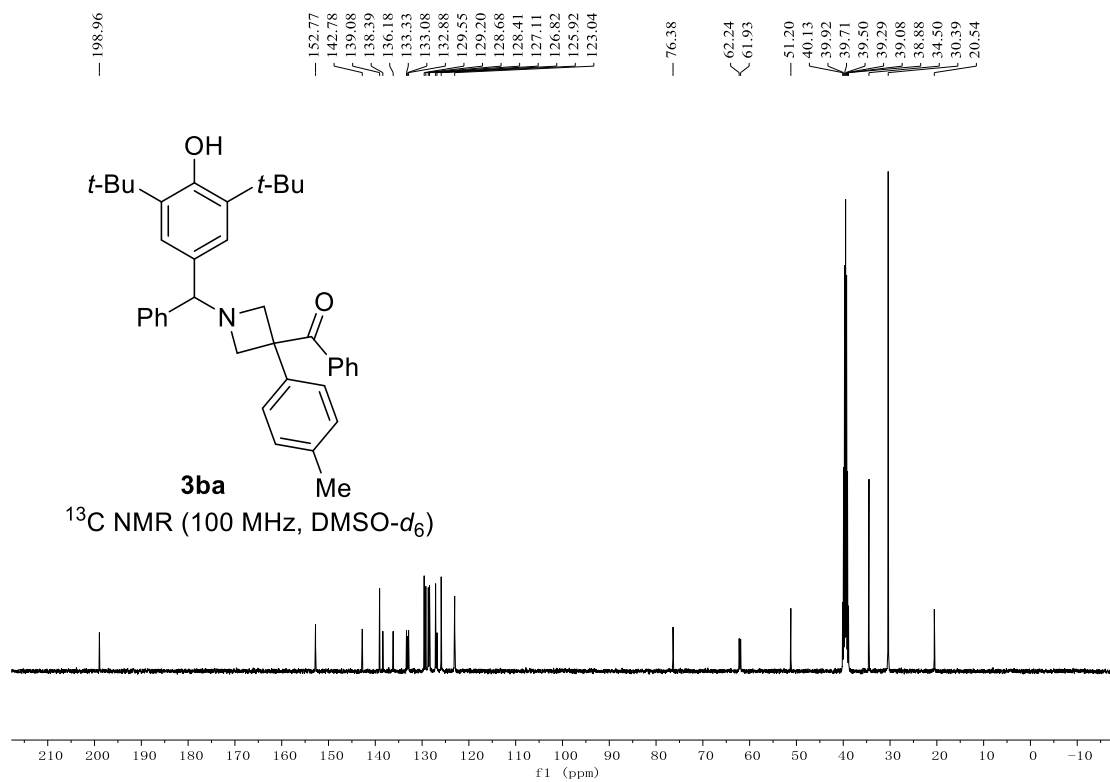


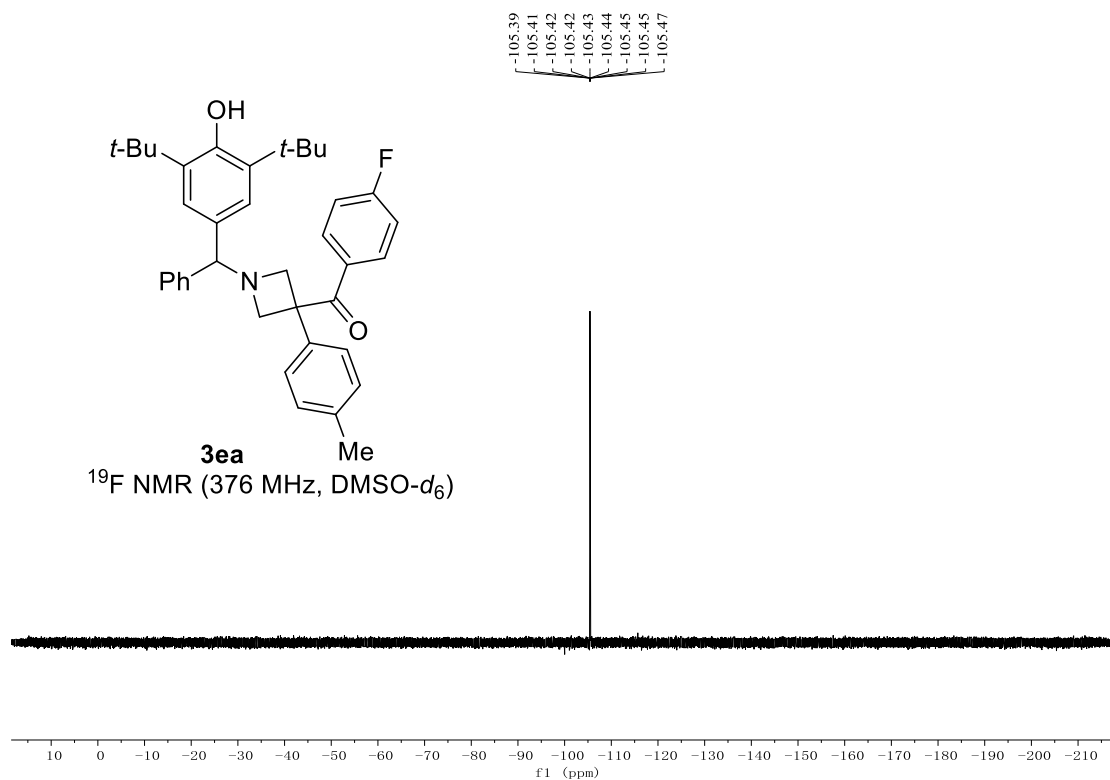
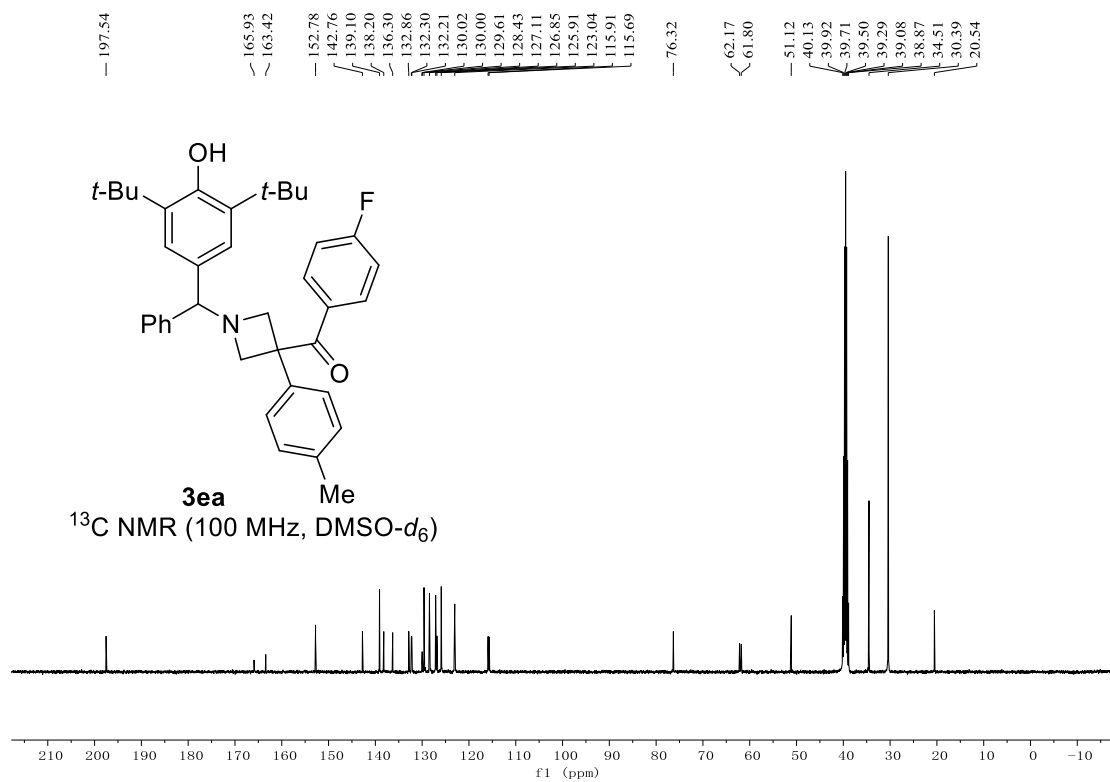
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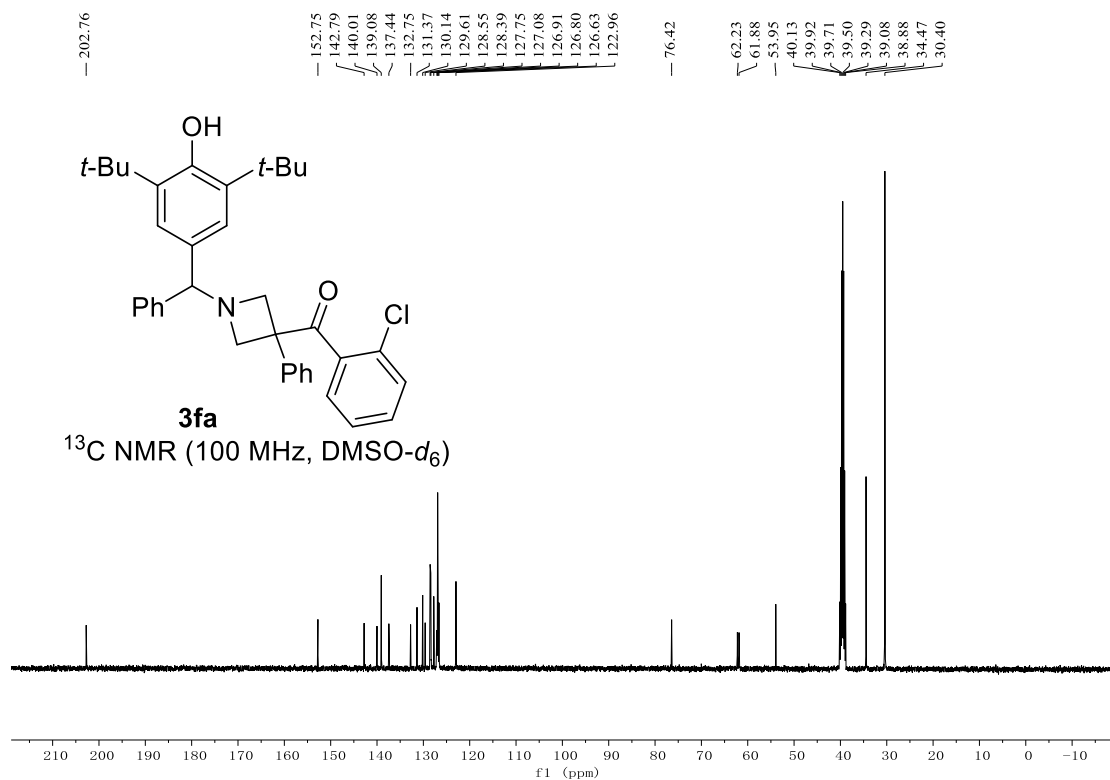
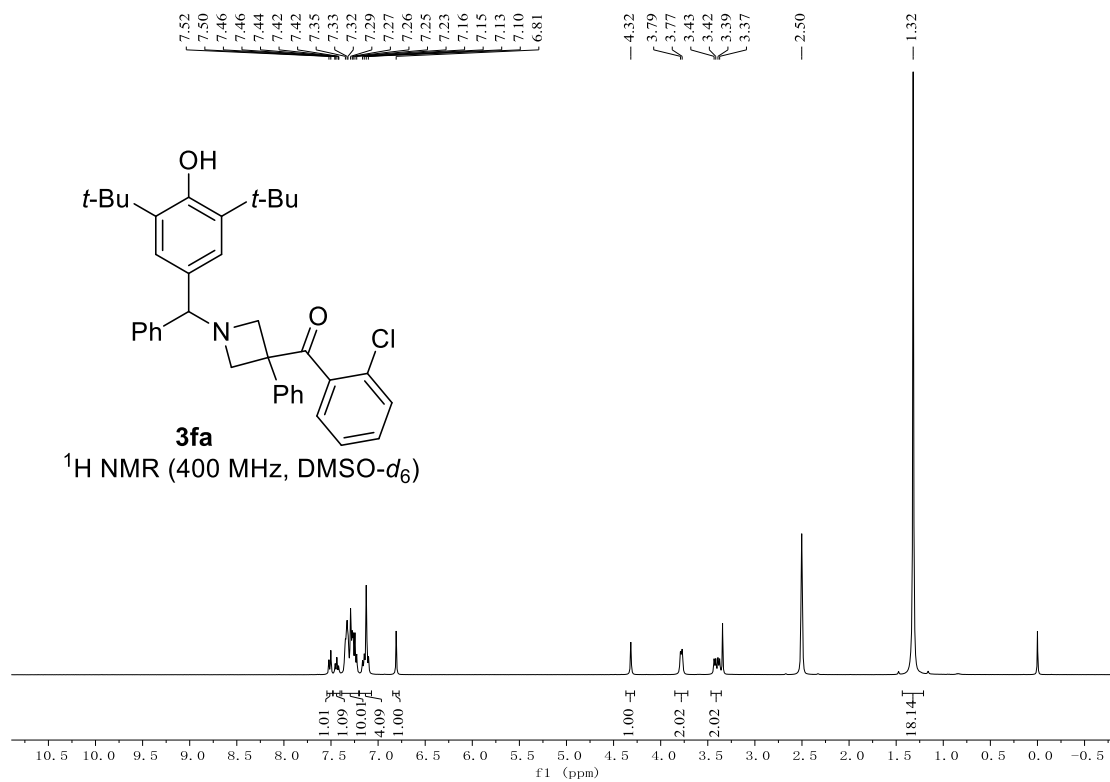


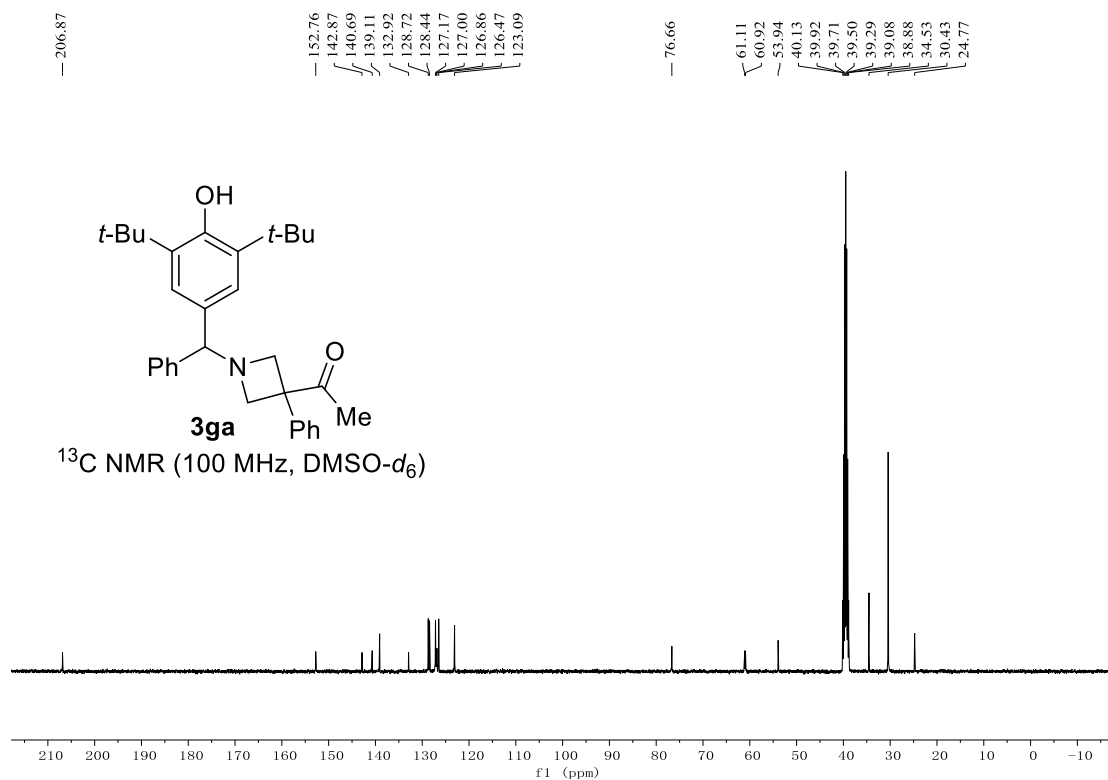
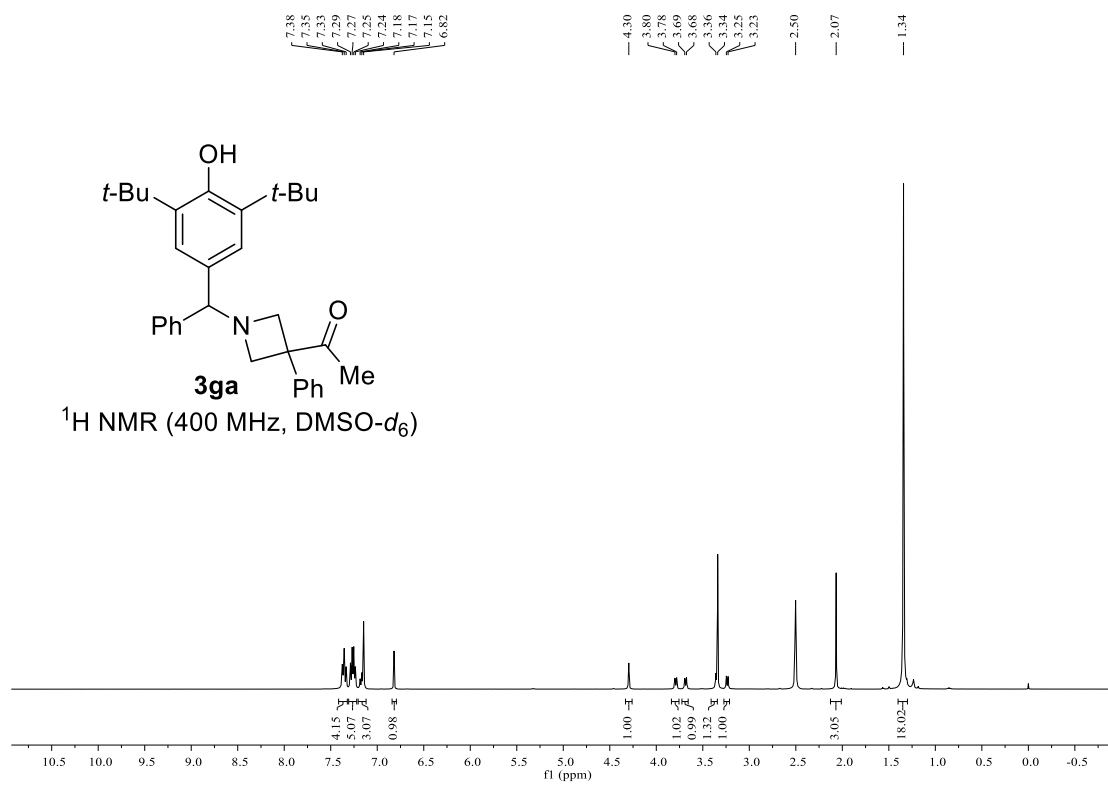


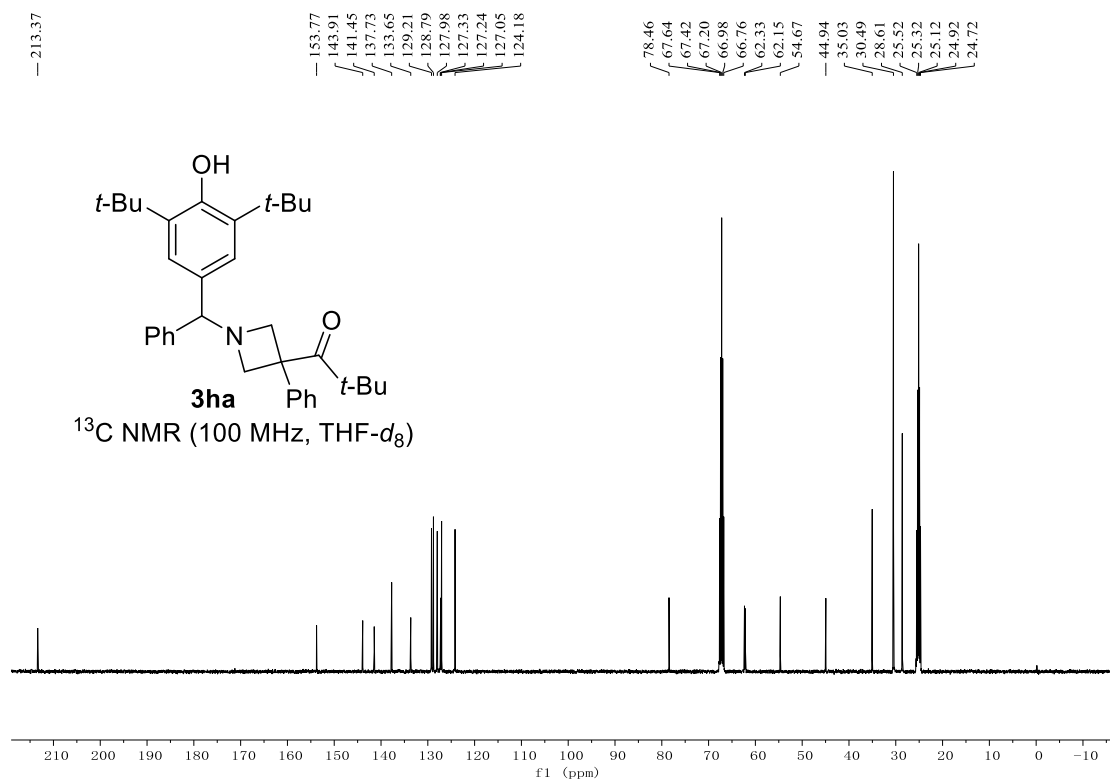
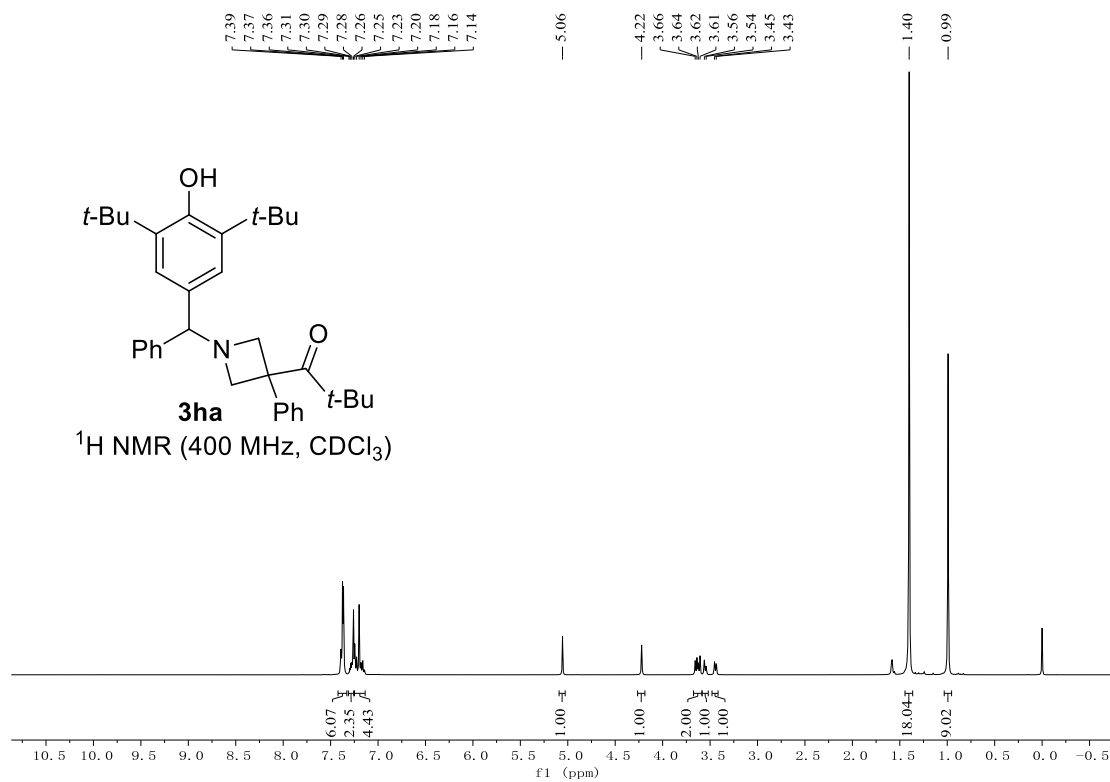


