

Supporting Information

Copper-Mediated C(sp³)-H Amination in a Multiple C-N Bond-Forming Strategy for the Synthesis of N-Heterocycles**

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I. General Information

All reagents were purchased without further purification unless otherwise noted. Reactions were monitored using thin-layer chromatography (TLC) on commercial silica gel plates (GF254). Visualization of the developed plates was performed under UV light (254 nm). Flash column chromatography was performed on silica gel (200-300 mesh). ^1H and ^{13}C NMR spectra were recorded on a 400 or 500 MHz spectrometer. Chemical shifts (δ) were reported in ppm referenced to an internal tetramethylsilane standard or the DMSO- d_6 residual peak (δ 2.50) for ^1H NMR. Chemical shifts of ^{13}C NMR were reported relative to DMSO- d_6 (δ 39.5). The following abbreviations were used to describe peak splitting patterns when appropriate: br = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. Coupling constants, J , were reported in Hertz unit (Hz). High resolution mass spectra (HRMS) were obtained on an ESI-LC-MS/MS spectrometer.

II. General Procedures

(a) substrate preparation

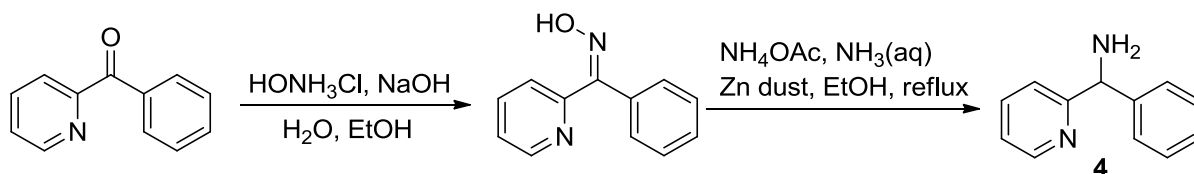
Substrates **1** were prepared according to literature reported procedures.^{1a-c}

(b) General Procedure for the Synthesis of **3**

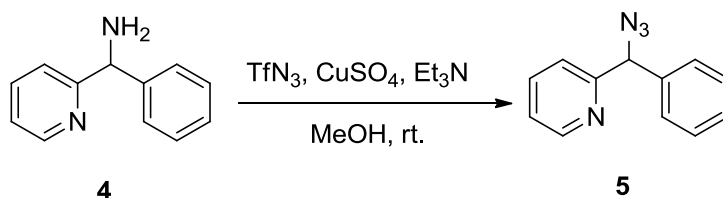
To a Schlenk tube were added 2-benzyl heterocycle **1** (0.4 mmol), aldehyde **2** (0.2 mmol), $\text{Cu}(\text{TFA})_2 \cdot x\text{H}_2\text{O}$ (0.24 mmol), and PivOH (0.2 mmol). Then, the tube was vacuumed and refilled with argon for 3 times, followed by introducing a solution of TMSN_3 (0.6 mmol) in 0.2 mL of DCB. The reaction mixture was stirred at 110 °C for 8 h. Then, a second portion of TMSN_3 (0.2 mmol, in 0.1 mL of DCB) was added and the solution was stirred for another 8 h. Then, the reaction was cooled down to room temperature before addition of saturated aqueous NaCl (10 mL), NH_4OH (1 mL) and EtOAc (10 mL) to the reaction mixture. The aqueous phase was further extracted with EtOAc (3 \times 10 mL). The combined organic layers were dried over anhydrous Na_2SO_4 and concentrated. The residue was purified by flash chromatography to provide the desired product **3**.