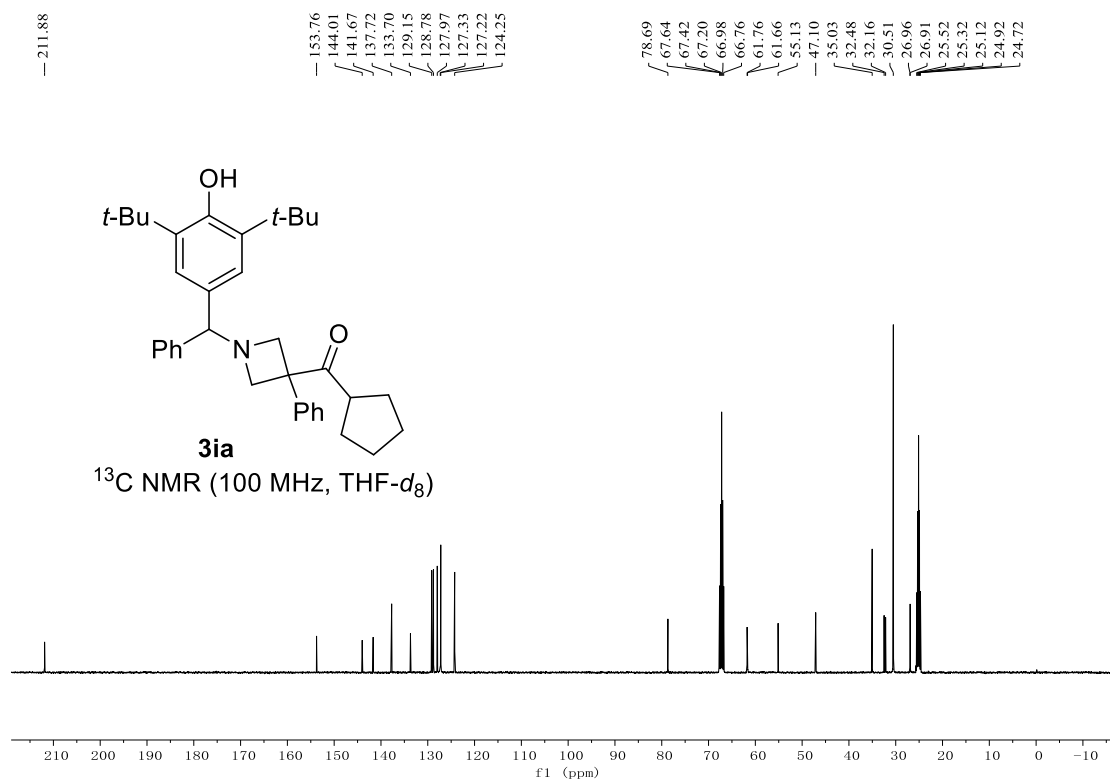
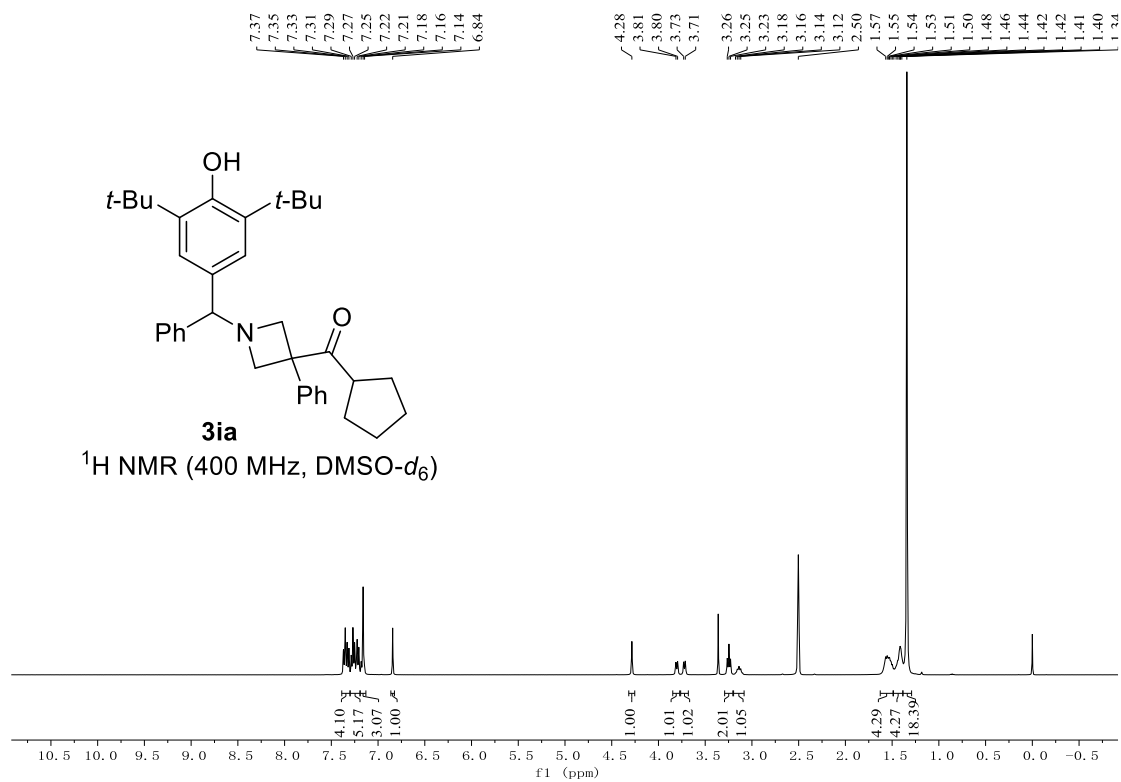


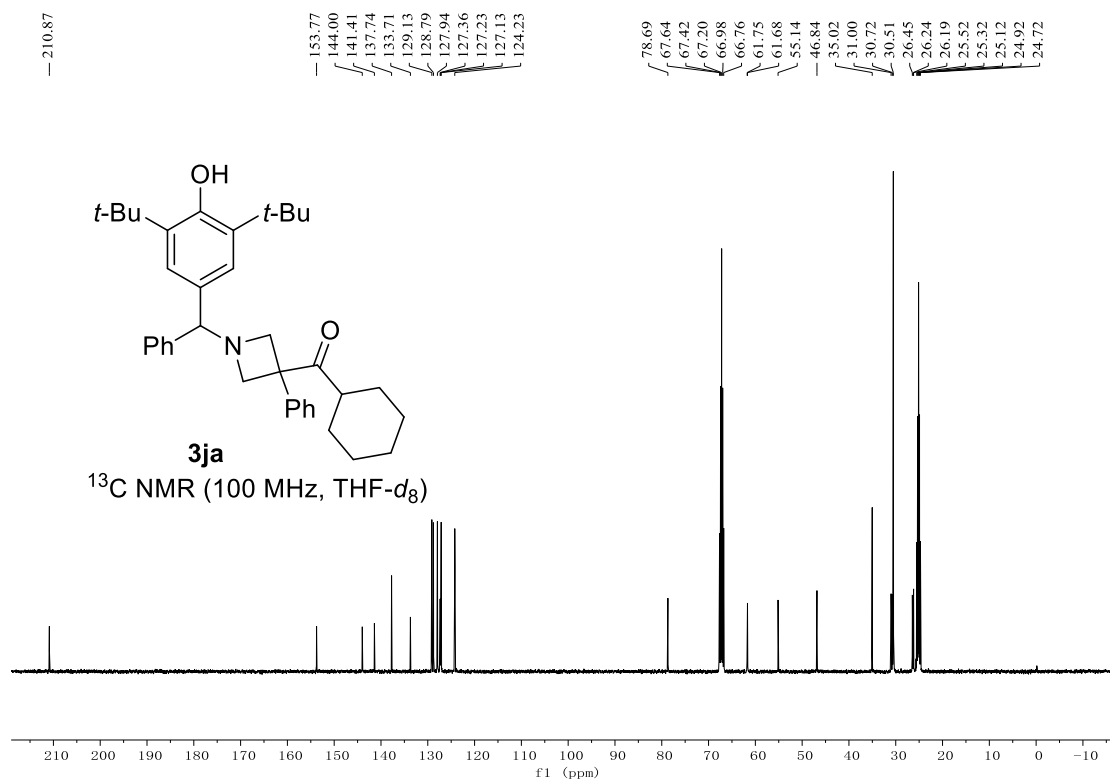
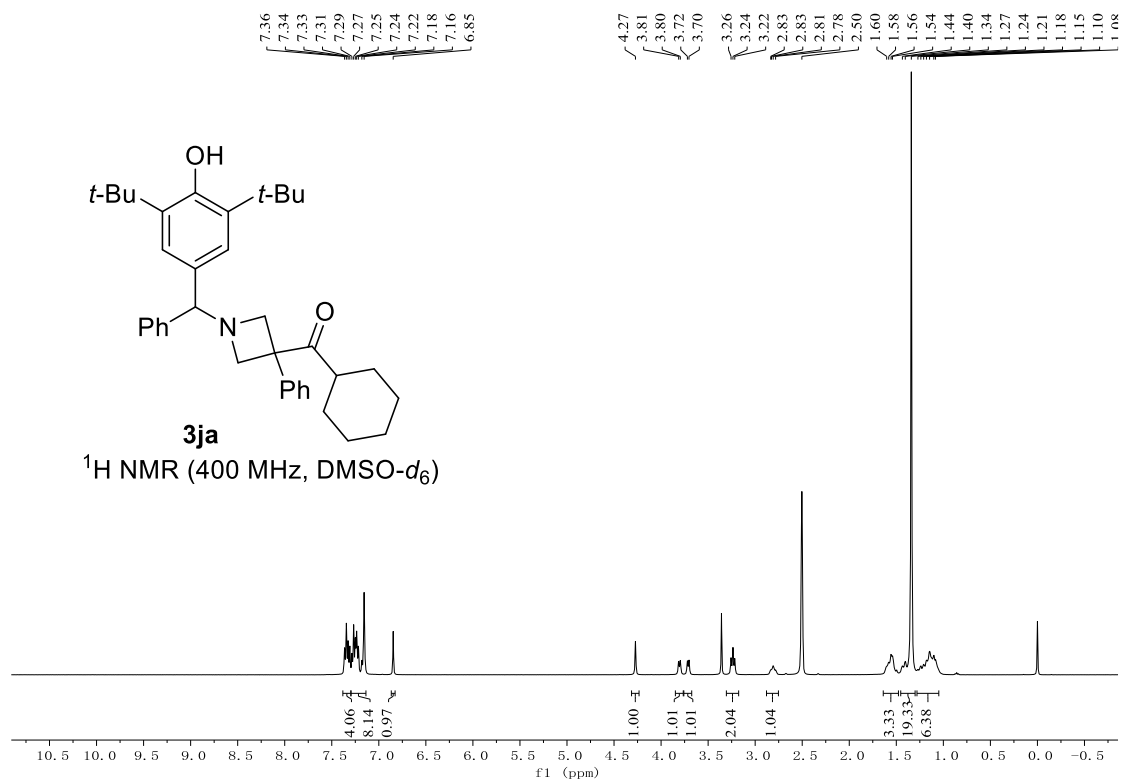


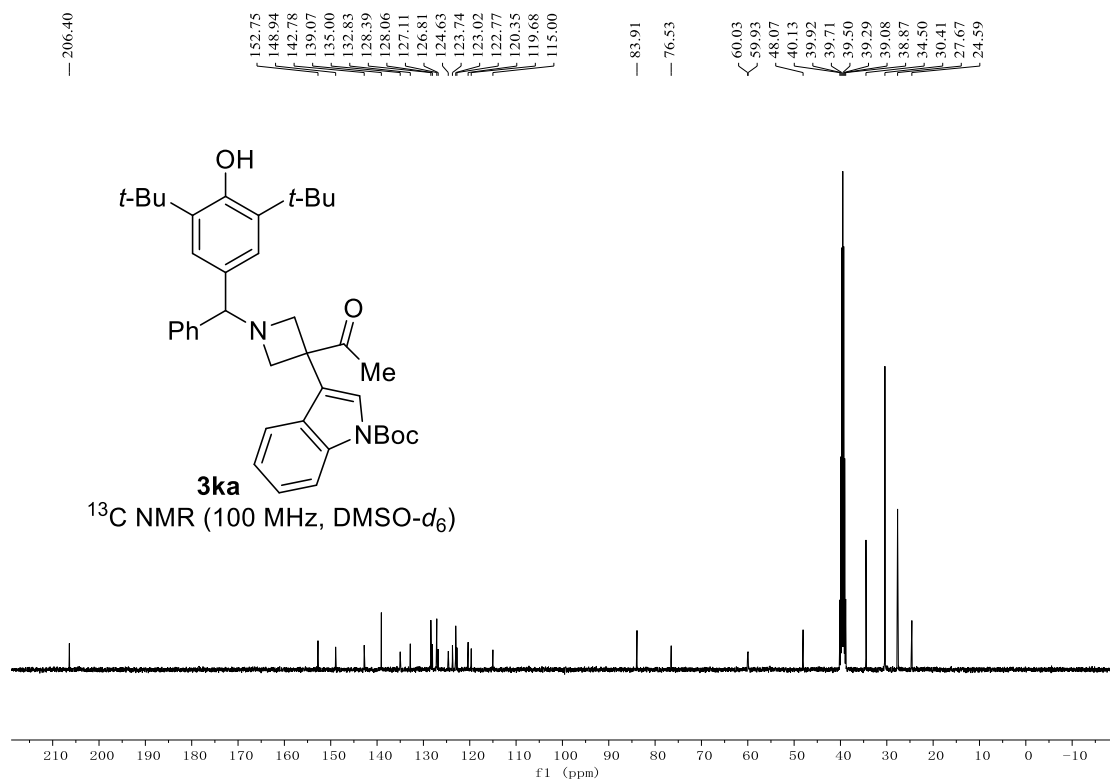
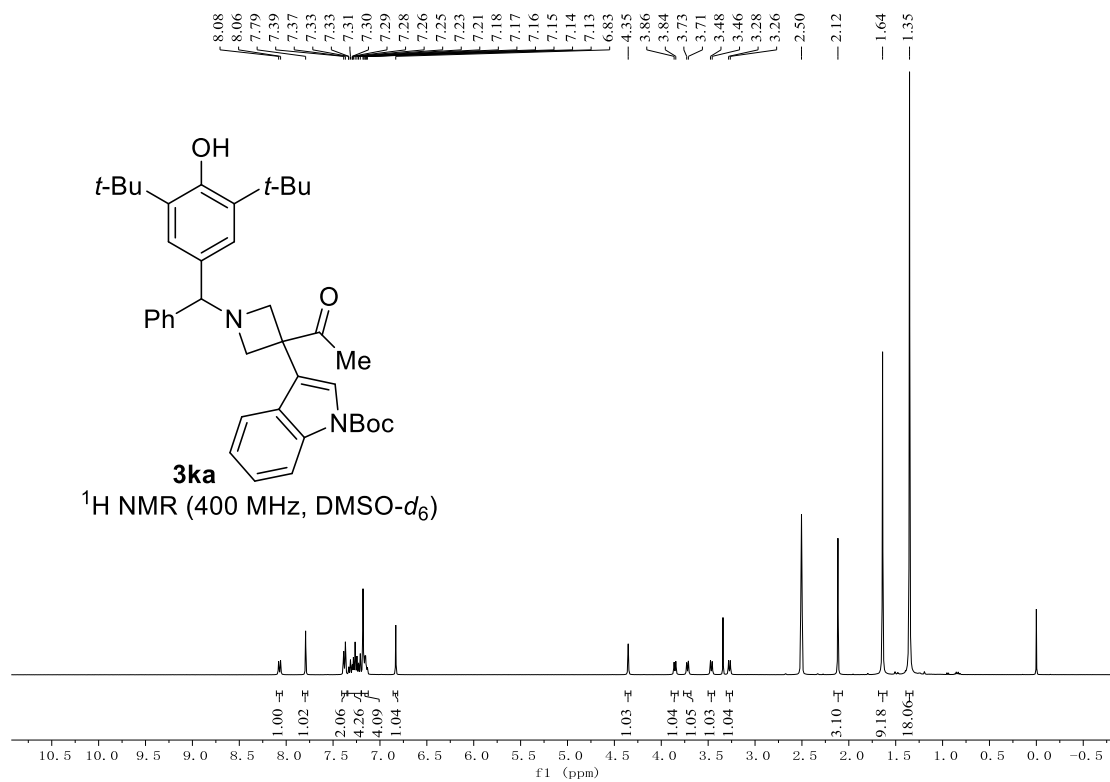


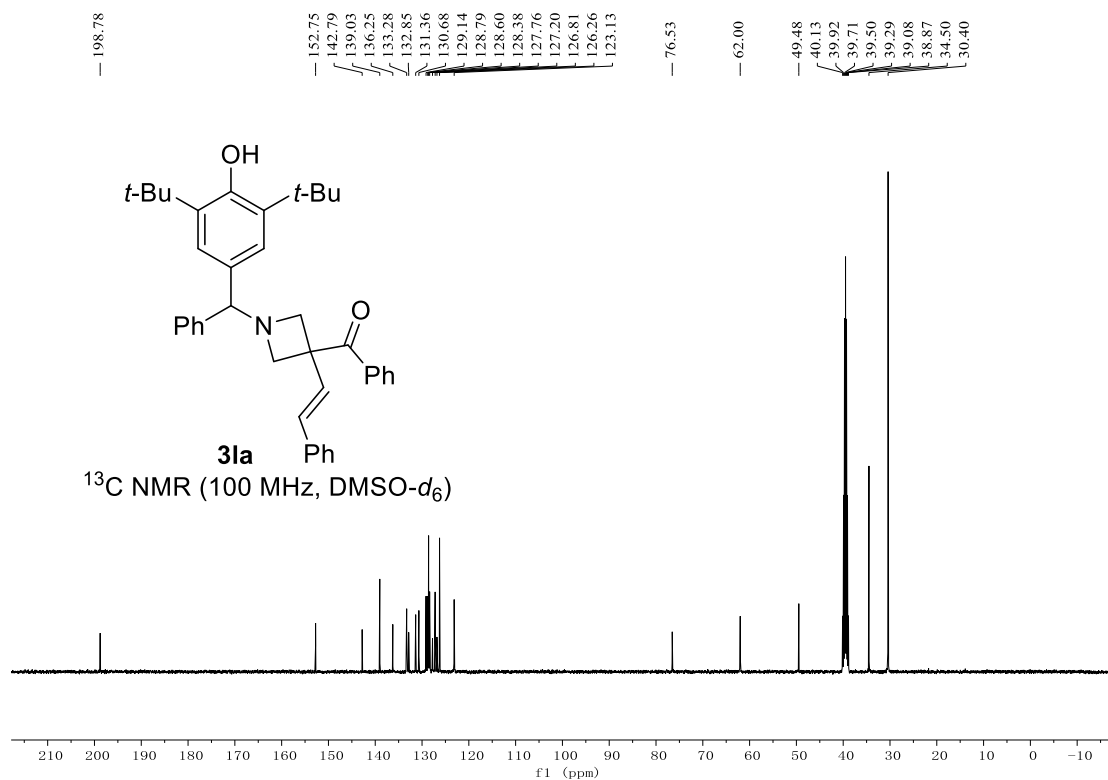
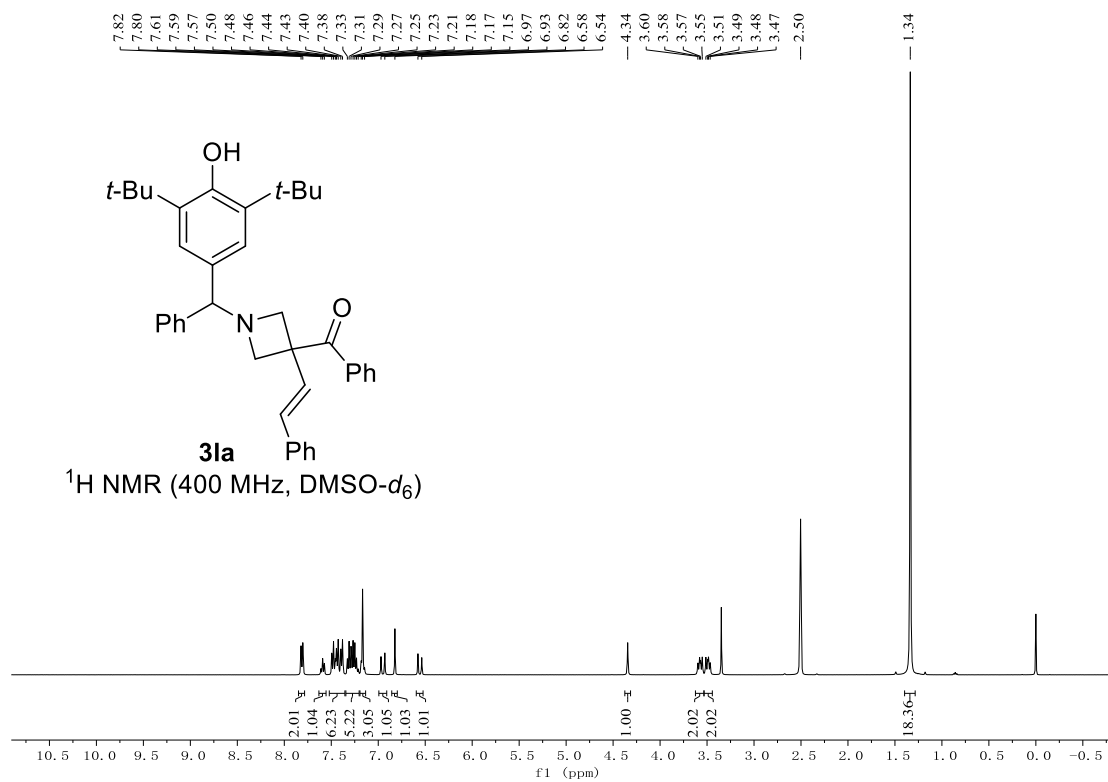


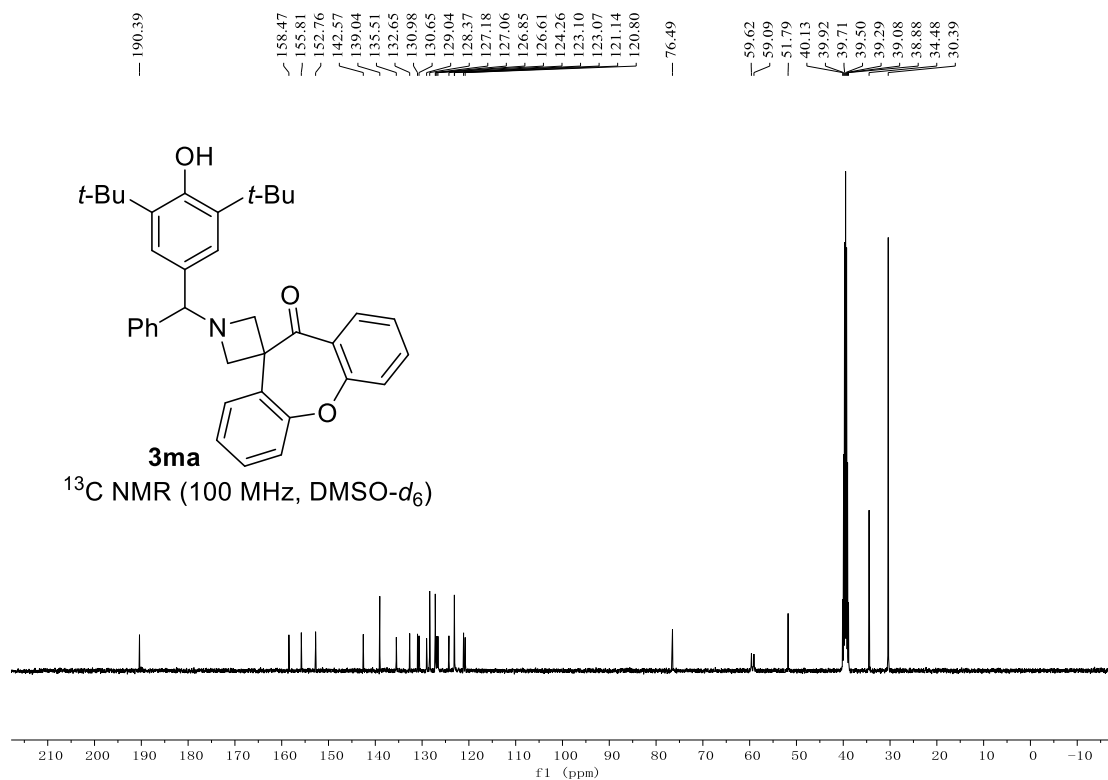
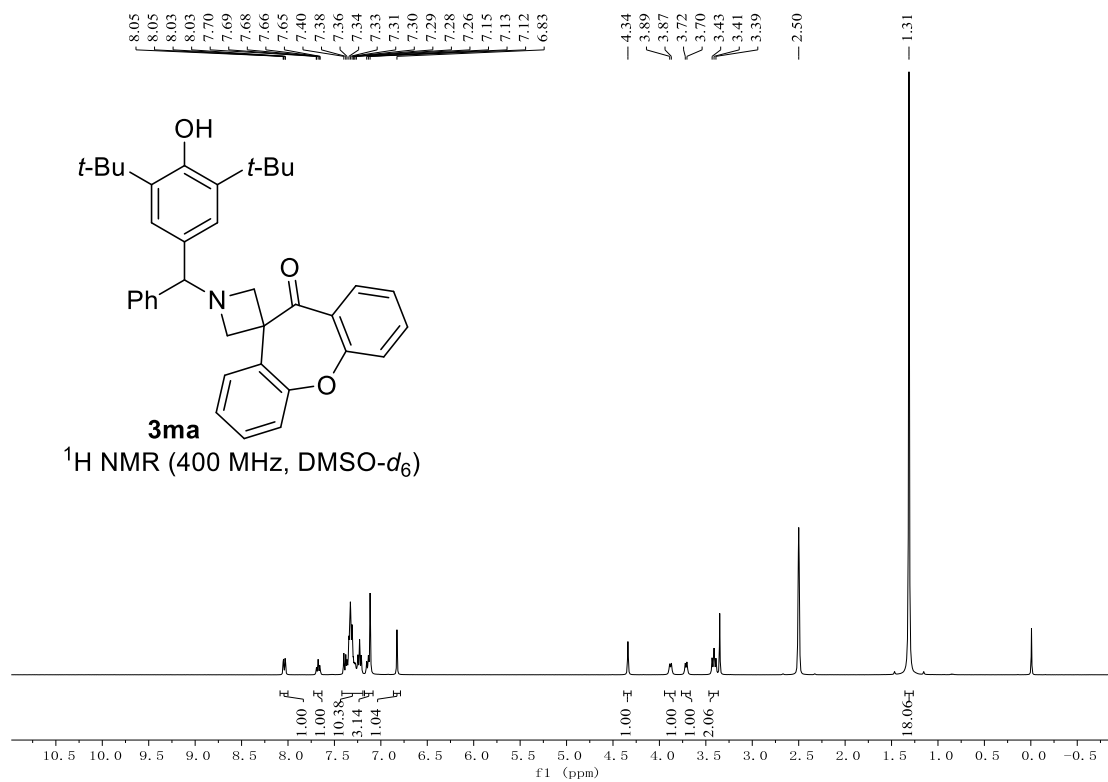


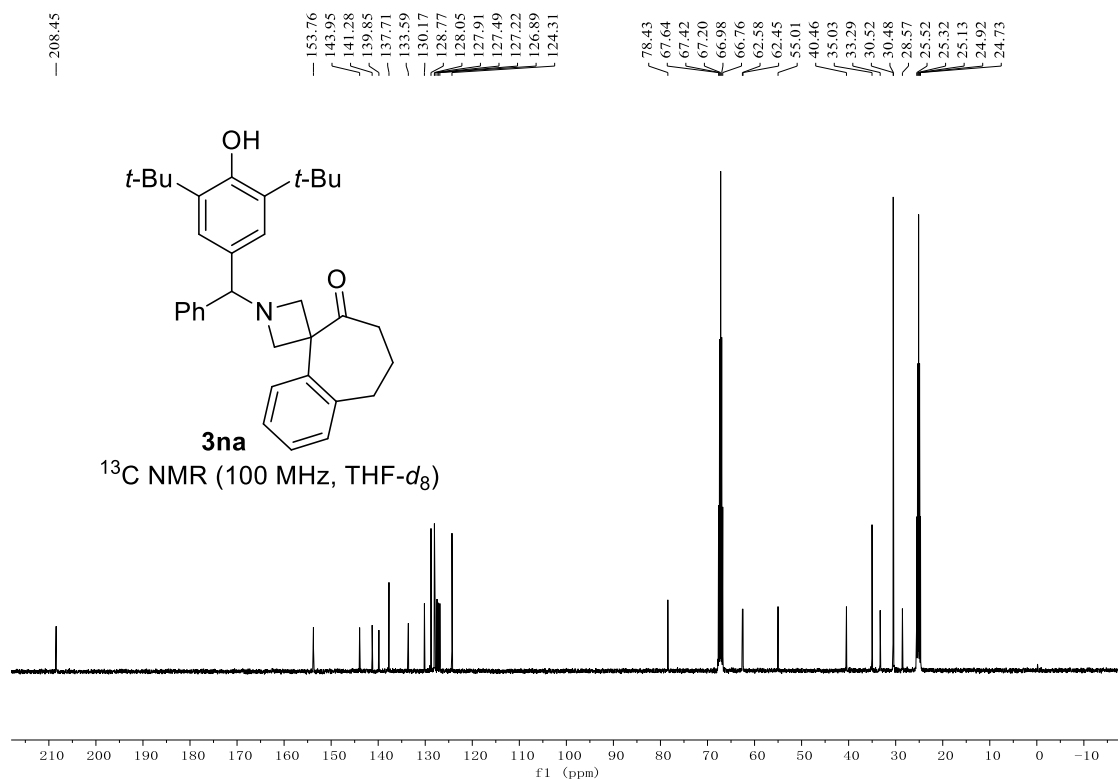
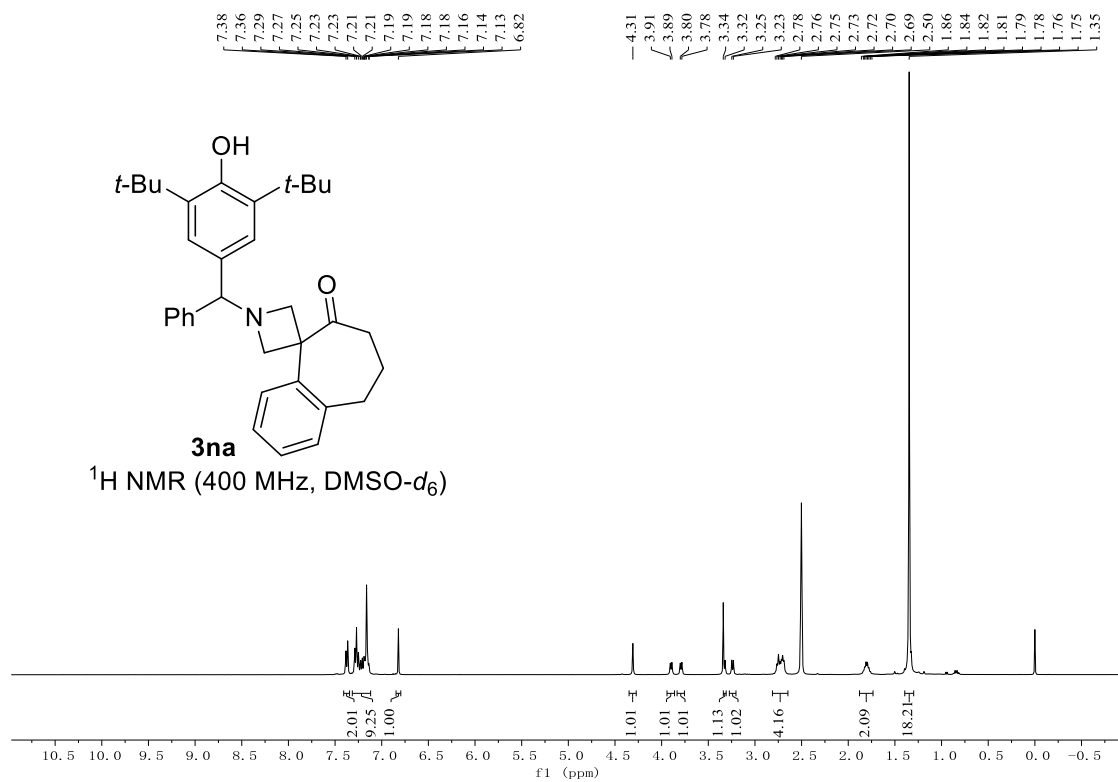


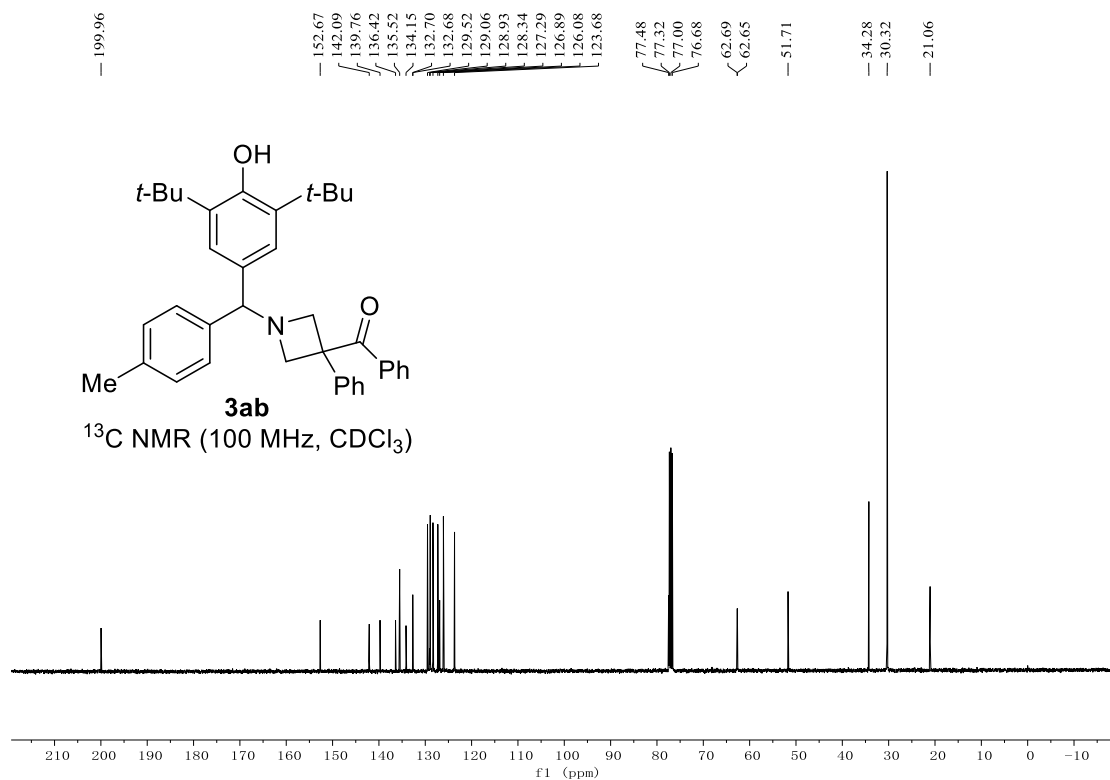
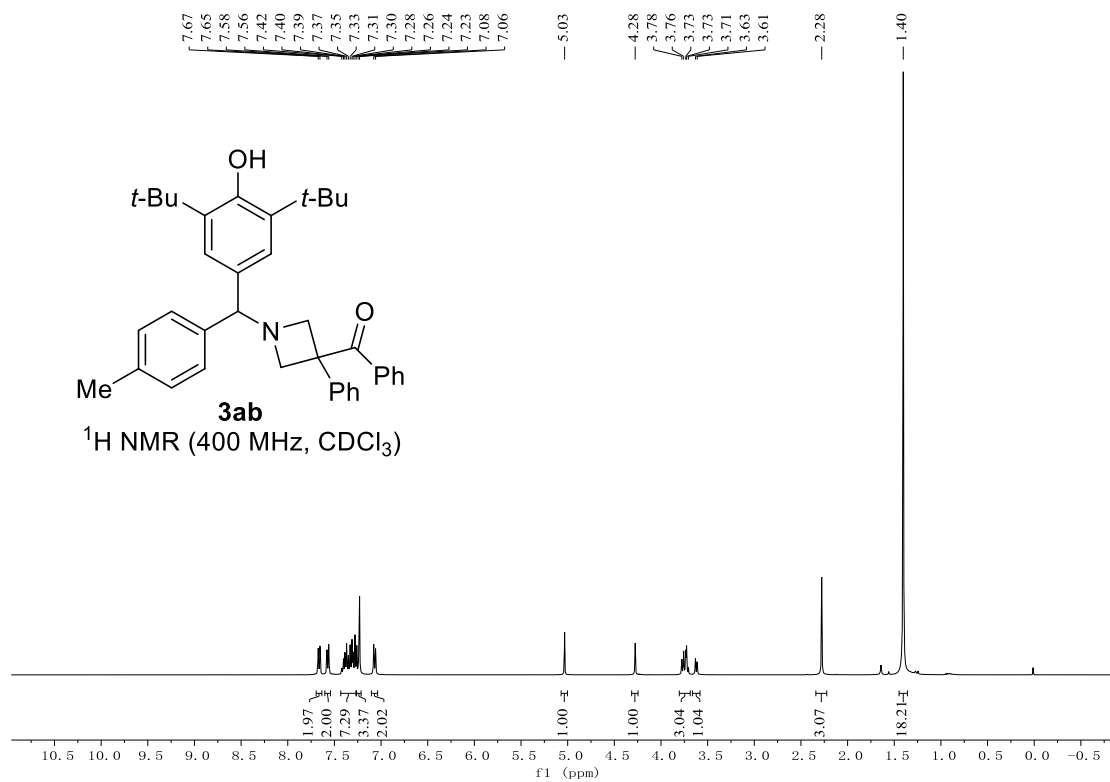


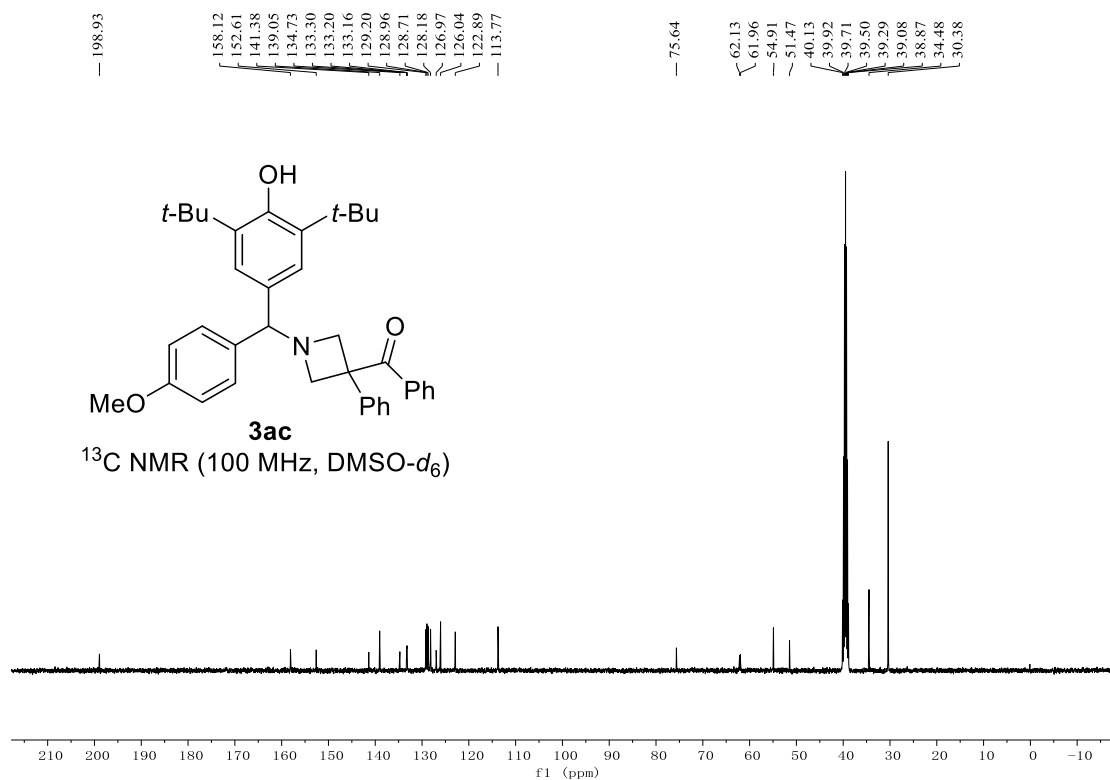
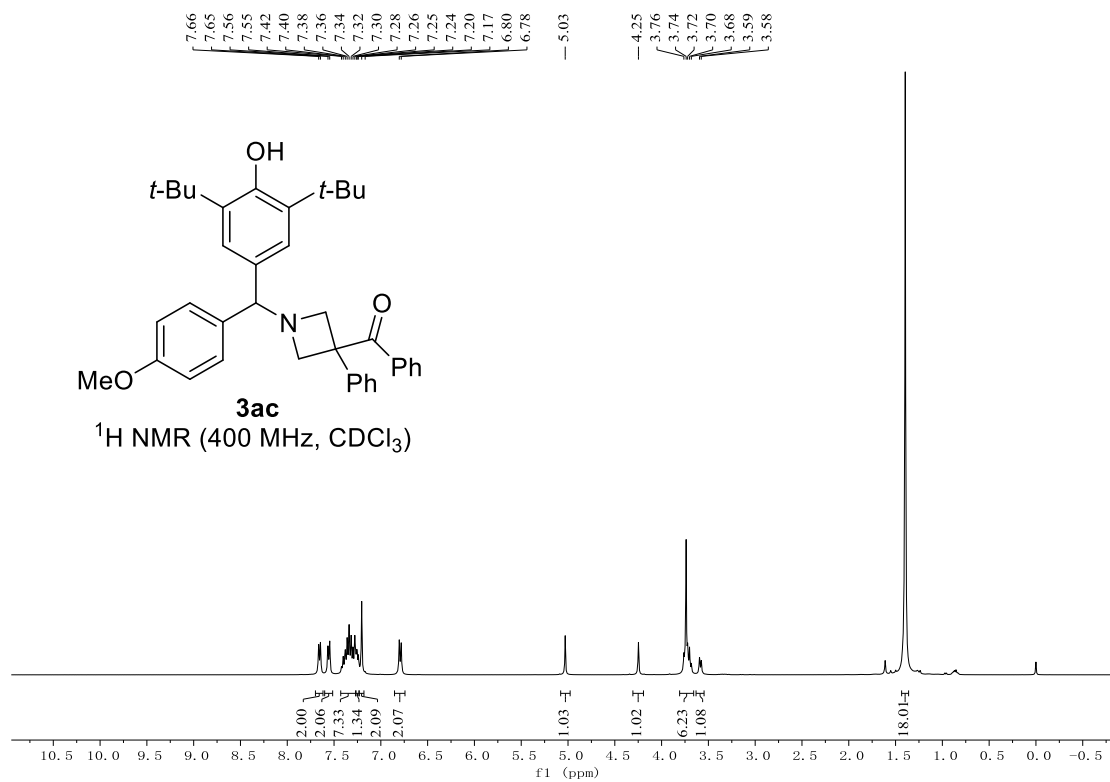


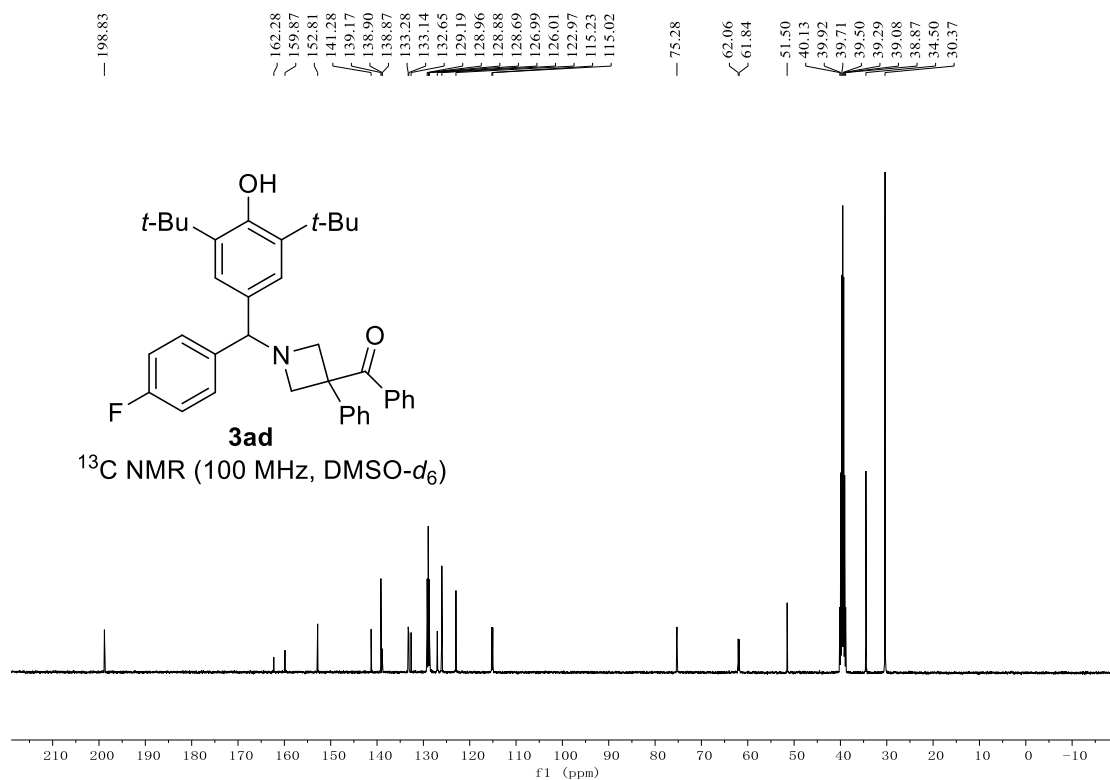
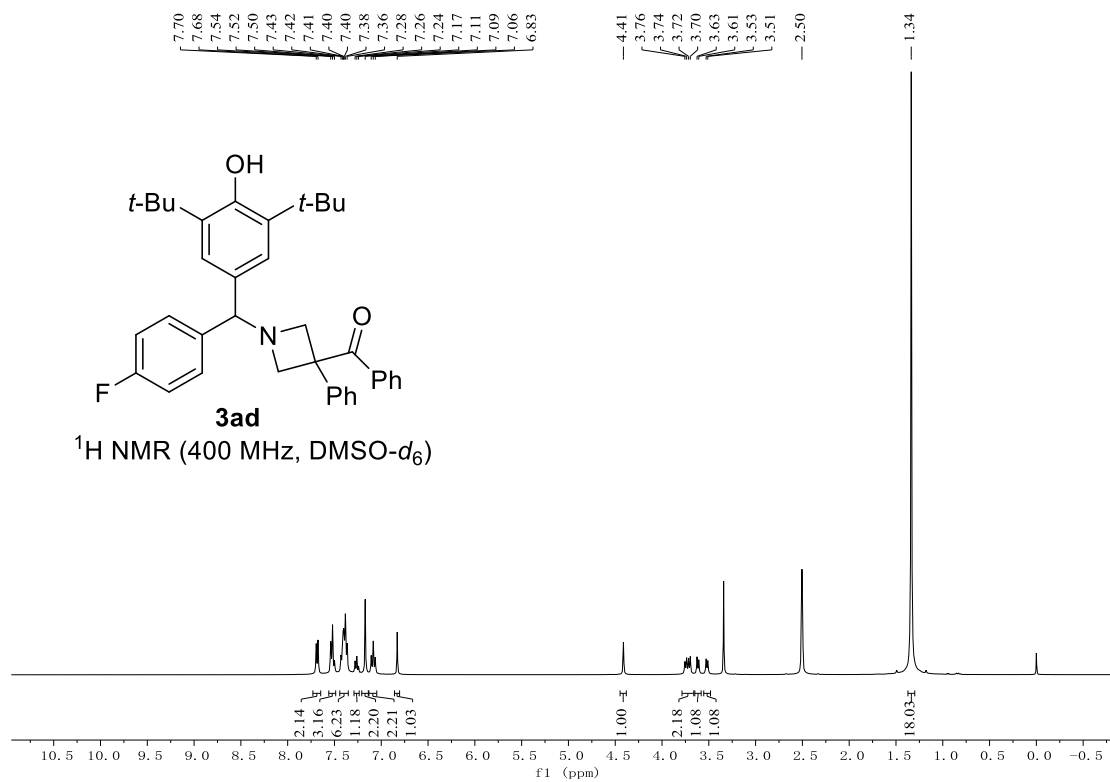