(c) Experiment Procedure for the Synthesis of **4** and **5**

The procedure for the synthesis of intermediate **4** was according to literature reported method.^{1d}

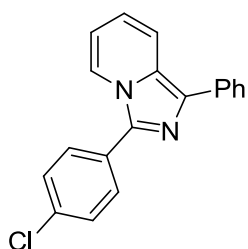


The procedure for the synthesis of intermediate **5** was started from intermediate **4**. To a solution of NaN_3 (261 mg, 4.01 mmol, 6.0 eq.) in a mixture of $\text{H}_2\text{O}/\text{CH}_2\text{Cl}_2$ (4 mL 1:1 v/v) at 0 °C, was added Tf_2O (337 μL , 2.01 mmol, 3.0 eq.). The mixture was stirred at 0 °C for 2 h. After quenching with saturated aqueous NaHCO_3 , the layers were separated and the aqueous layers were combined to afford 5 mL of TfN_3 solution. These TfN_3 was then added to a solution of **4** (222 mg, 1.2 mmol) in MeOH (5 mL), followed by H_2O (2 mL), a solution of CuSO_4 (22 mg, 0.1 eq.) in MeOH (2 mL), and Et_3N (594 μL , 4.0 mmol, 3.0 eq.). The reaction mixture was stirred overnight at rt. The saturated aqueous NaHCO_3 (25 mL) was added and the organic solvents were evaporated. The aqueous residue was extracted with EA (3×25 mL) and the organic layers were combined, dried (Na_2SO_4), and concentrated in vacuo to give the yellow oil, followed by purification by column chromatography to give the product **5** in 90% yield. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 8.60-8.59 (m, 1H), 7.71-7.67 (m, 1H), 7.39-7.29 (m, 6H), 7.22-7.19 (m, 1H), 5.80 (s, 1H); $^{13}\text{C NMR}$ (125 MHz, CDCl_3): δ 159.1, 149.5, 138.4, 137.0, 128.8, 128.3, 127.6, 122.8, 121.4, 69.5; HRMS (ESI) calcd for $\text{C}_{12}\text{H}_{10}\text{N}_4$ $[\text{M}+\text{H}]^+$ 211.0978, found 211.0976.



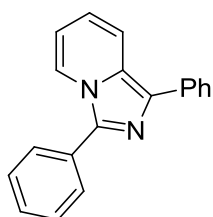
IV. Analytical Data

3-(4-chlorophenyl)-1-phenylimidazo[1,5-*a*]pyridine (**3a**)²



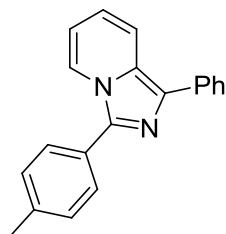
Light yellow solid, 44 mg, 72% yield, eluent: petroleum ether/ethyl acetate 20/1. ¹H NMR (400 MHz, CDCl₃): δ 8.20 (d, *J* = 7.2 Hz, 1H), 7.92 (d, *J* = 8.0 Hz, 2H), 7.86 (d, *J* = 9.2 Hz, 1H), 7.80 (d, *J* = 8.0 Hz, 2H), 7.53-7.46 (m, 4H), 7.31 (dd, *J* = 7.6, 7.2 Hz, 1H), 6.83 (dd, *J* = 8.8, 6.8 Hz, 1H), 6.62 (dd, *J* = 6.8, 6.4 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃): δ 136.9, 134.8, 134.7, 132.4, 129.5, 129.3, 128.8, 128.7, 127.9, 126.8, 126.7, 121.5, 119.8, 119.3, 113.6.

1,3-diphenylimidazo[1,5-*a*]pyridine (**3b**)³



Light yellow solid, 27 mg, 50% yield, eluent: petroleum ether/ethyl acetate 20/1. ¹H NMR (400 MHz, CDCl₃): δ 8.25 (d, *J* = 7.2 Hz, 1H), 7.95 (d, *J* = 8.0 Hz, 2H), 7.86-7.84 (m, 3H), 7.54 (t, *J* = 7.6 Hz, 2H), 7.49-7.44 (m, 3H), 7.30 (dd, *J* = 7.6, 7.2 Hz, 1H), 6.80 (dd, *J* = 9.2, 6.4 Hz, 1H), 6.58 (t, *J* = 6.8 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃): δ 138.1, 134.9, 132.0, 129.0, 128.8, 128.7, 128.4, 127.7, 126.8, 126.6, 121.8, 119.7, 119.2, 113.2.