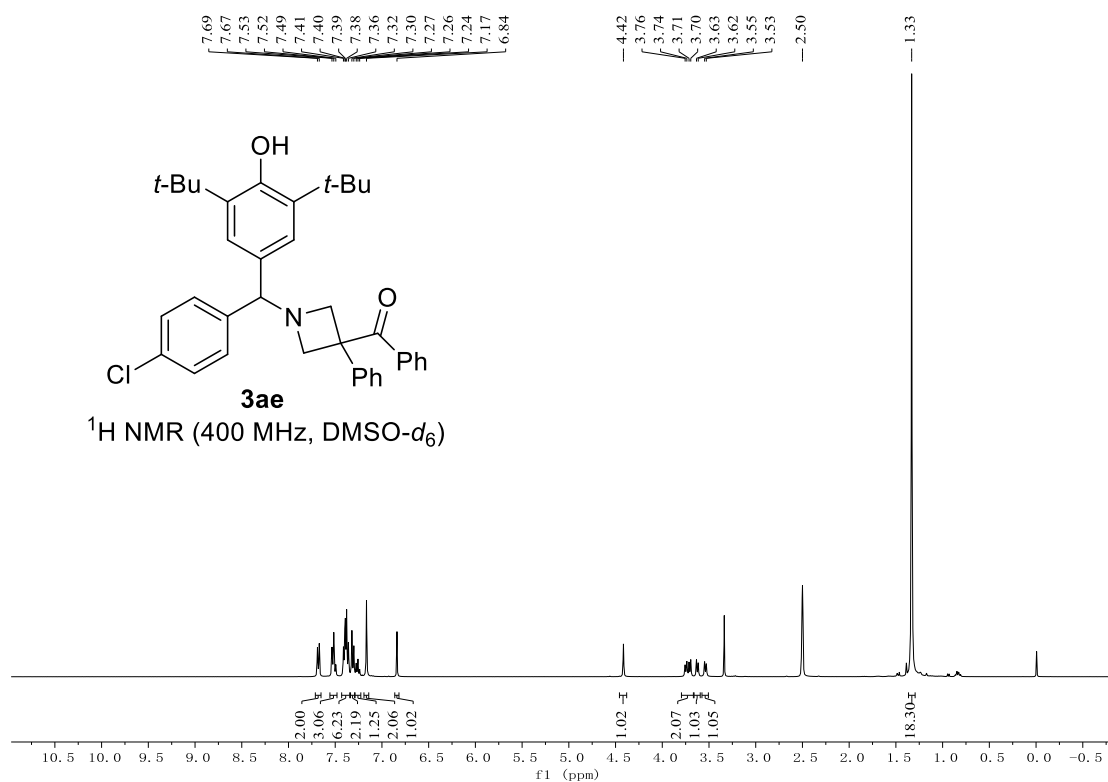
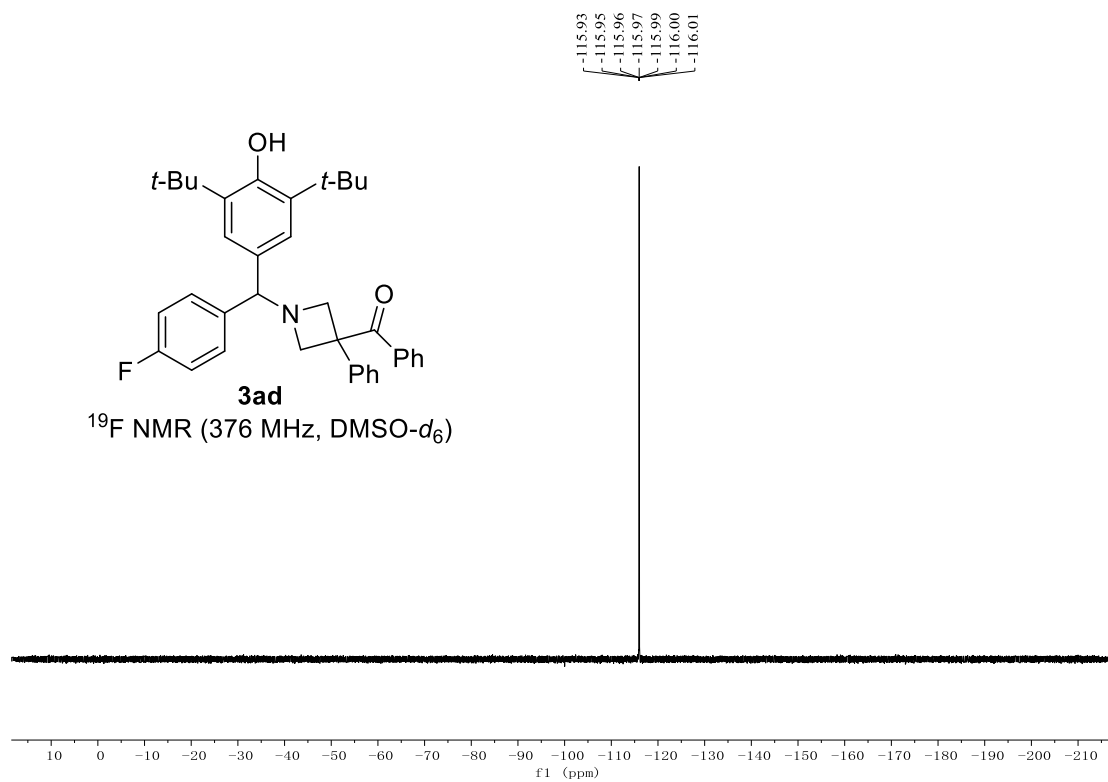


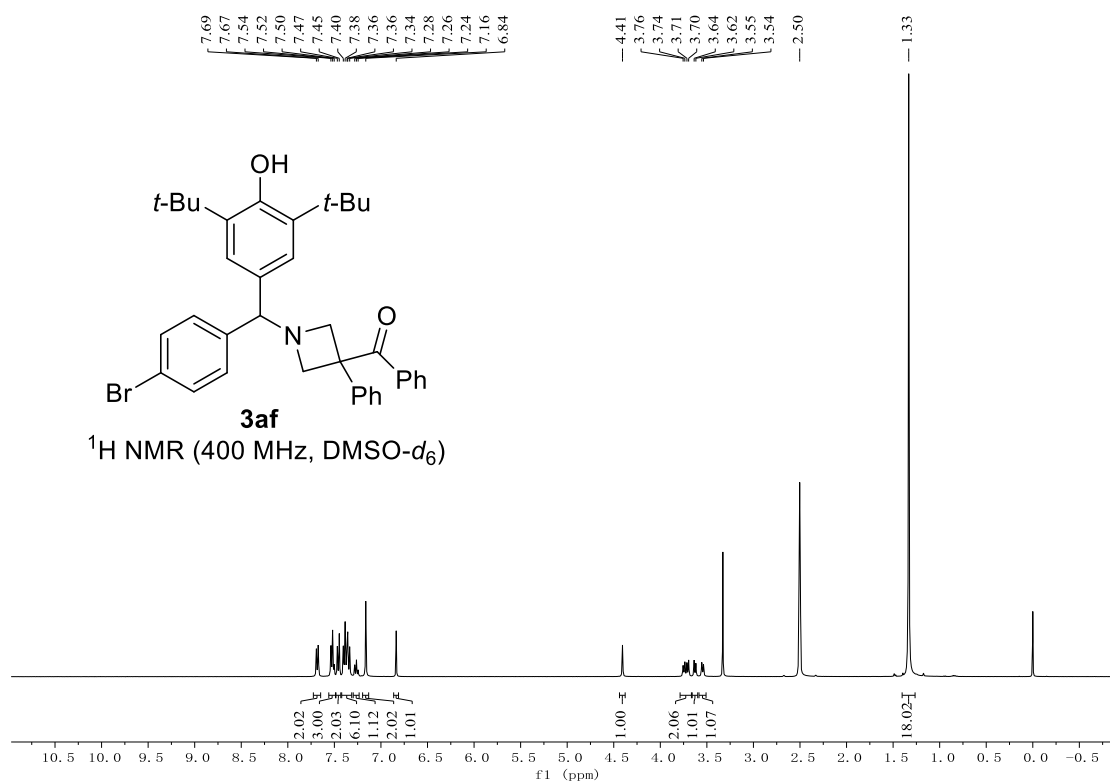
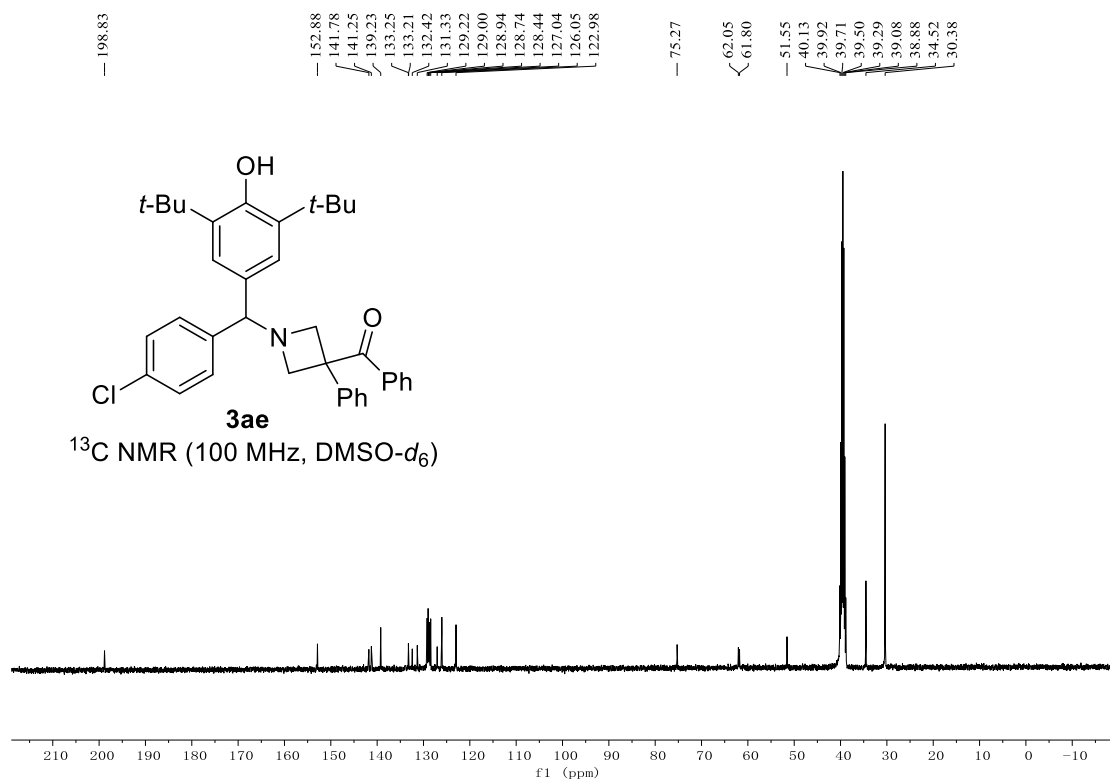


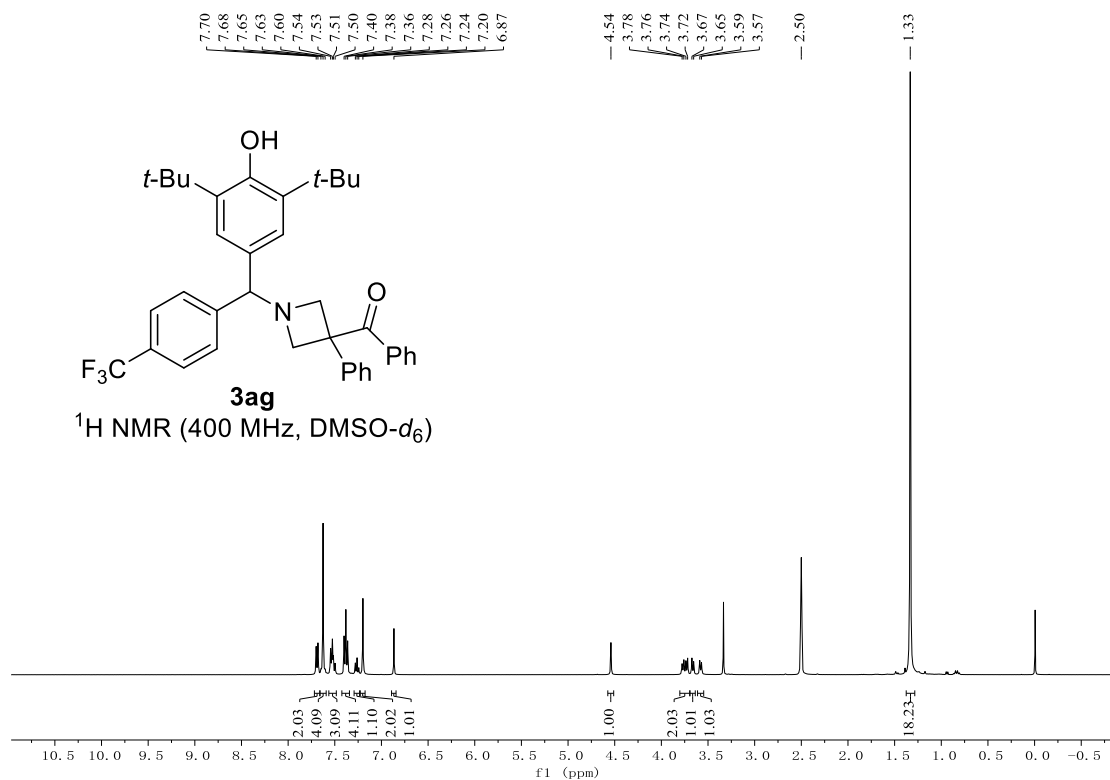
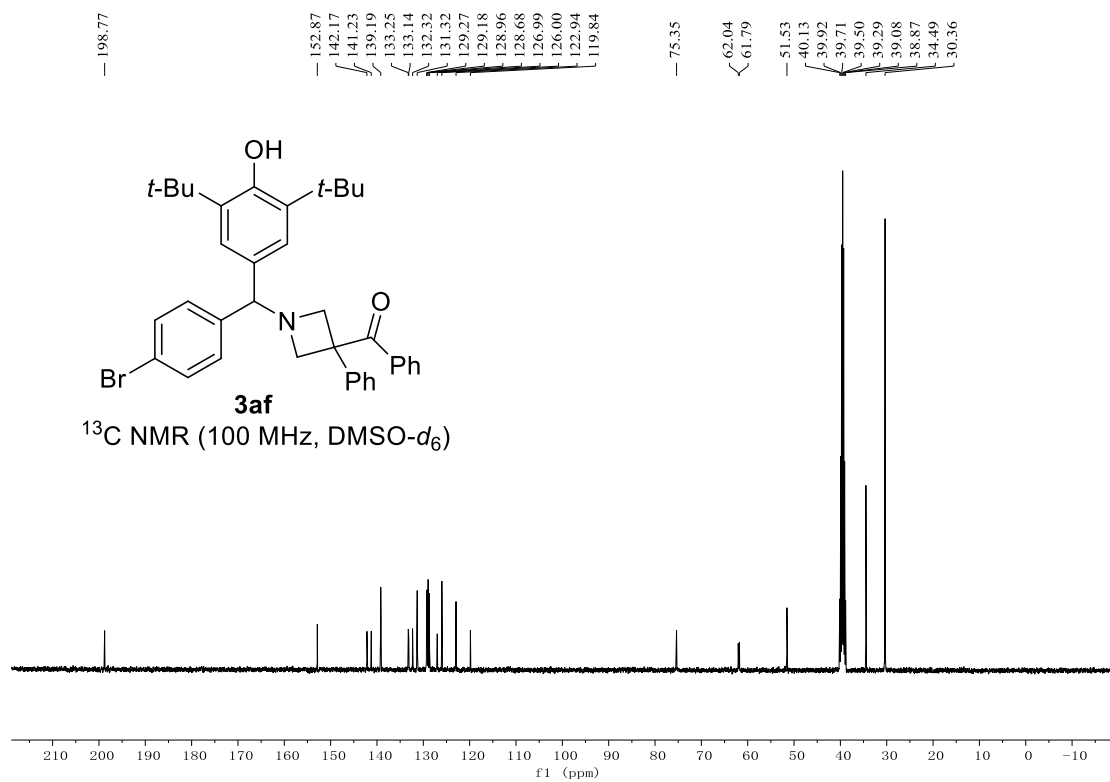


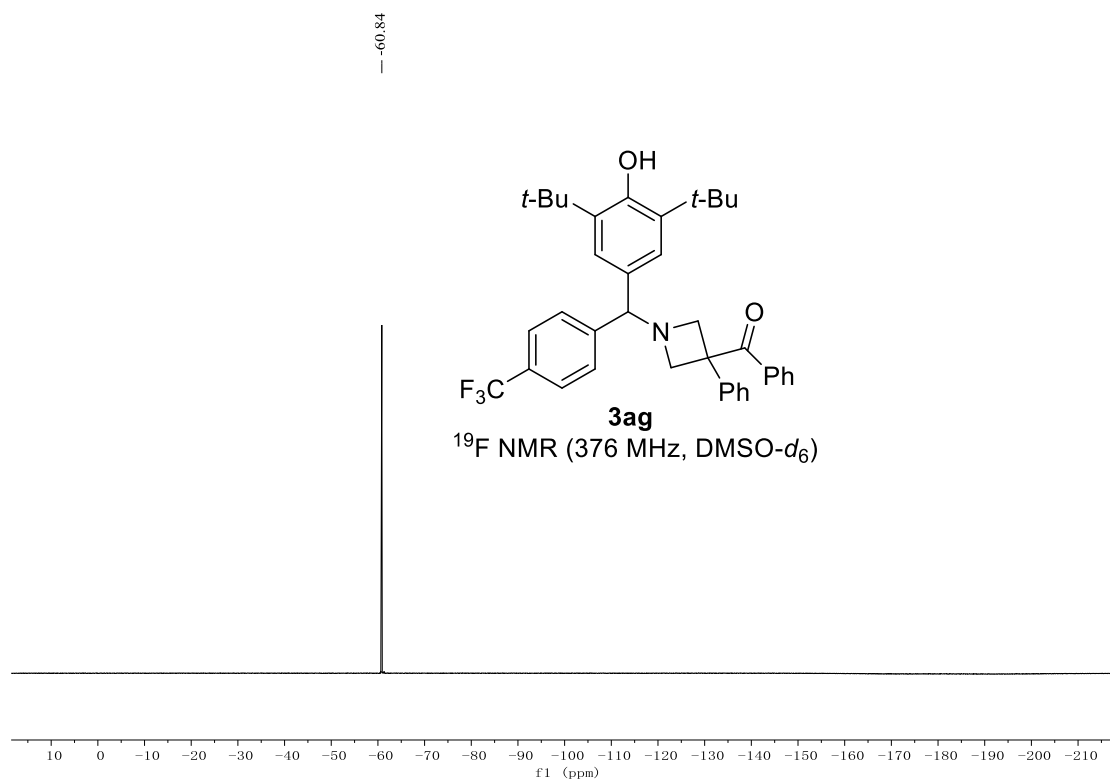
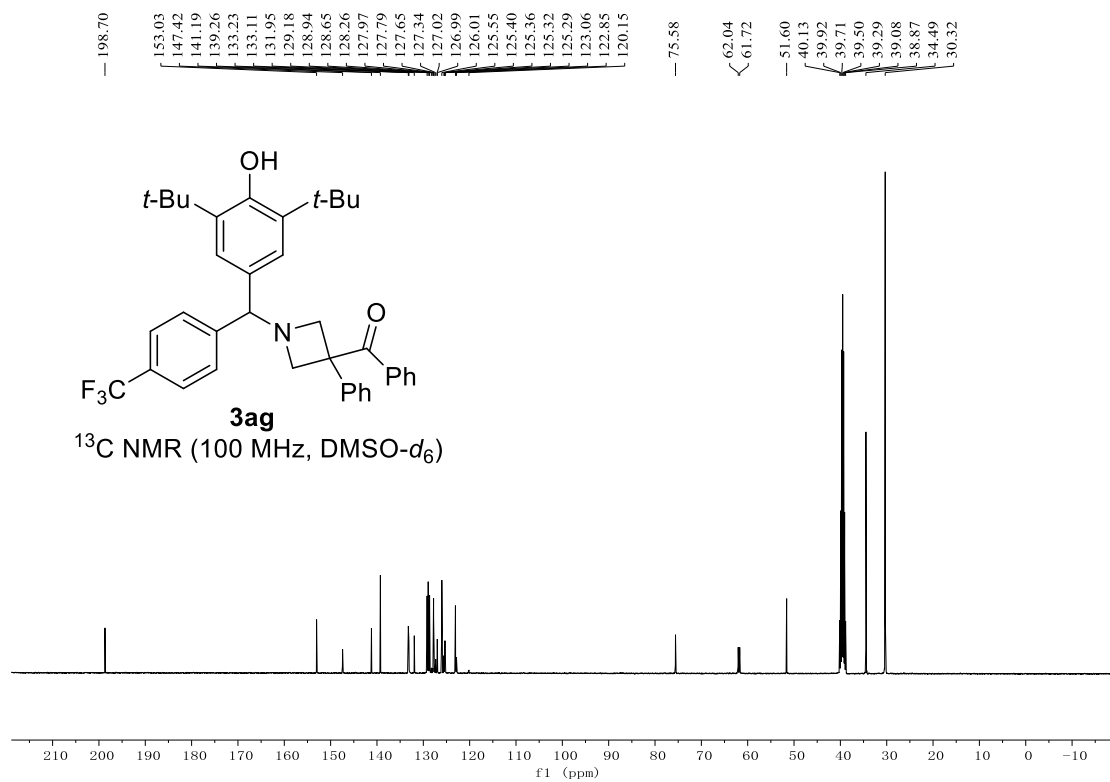