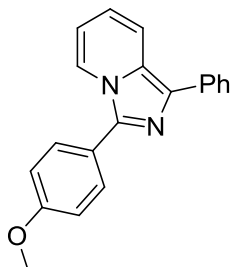
1-phenyl-3-(*p*-tolyl)imidazo[1,5-*a*]pyridine (**3c**)



Light yellow solid, 35 mg, 62% yield, eluent: petroleum ether/ethyl acetate 20/1. ¹H NMR (400 MHz, CDCl₃): δ 8.21 (d, *J* = 7.2 Hz, 1H), 7.94 (d, *J* = 7.6 Hz, 2H), 7.84 (d, *J* = 9.2 Hz, 1H), 7.73 (d, *J* = 8.0 Hz, 2H), 7.47 (t, *J* = 7.6 Hz, 2H), 7.35 (d, *J* = 8.0 Hz, 2H), 7.30 (dd, *J* = 7.6, 7.2 Hz, 1H),

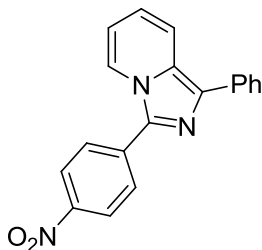
6.77 (dd, $J = 9.2, 6.4$ Hz, 1H), 6.56 (dd, $J = 7.2, 6.4$ Hz, 1H), 2.45 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 138.8, 138.3, 135.0, 131.8, 129.7, 128.7, 128.3, 127.5, 127.3, 126.8, 126.4, 121.9, 119.5, 119.2, 113.1, 21.4; FT-IR (film): 3038, 2919, 2851, 1600, 1534, 1515, 1332, 824, 769, 696 cm^{-1} ; HRMS (ESI) calcd for $\text{C}_{20}\text{H}_{16}\text{N}_2$ $[\text{M}+\text{H}]^+$ 285.1386, found 285.1385; mp: 129-131 $^\circ\text{C}$.

3-(4-methoxyphenyl)-1-phenylimidazo[1,5-*a*]pyridine (**3d**)³



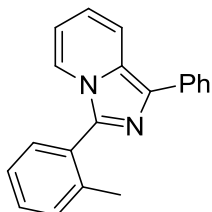
Light yellow solid, 39.6 mg, 66% yield, eluent: petroleum ether/ethyl acetate 10/1. ^1H NMR (400 MHz, CDCl_3): δ 8.17 (d, $J = 7.2$ Hz, 1H), 7.93 (d, $J = 7.6$ Hz, 2H), 7.83 (d, $J = 9.2$ Hz, 1H), 7.76 (d, $J = 8.4$ Hz, 2H), 7.46 (t, $J = 7.6$ Hz, 2H), 7.29 (t, $J = 7.2$ Hz, 1H), 7.07 (d, $J = 8.4$ Hz, 2H), 6.77 (dd, $J = 9.2, 6.4$ Hz, 1H), 6.55 (t, $J = 6.8$ Hz, 1H), 3.89 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 160.4, 157.9, 142.0, 135.6, 130.0, 128.8, 127.2, 127.0, 126.9, 121.8, 119.2, 114.5, 113.7, 55.4.

3-(4-nitrophenyl)-1-phenylimidazo[1,5-*a*]pyridine (**3e**)²



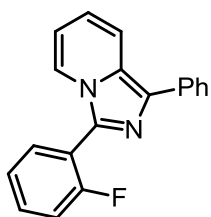
Yellow solid, 43.5 mg, 69% yield, eluent: petroleum ether/ethyl acetate 10/1. ^1H NMR (400 MHz, CDCl_3): δ 8.40-8.35 (m, 3H), 8.10 (d, $J = 8.8$ Hz, 2H), 7.93 (d, $J = 7.2$ Hz, 3H), 7.50 (t, $J = 7.6$ Hz, 2H), 7.35 (dd, $J = 7.6, 7.2$ Hz, 1H), 6.92 (dd, $J = 8.8, 6.4$ Hz, 1H), 6.75 (t, $J = 6.8$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3): δ 147.1, 136.4, 135.6, 134.3, 133.9, 129.1, 128.9, 128.1, 127.1, 126.9, 124.4, 121.6, 120.8, 119.6, 114.6.