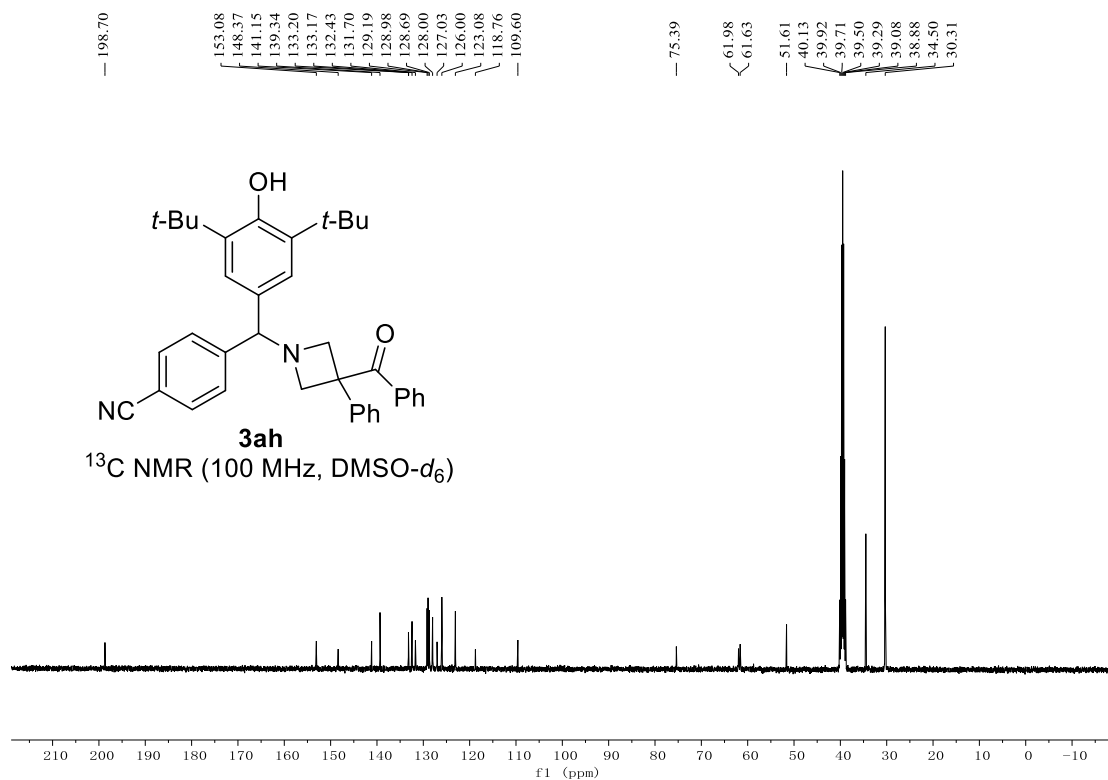
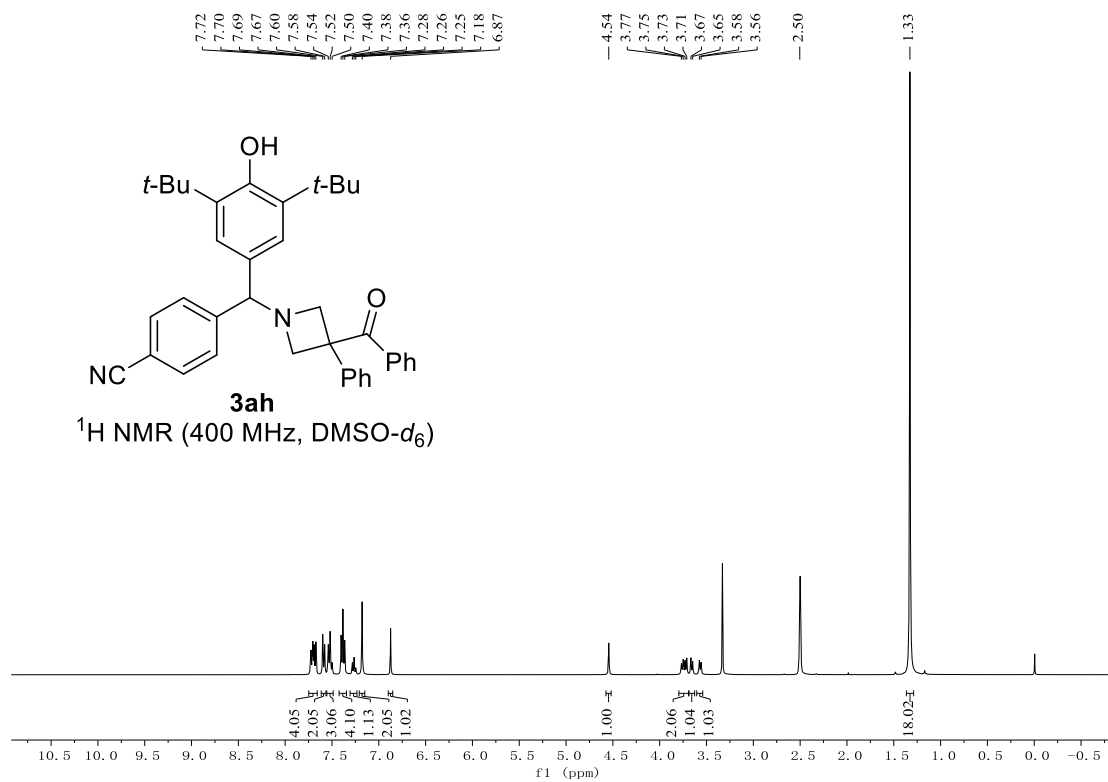


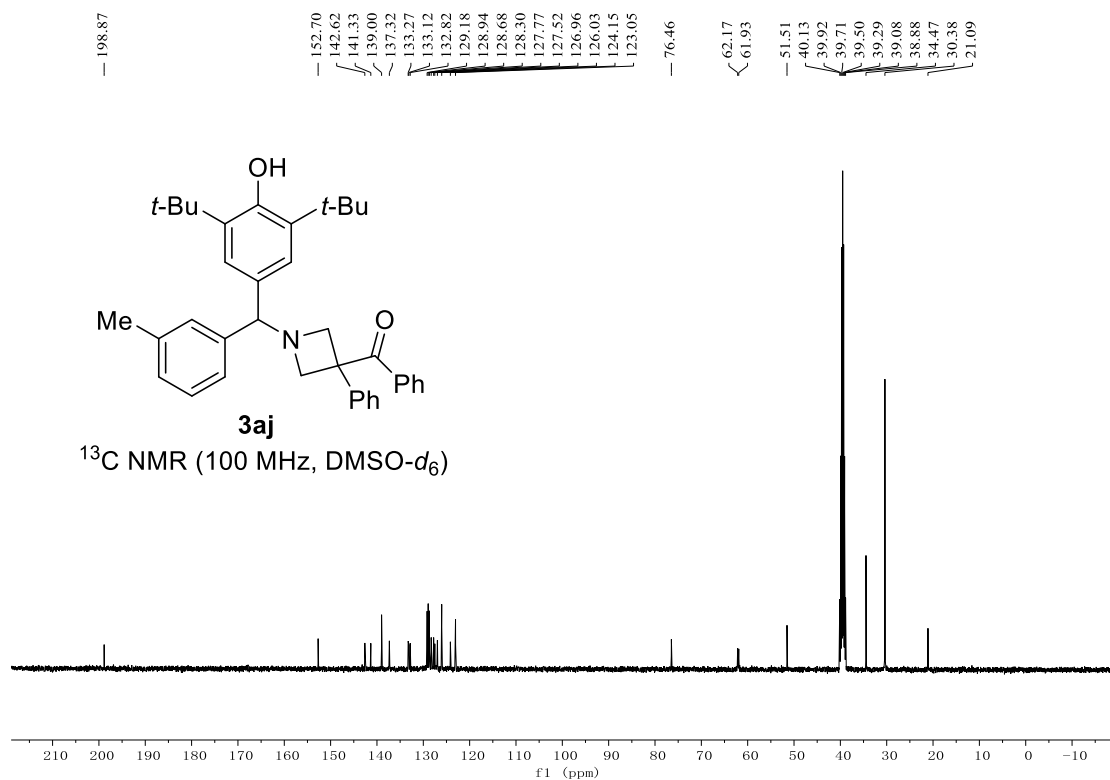
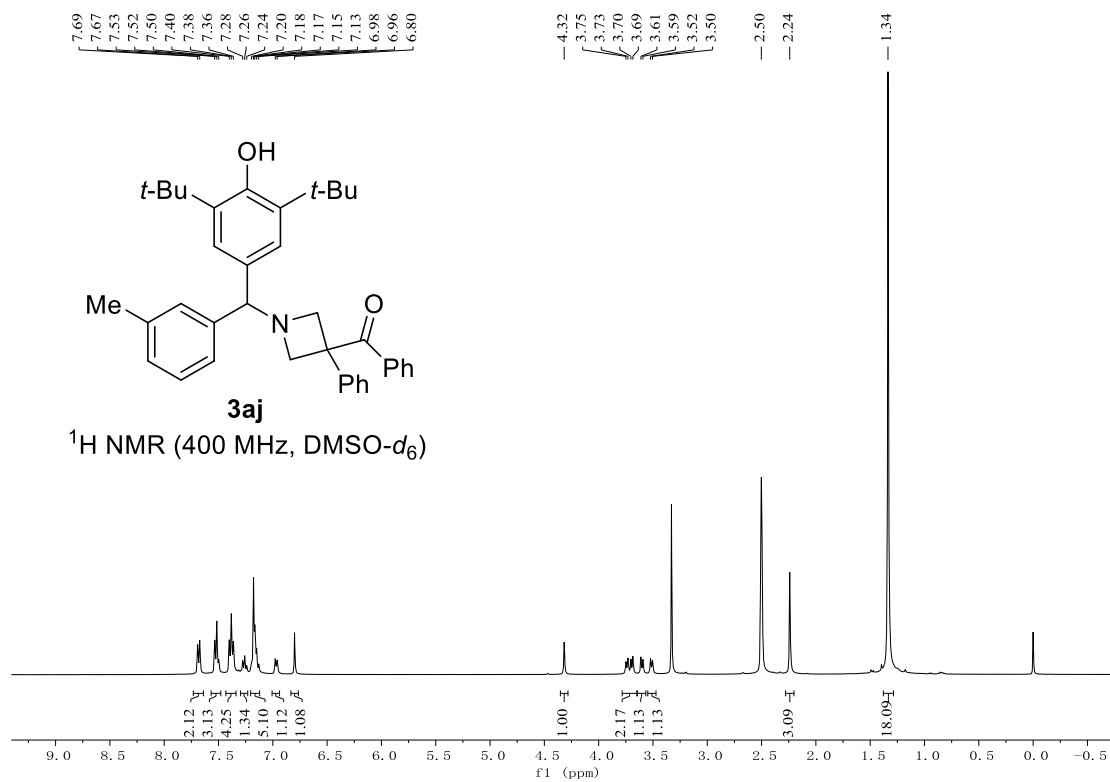


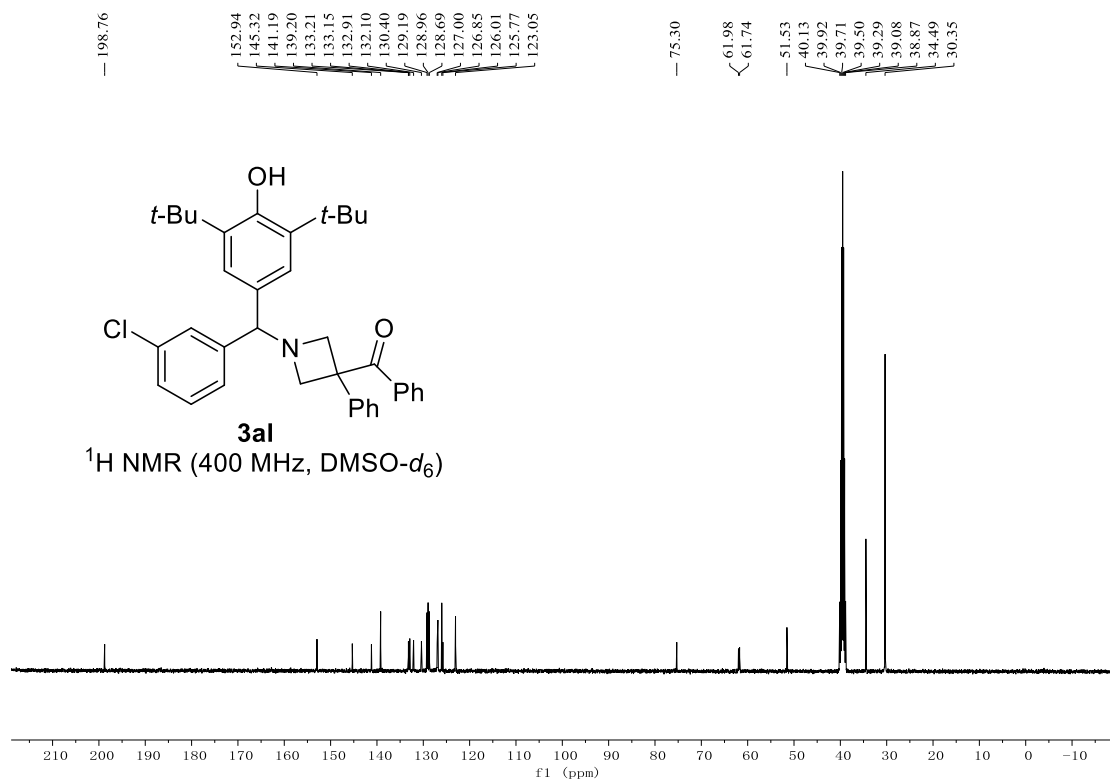
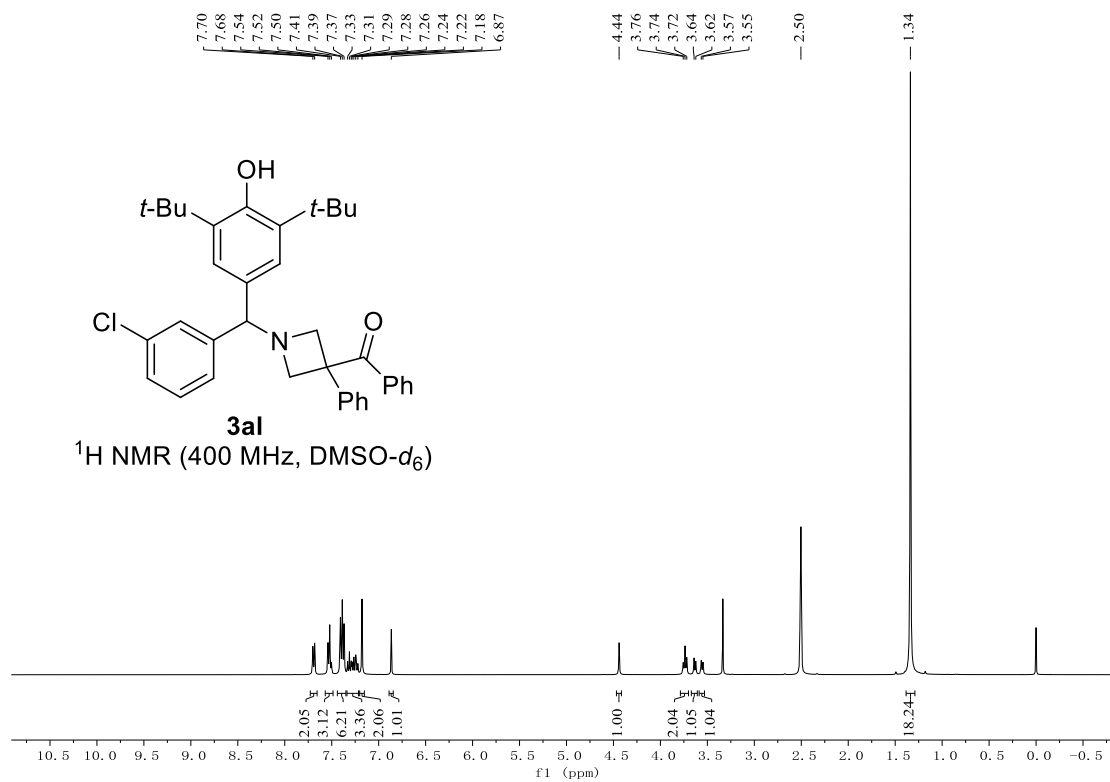


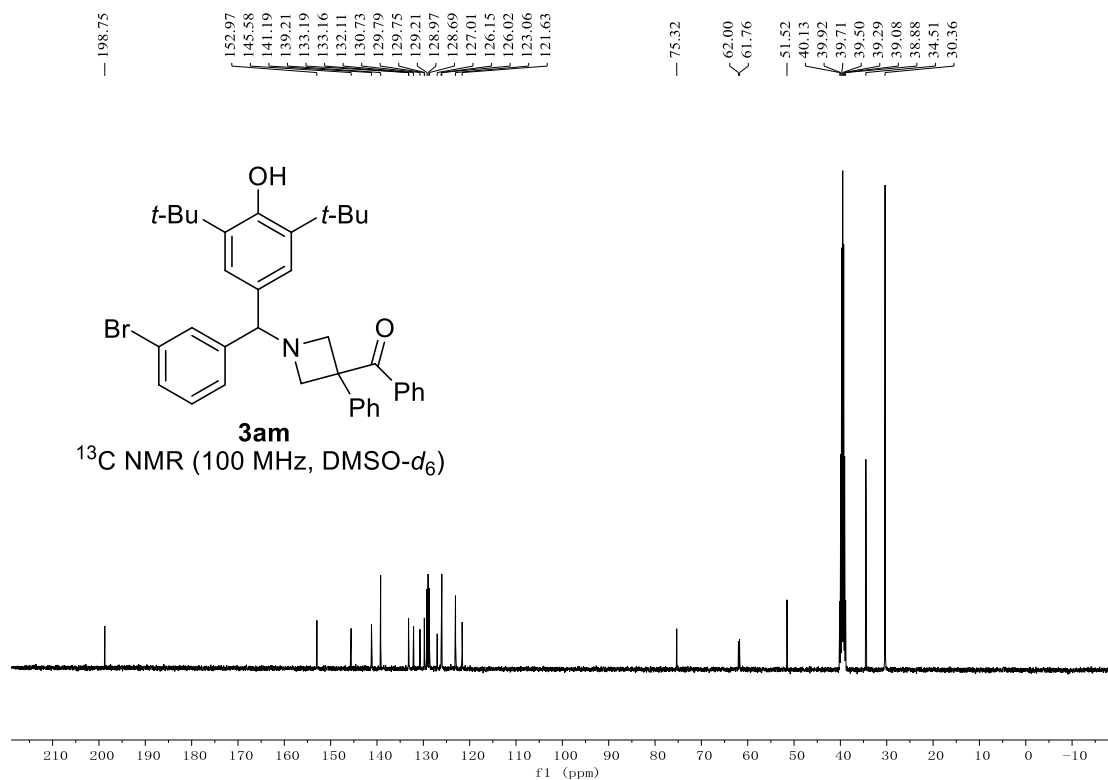
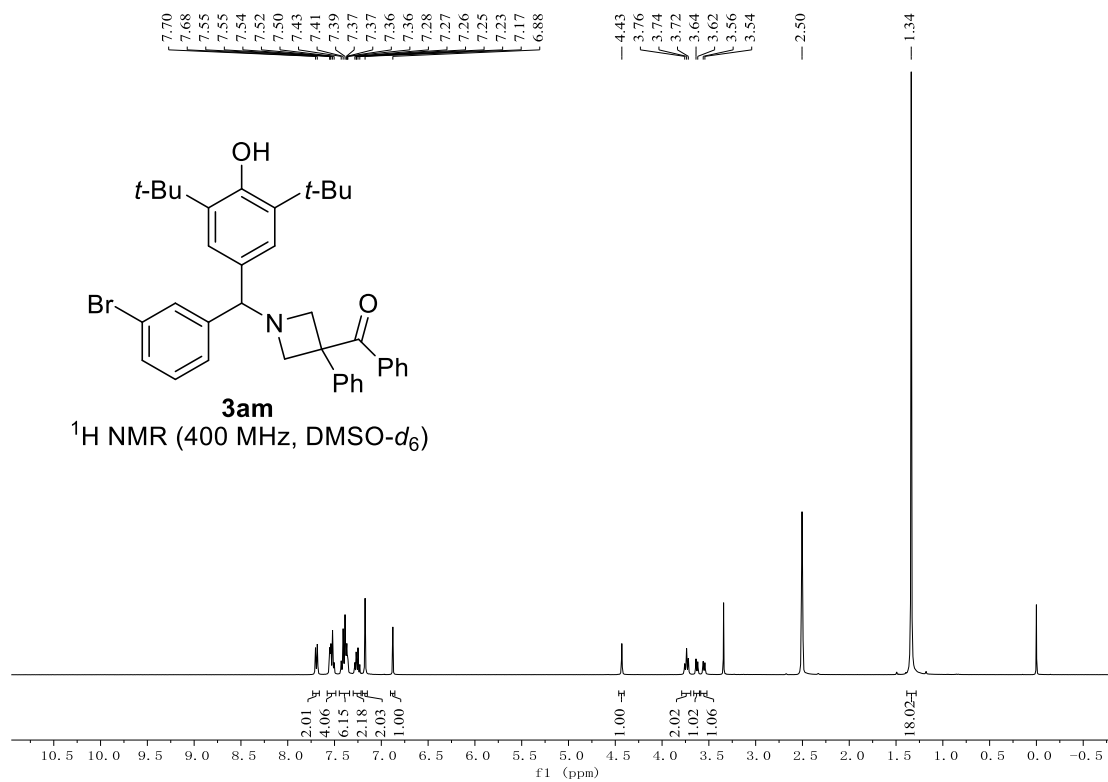


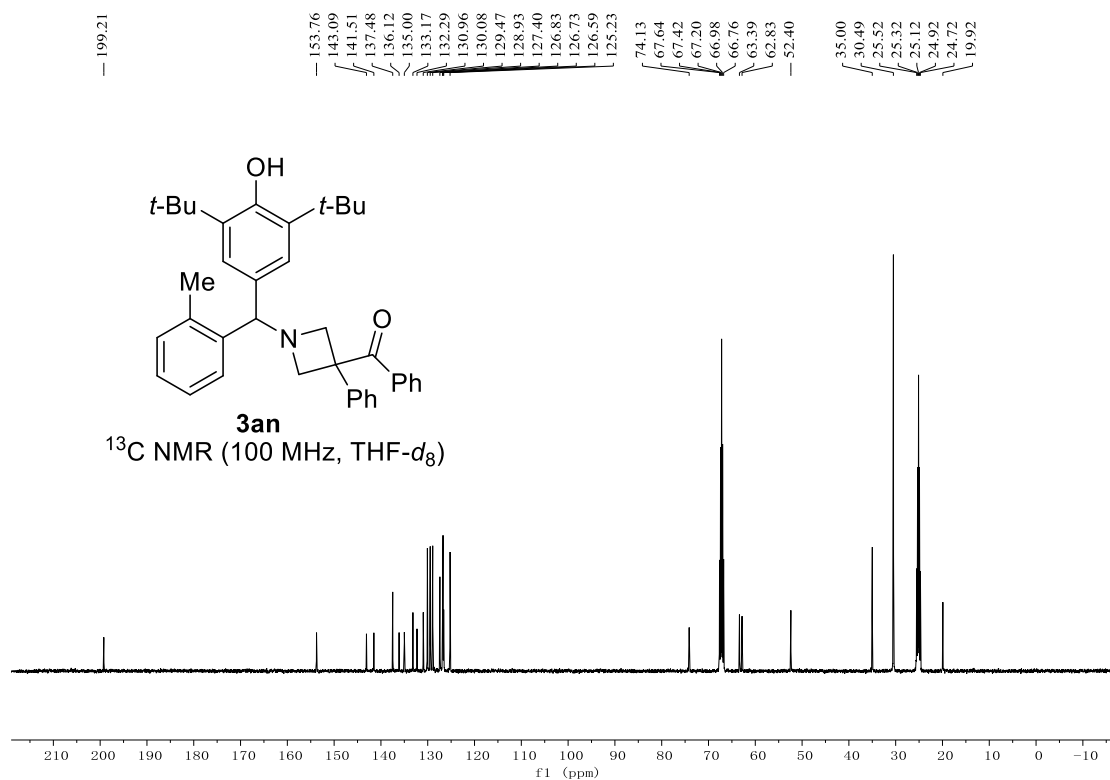
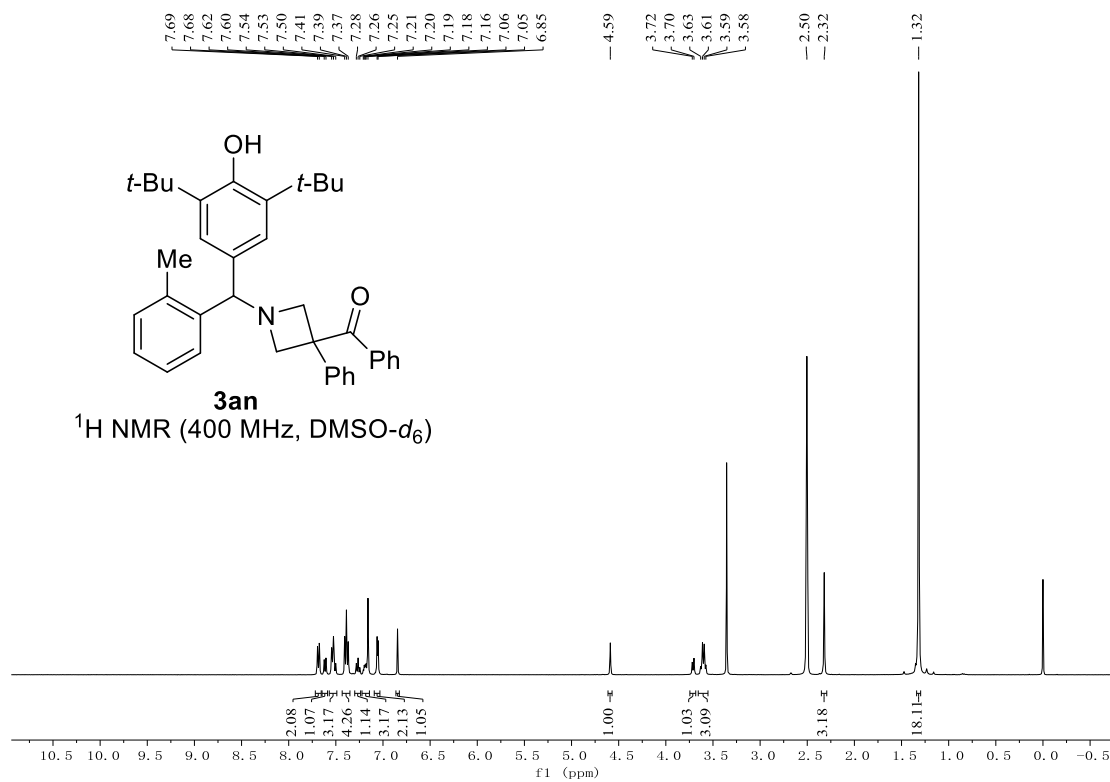


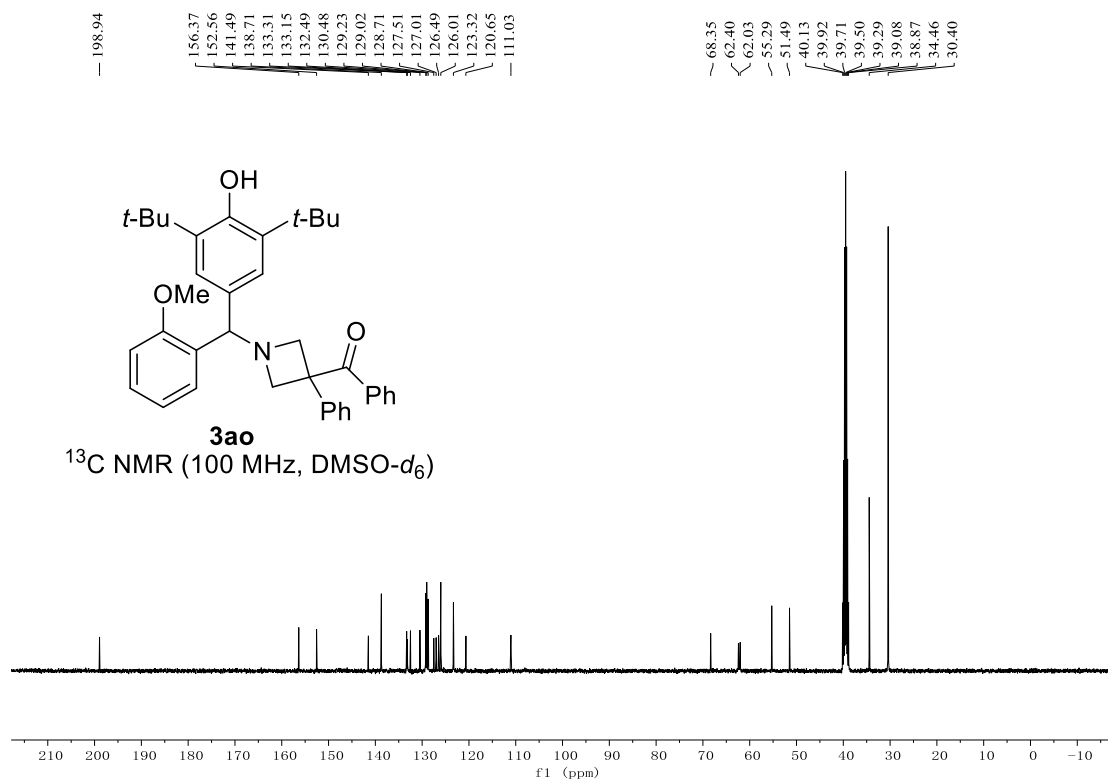
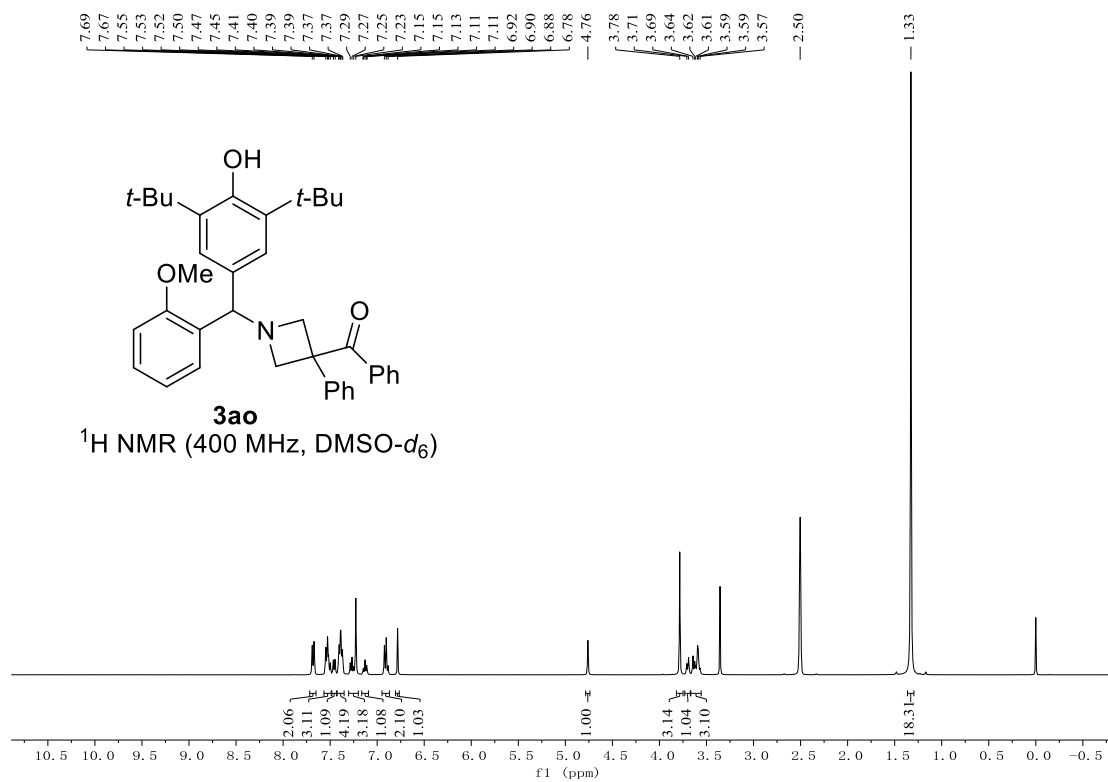


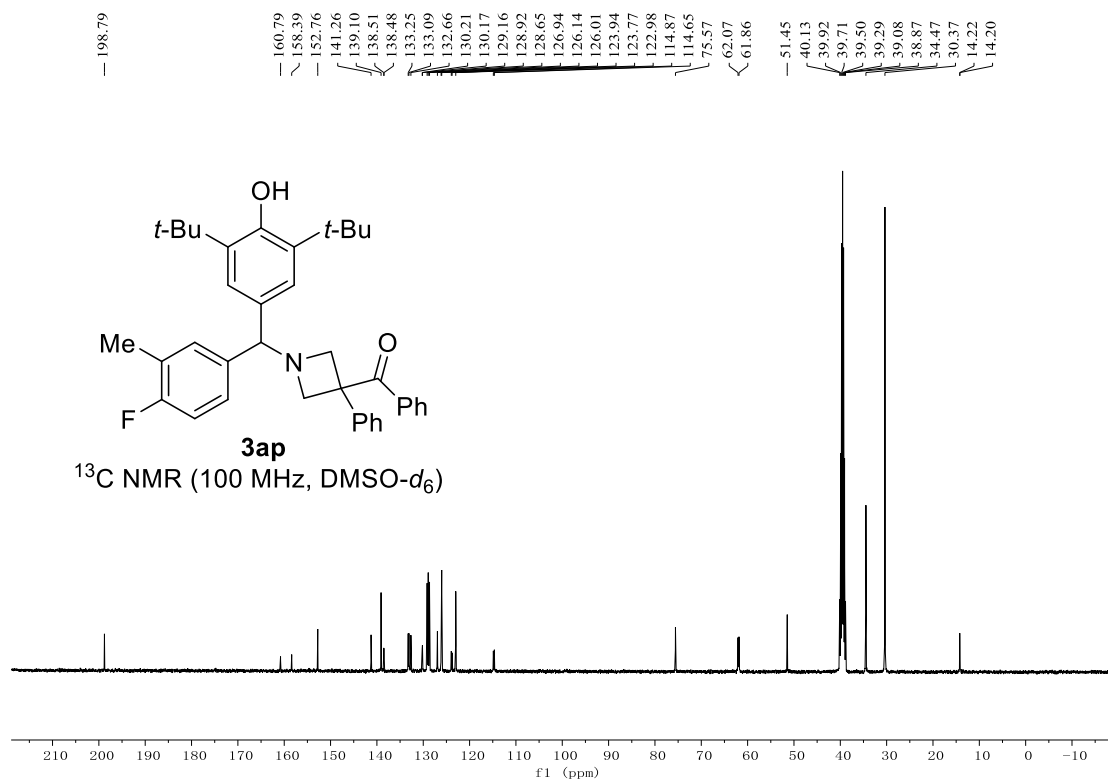
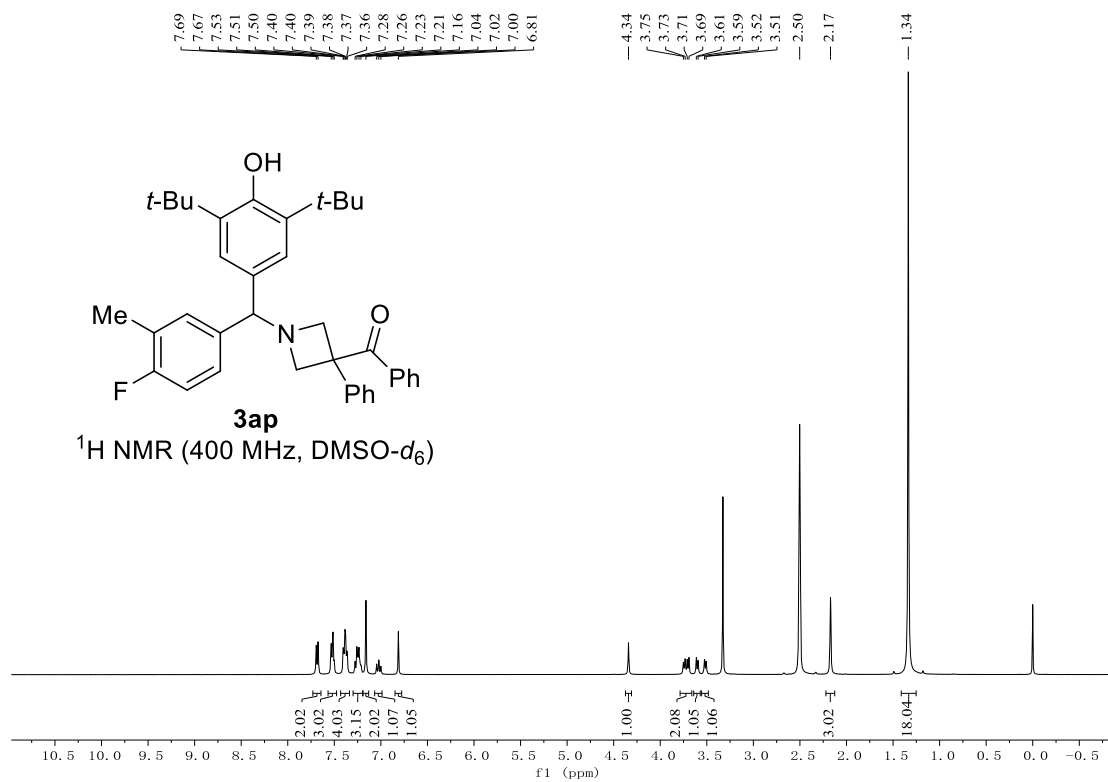


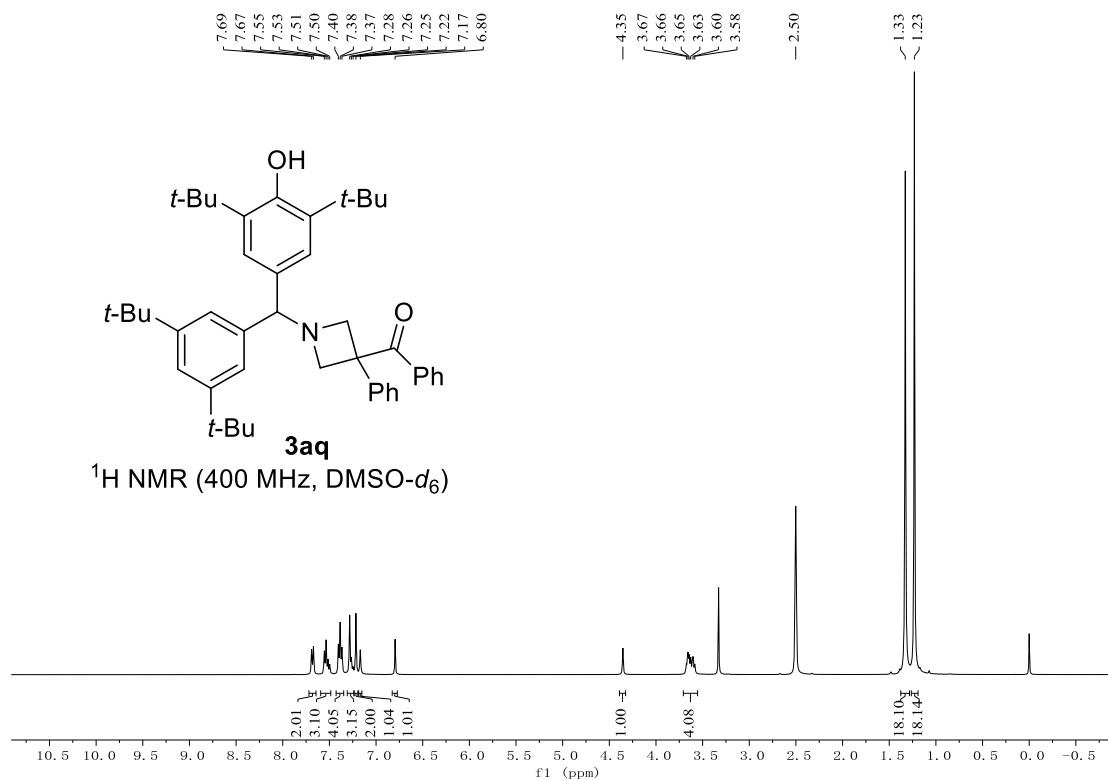
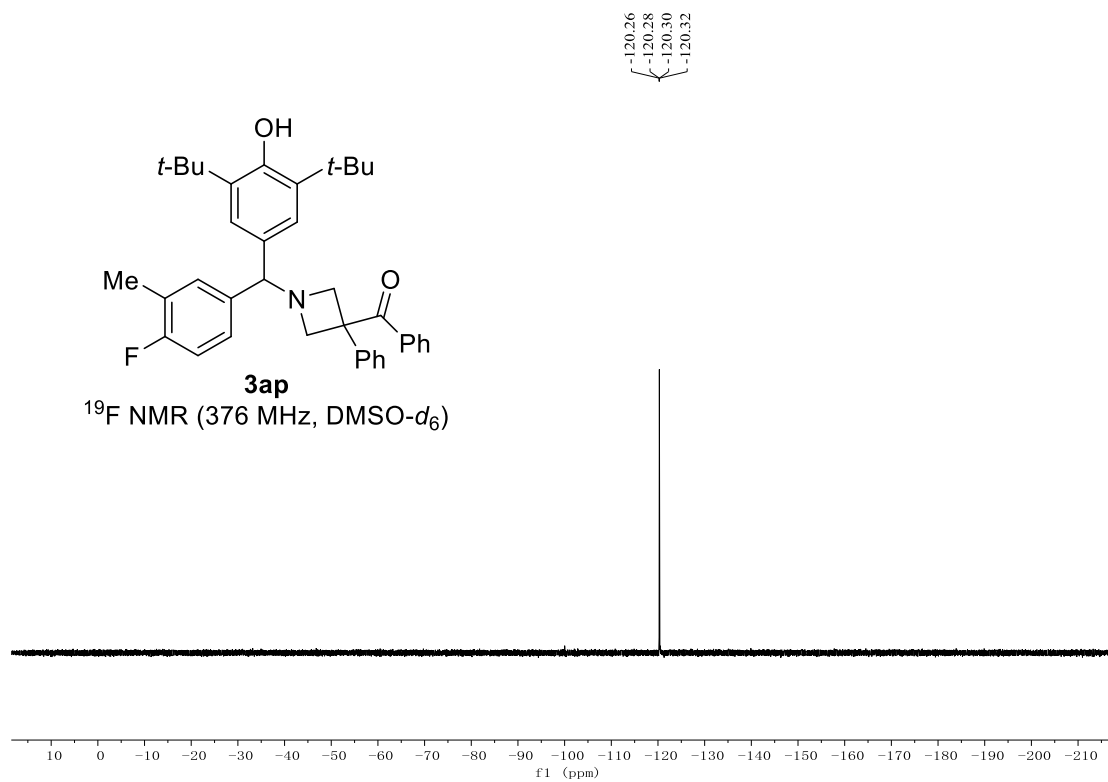


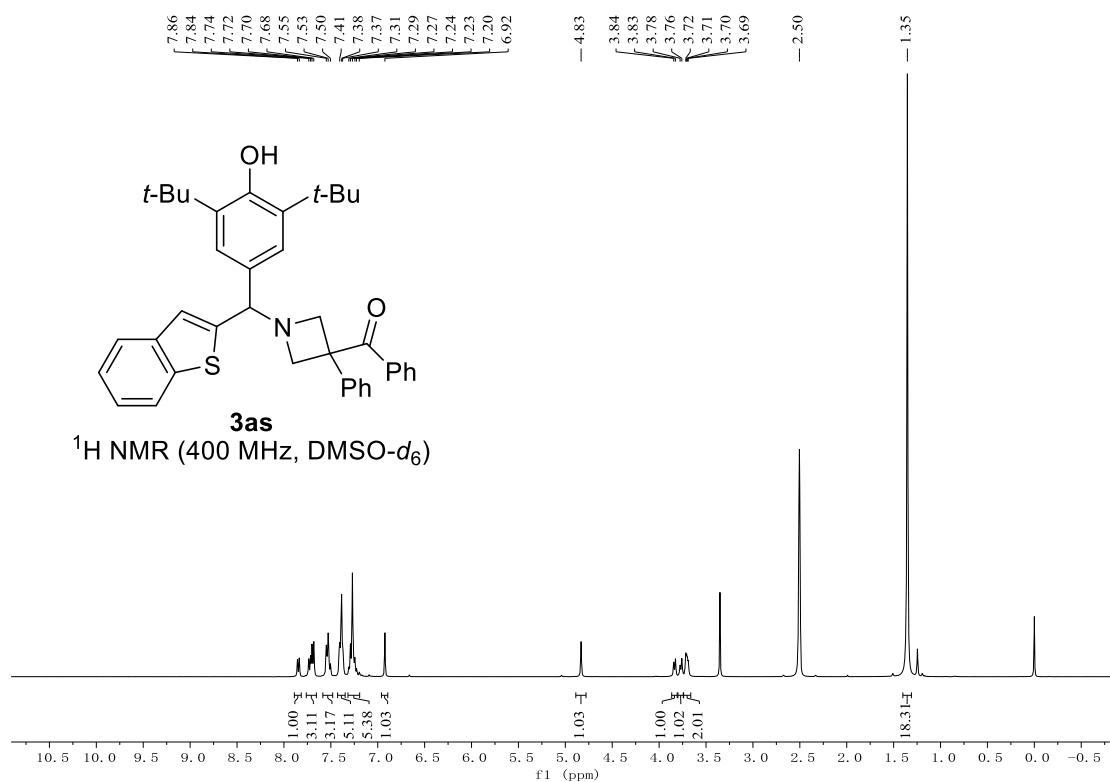
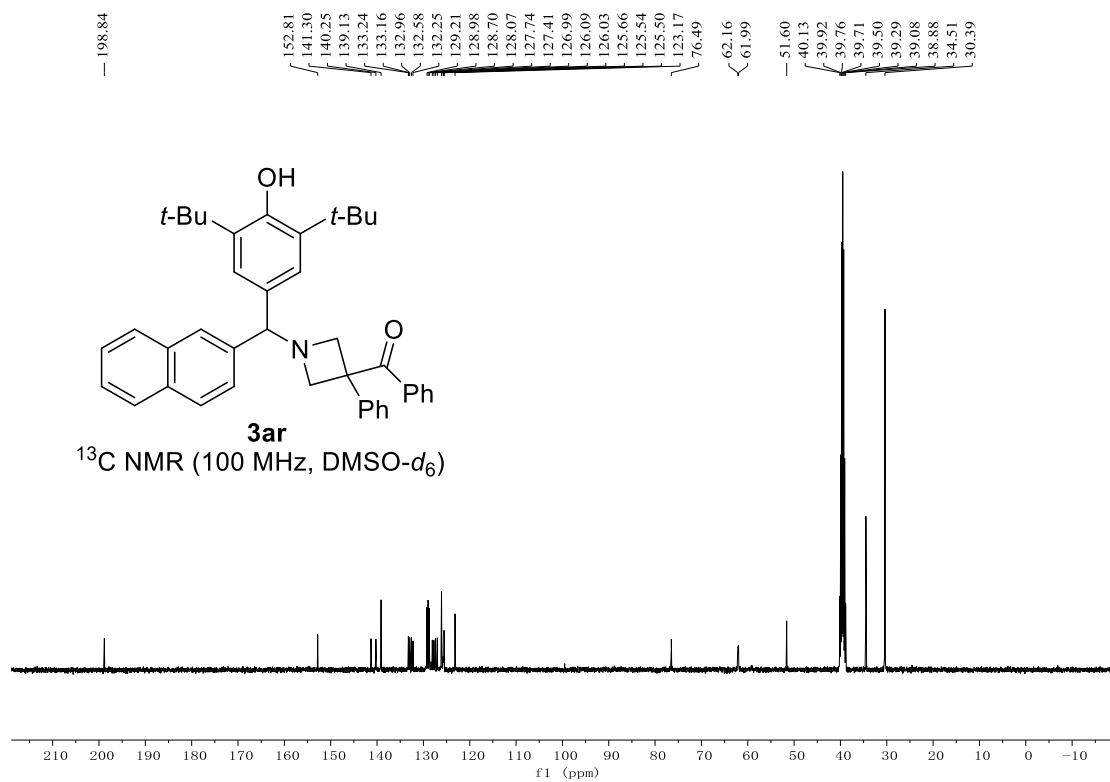


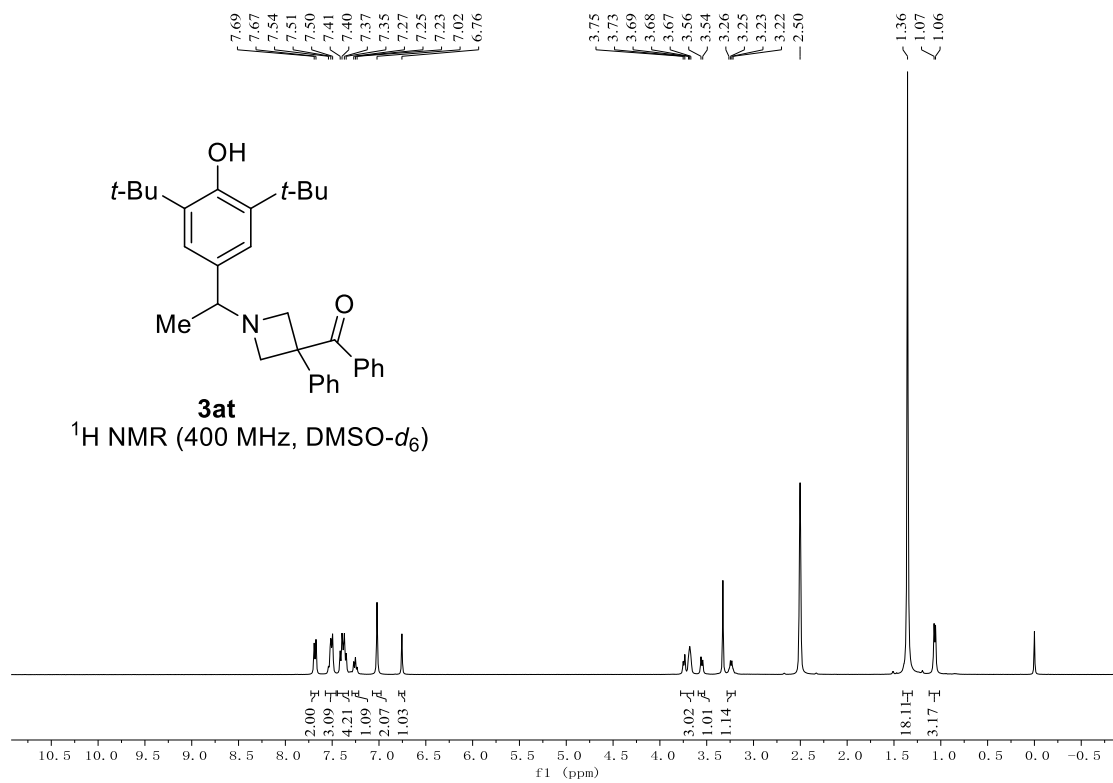
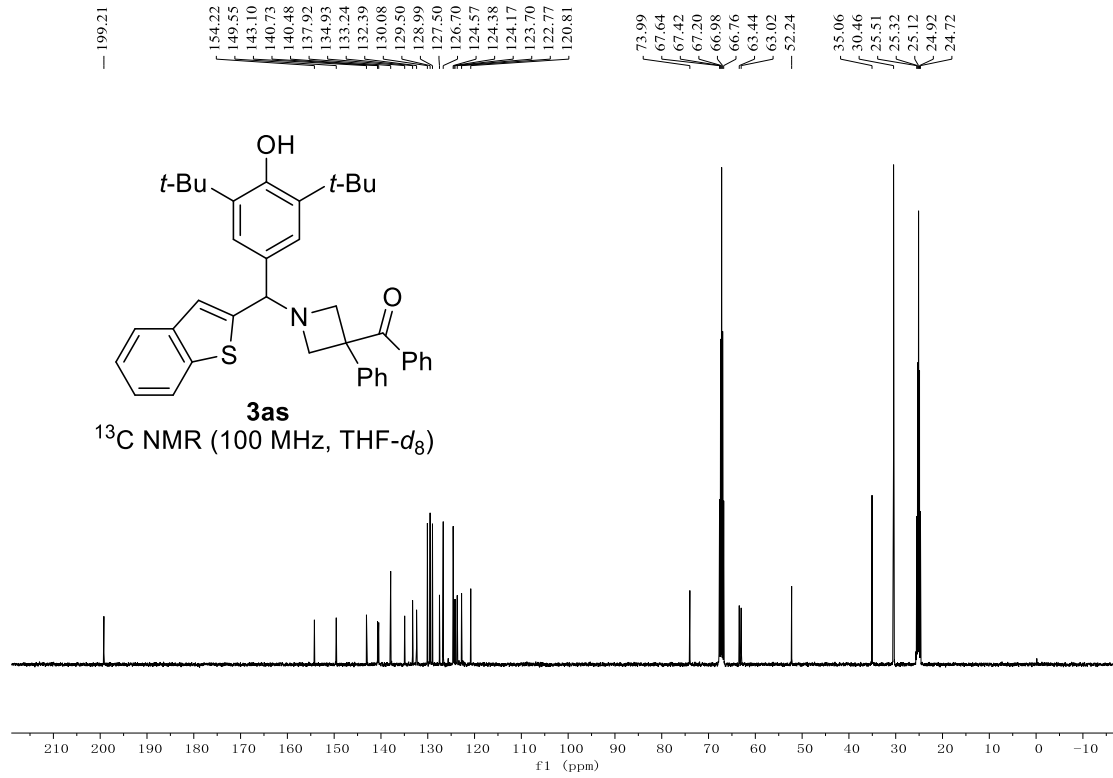


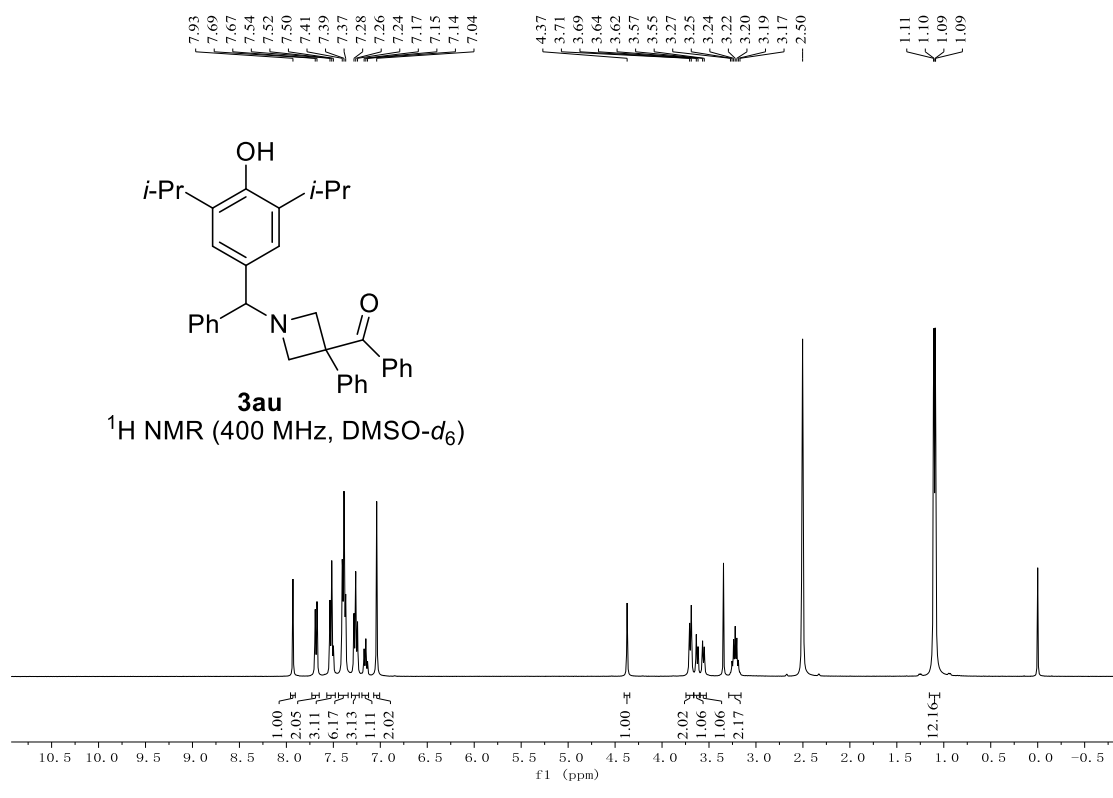
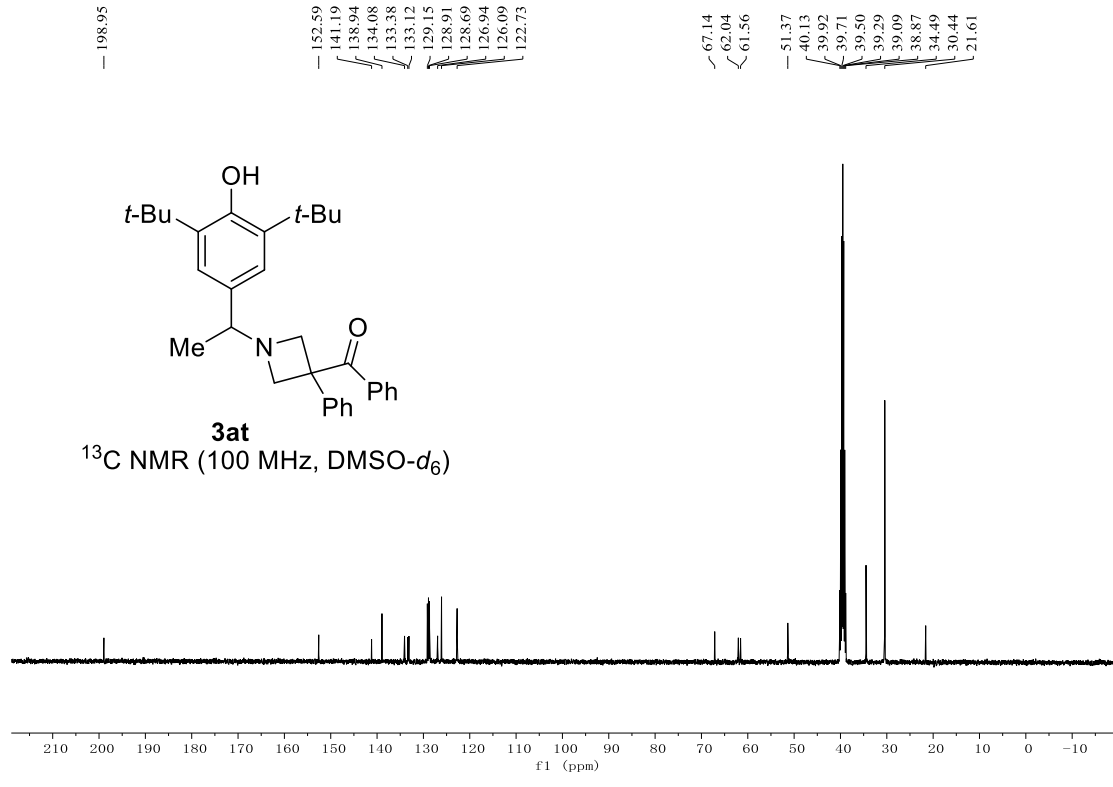


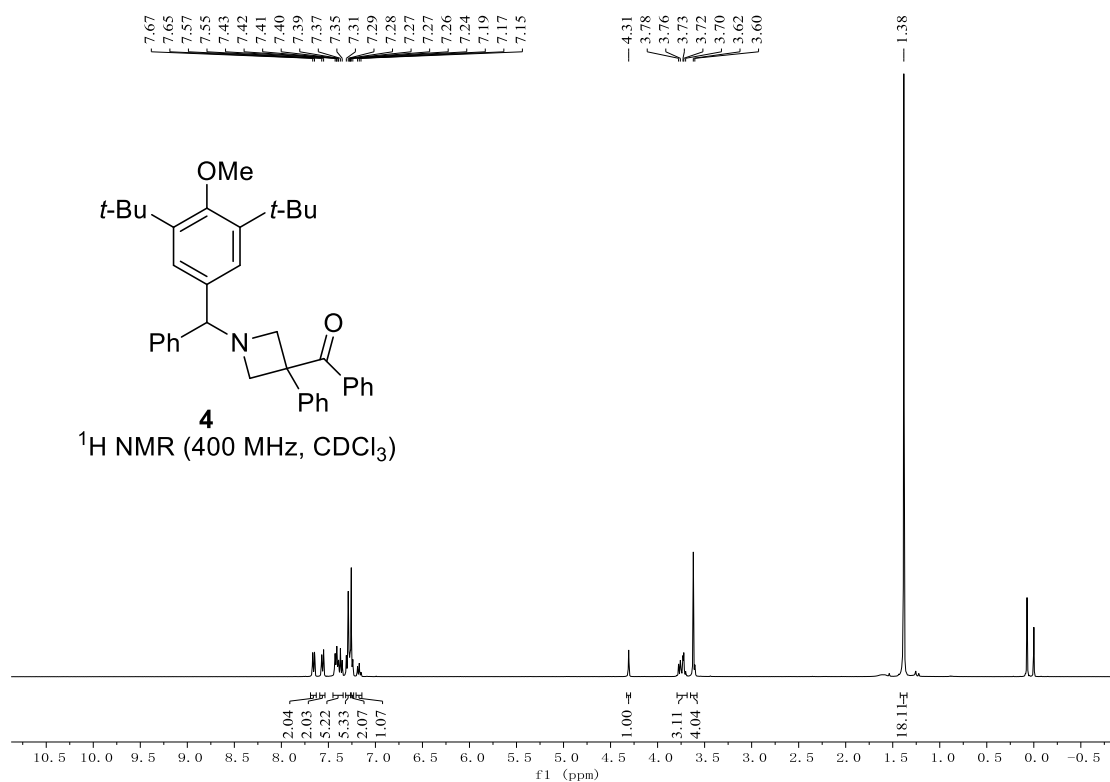
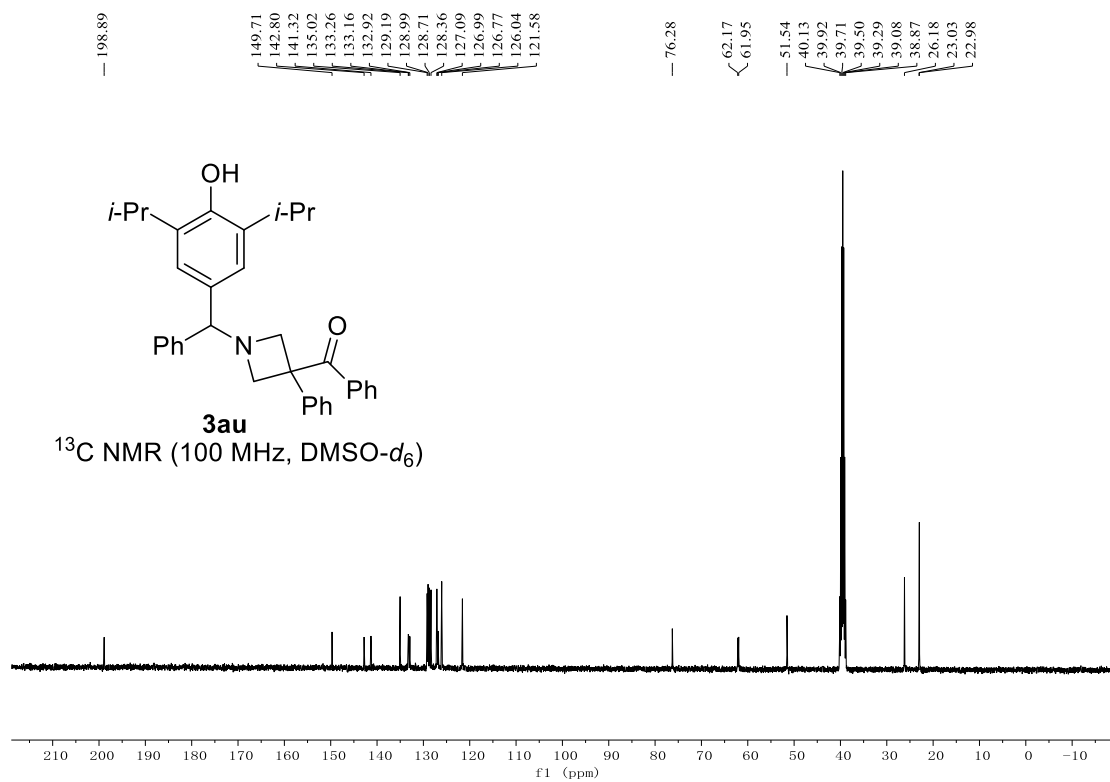


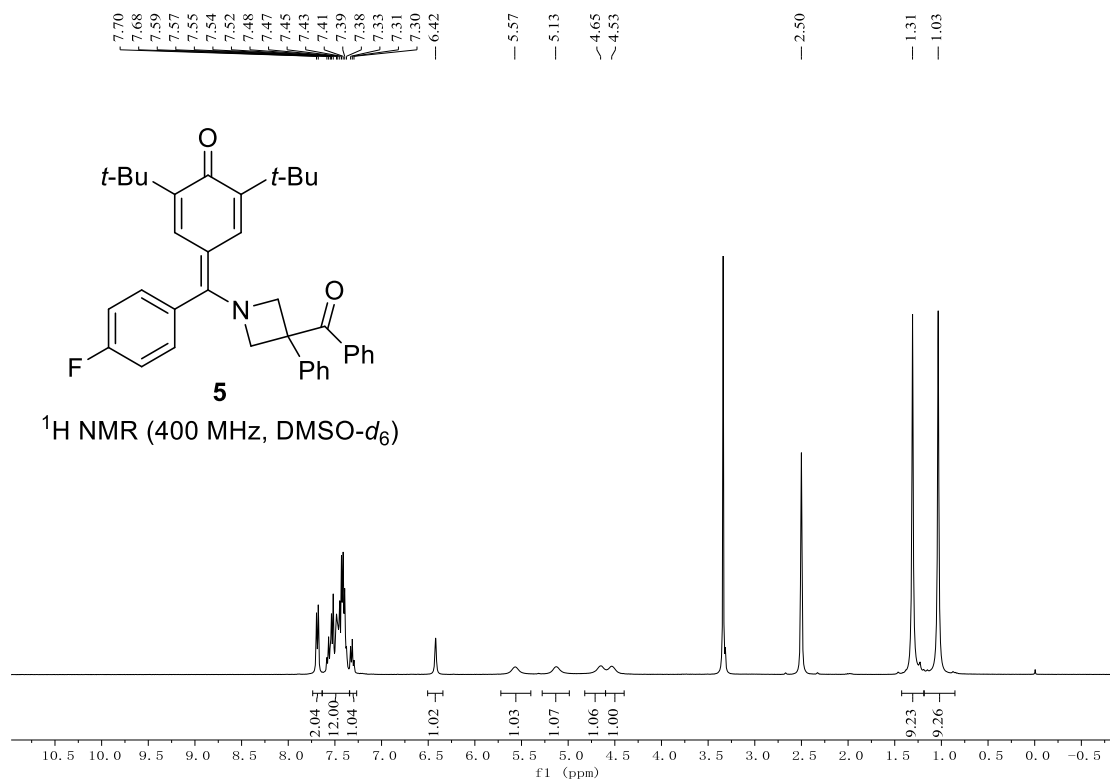
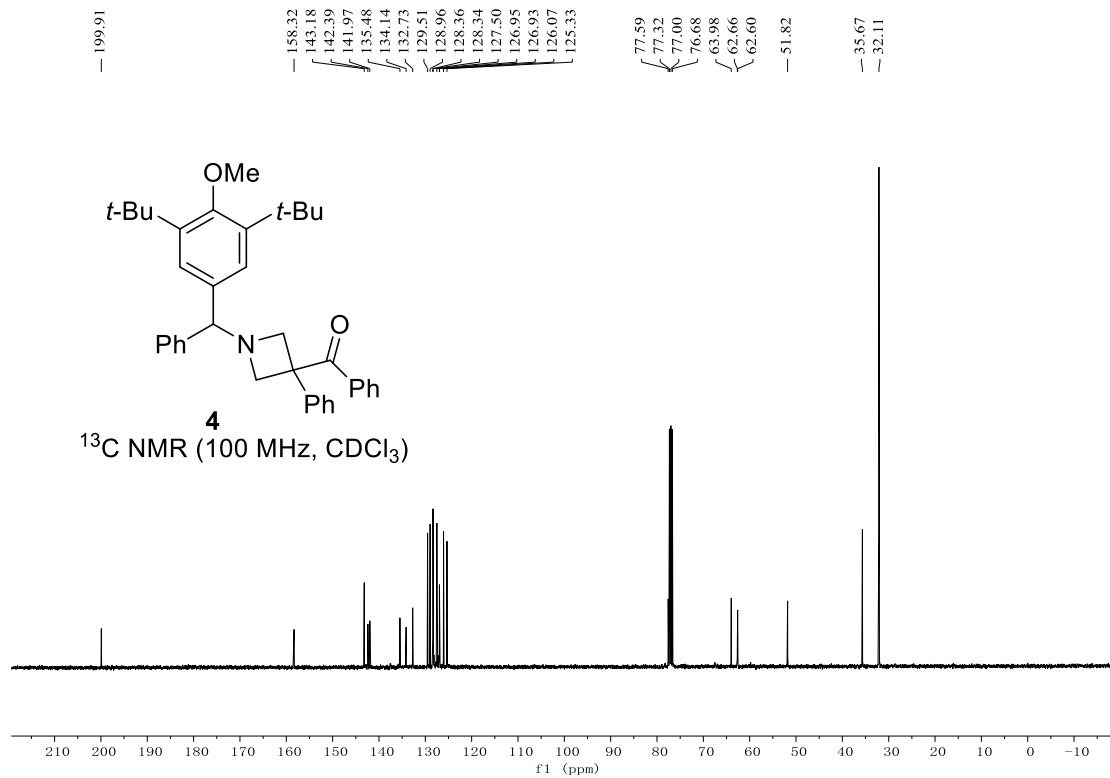


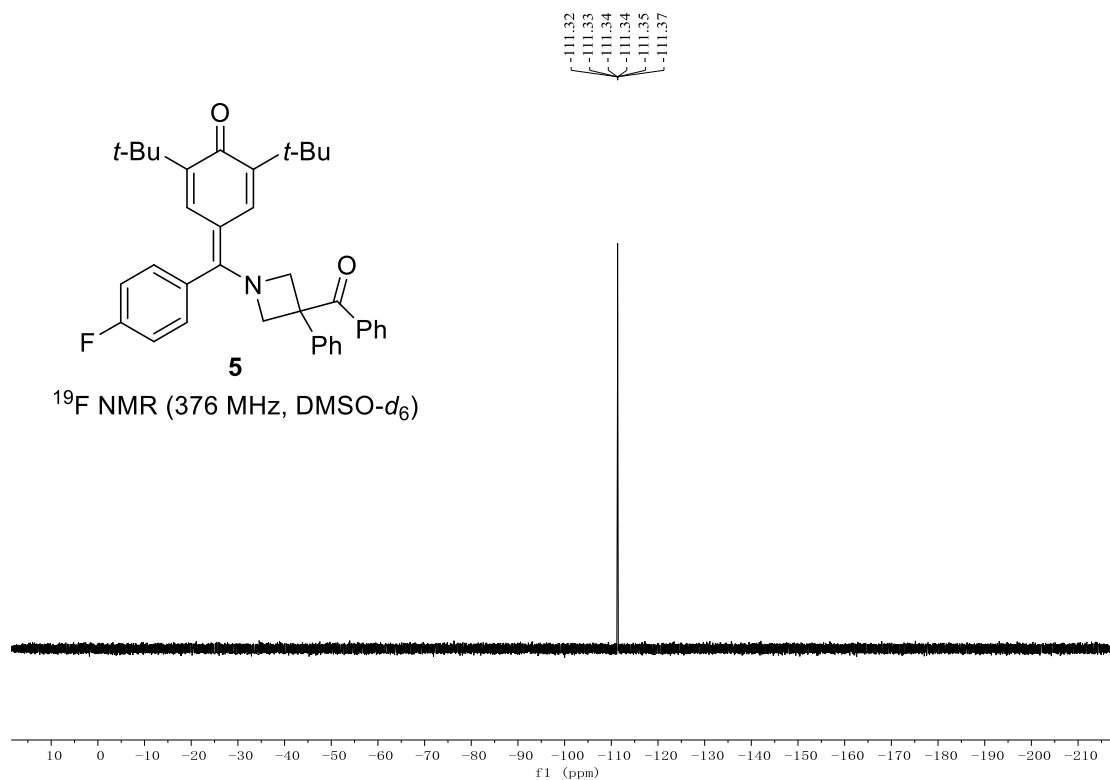
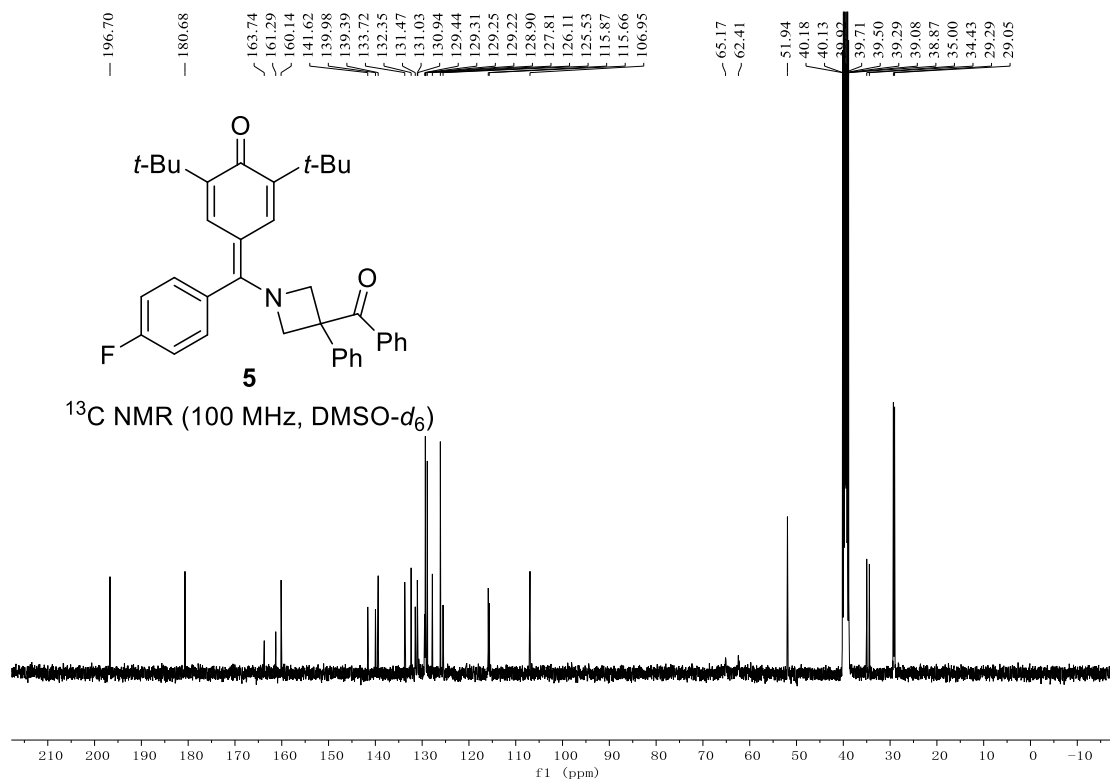


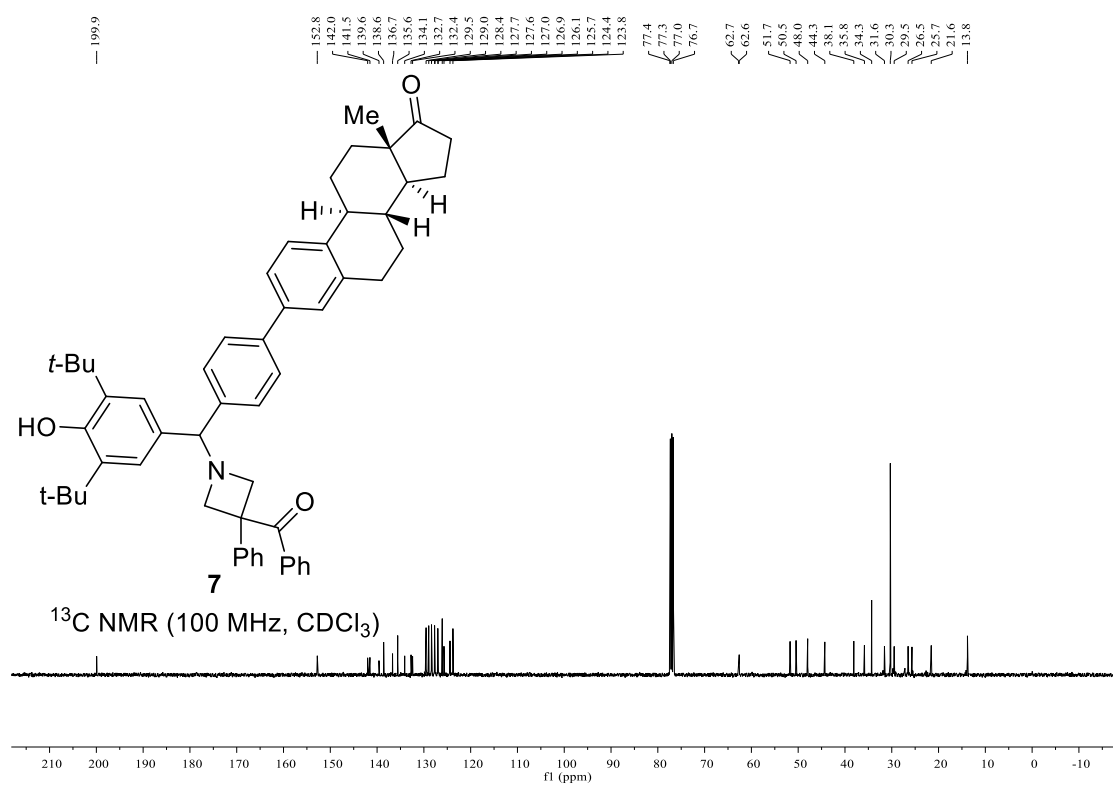
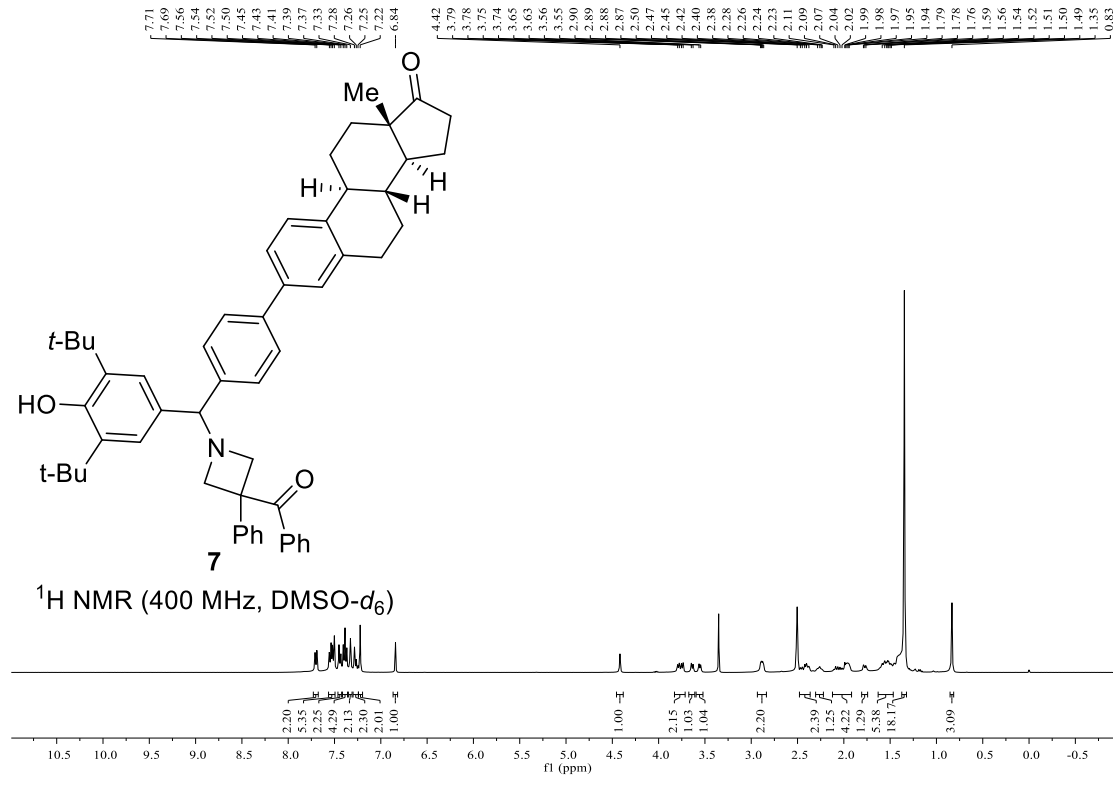


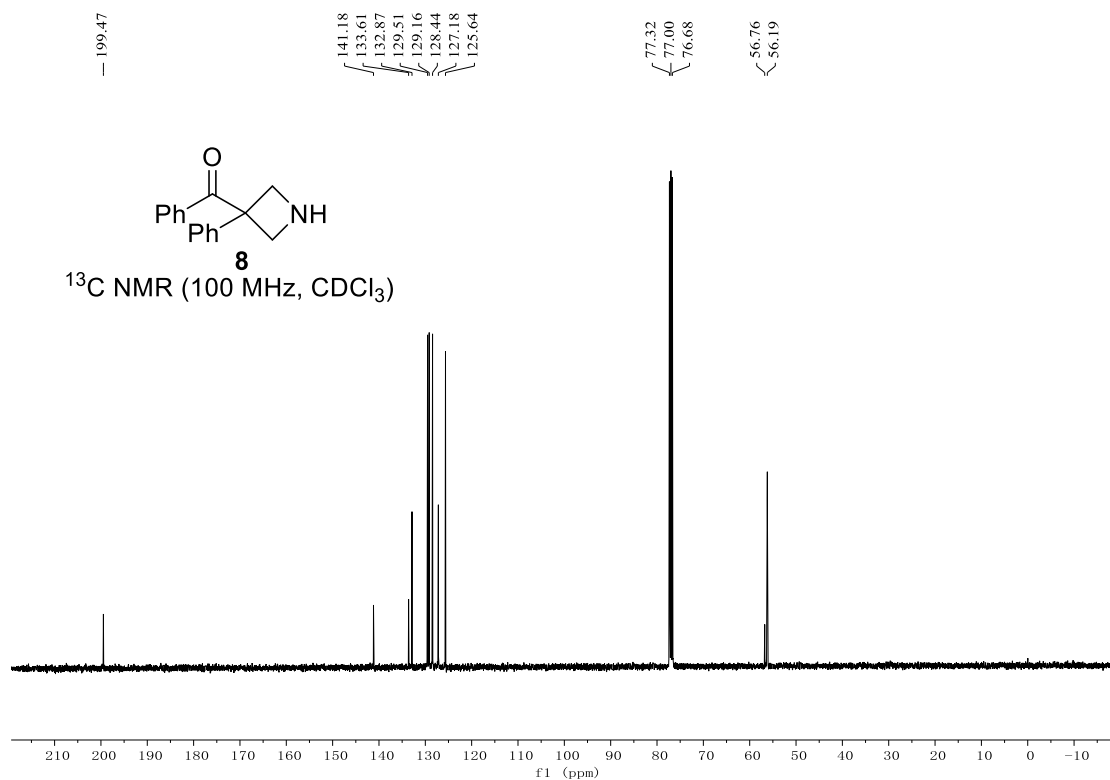
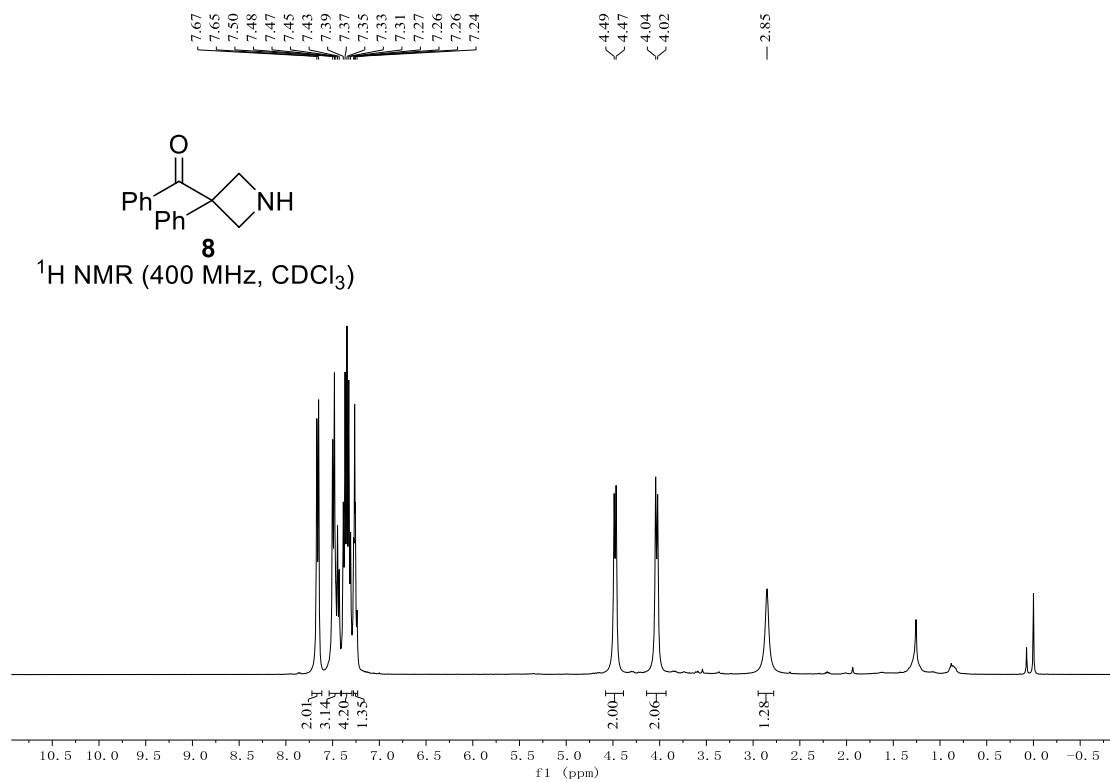






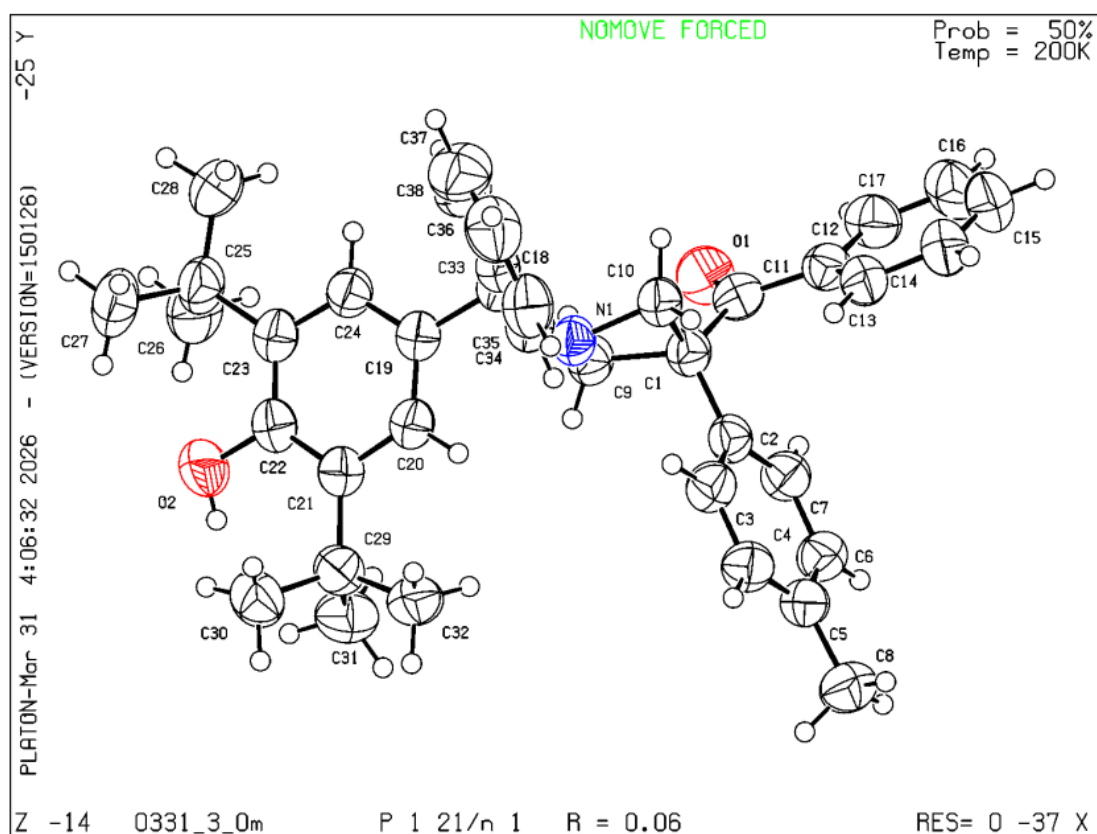
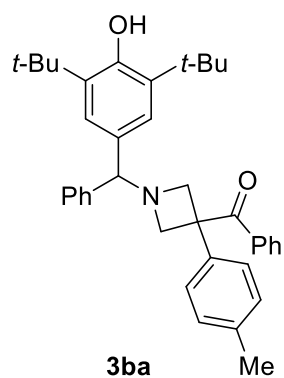






X-Ray crystallographic data

X-ray structure of product **3ba** (thermal ellipsoids are shown at 50% probability level). The crystal of product **3ba** was obtained by slow evaporation in ethyl acetate and petroleum ether. Crystal data have been deposited to CCDC, number 2542487.



Bond precision: C-C = 0.0023 Å Wavelength=1.34138
 Cell: a=12.2137(5) b=16.3186(6) c=17.2412(6)
 alpha=90 beta=110.720(1) gamma=90
 Temperature: 200 K

| | Calculated | Reported |
|------------------------|--------------|--------------|
| Volume | 3214.1(2) | 3214.1(2) |
| Space group | P 21/n | P 1 21/n 1 |
| Hall group | -P 2yn | -P 2yn |
| Moiety formula | C38 H43 N O2 | C38 H43 N O2 |
| Sum formula | C38 H43 N O2 | C38 H43 N O2 |
| Mr | 545.73 | 545.73 |
| Dx, g cm ⁻³ | 1.128 | 1.128 |
| Z | 4 | 4 |
| Mu (mm ⁻¹) | 0.335 | 0.349 |
| F000 | 1176.0 | 1176.0 |
| F000' | 1178.33 | |
| h, k, lmax | 15, 20, 21 | 15, 20, 21 |
| Nref | 6605 | 6568 |
| Tmin, Tmax | 0.939, 0.966 | 0.011, 0.069 |
| Tmin' | 0.933 | |

Correction method= # Reported T Limits: Tmin=0.011 Tmax=0.069

AbsCorr = MULTI-SCAN

Data completeness= 0.994

Theta(max)= 57.017

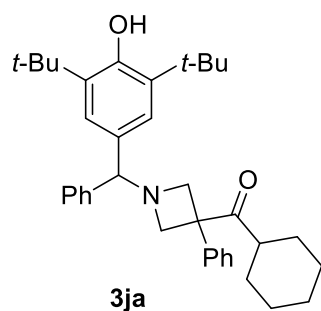
R(reflections)= 0.0557(5416)

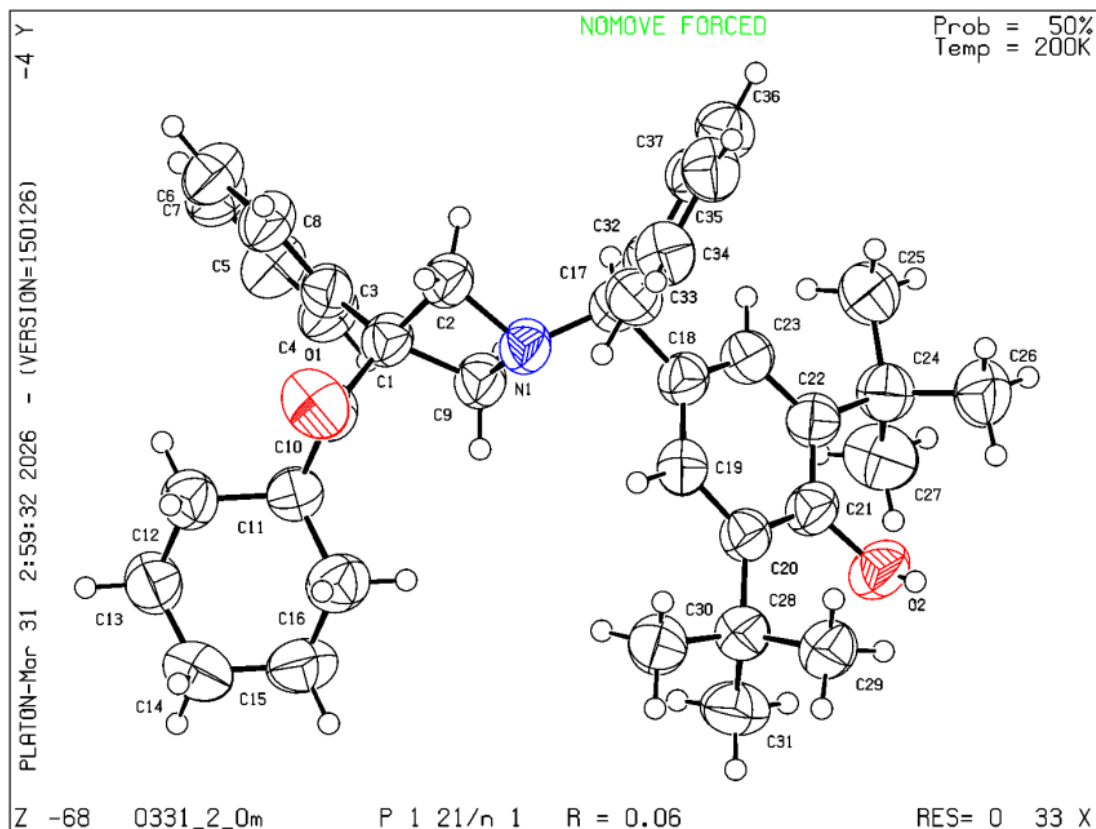
wR2(reflections)=
0.1753(6568)

S = 1.071

Npar= 377

X-ray structure of product **3ja** (thermal ellipsoids are shown at 50% probability level). The crystal of product **3ja** was obtained by slow evaporation in ethyl acetate and petroleum ether. Crystal data have been deposited to CCDC, number 2542486.





Bond precision: C-C = 0.0032 Å

Wavelength=1.34138

Cell: a=9.7604 (5)

b=31.8875 (15)

c=10.8660 (4)

alpha=90

beta=109.571 (2)

gamma=90

Temperature: 200 K

| | Calculated | Reported |
|------------------------|--------------|--------------|
| Volume | 3186.5 (3) | 3186.5 (3) |
| Space group | P 21/n | P 1 21/n 1 |
| Hall group | -P 2yn | -P 2yn |
| Moiety formula | C37 H47 N O2 | C37 H47 N O2 |
| Sum formula | C37 H47 N O2 | C37 H47 N O2 |
| Mr | 537.76 | 537.75 |
| Dx, g cm ⁻³ | 1.121 | 1.121 |
| Z | 4 | 4 |
| Mu (mm ⁻¹) | 0.331 | 0.345 |
| F000 | 1168.0 | 1168.0 |
| F000' | 1170.28 | |
| h, k, lmax | 12, 39, 13 | 12, 39, 13 |
| Nref | 6561 | 6496 |
| Tmin, Tmax | 0.940, 0.966 | 0.010, 0.069 |
| Tmin' | 0.933 | |

Correction method= # Reported T Limits: Tmin=0.010 Tmax=0.069

AbsCorr = MULTI-SCAN

Data completeness= 0.990

Theta(max)= 57.163

R(reflections)= 0.0595(4494)

wR2(reflections)=
0.1784(6496)

S = 1.093

Npar= 367

X-ray structure of product **3af** (thermal ellipsoids are shown at 50% probability level). The crystal of product **3af** was obtained by slow evaporation in ethyl acetate and petroleum ether. Crystal data have been deposited to CCDC, number 2521052.