1-phenyl-3-(*o*-tolyl)imidazo[1,5-*a*]pyridine (**3f**)



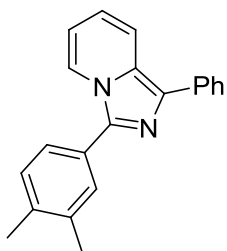
Kelly gum, 38.6 mg, 68% yield, eluent: petroleum ether/ethyl acetate 20/1. ^1H NMR (400 MHz, $\text{DMSO-}d_6$): δ 8.47 (d, $J = 7.2$ Hz, 1H), 8.00 (d, $J = 9.2$ Hz, 1H), 7.95 (d, $J = 7.6$ Hz, 2H), 7.69-7.66 (m, 2H), 7.47 (t, $J = 7.6$ Hz, 3H), 7.33-7.27 (m, 2H), 6.97 (dd, $J = 7.2, 6.8$ Hz, 1H), 6.78 (t, $J = 6.8$ Hz, 1H), 2.43 (s, 3H); ^{13}C NMR (125 MHz, $\text{DMSO-}d_6$): δ 138.3, 137.4, 134.7, 130.3, 129.6, 129.3, 128.8, 128.6, 128.4, 127.2, 126.1, 125.9, 124.9, 122.5, 120.8, 118.5, 113.6, 21.0; FT-IR (film): 3048, 2919, 2851, 1602, 1582, 1518, 1333, 1312, 1016, 770, 740, 698 cm^{-1} ; HRMS (ESI) calcd for $\text{C}_{20}\text{H}_{16}\text{N}_2$ $[\text{M}+\text{H}]^+$ 285.1386, found 235.1388.

3-(2-fluorophenyl)-1-phenylimidazo[1,5-*a*]pyridine (**3g**)



Kelly solid, 42 mg, 73% yield, eluent: petroleum ether/ethyl acetate 20/1. ^1H NMR (400 MHz, $\text{DMSO-}d_6$): δ 8.02 (d, $J = 11.6$ Hz, 1H), 7.99-7.94 (m, 3H), 7.78 (dd, $J = 7.6, 7.2$ Hz, 1H), 7.67-7.62 (m, 1H), 7.51-7.43 (m, 4H), 7.30 (dd, $J = 7.6, 7.2$ Hz, 1H), 7.02 (dd, $J = 8.8, 6.8$ Hz, 1H), 6.81 (t, $J = 6.8$ Hz, 1H); ^{13}C NMR (125 MHz, $\text{DMSO-}d_6$): 161.0, 159.0, 134.0 (d, $J = 240.8$ Hz), 132.6 (d, $J = 3.6$ Hz), 132.1 (d, $J = 8.1$ Hz), 131.1, 129.2, 127.7, 126.7, 126.4, 125.6 (d, $J = 3.3$ Hz), 123.5 (d, $J = 5.6$ Hz), 121.6, 118.8, 117.9 (d, $J = 15.0$ Hz), 116.8 (d, $J = 21.0$ Hz), 114.0; FT-IR (film): 3053, 2921, 2851, 1602, 1571, 1518, 1452, 1214, 1204, 1073, 759, 659 cm^{-1} ; HRMS (ESI) calcd for $\text{C}_{19}\text{H}_{13}\text{FN}_2$ $[\text{M}+\text{H}]^+$ 289.1136, found 289.1137; mp: 93-95 $^\circ\text{C}$.

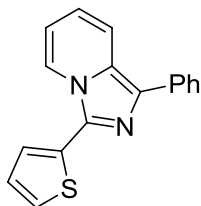
3-(3,4-dimethylphenyl)-1-phenylimidazo[1,5-*a*]pyridine (**3h**)



Kelly solid, 39.9 mg, 67% yield, eluent: petroleum ether/ethyl acetate 20/1. ^1H NMR (400 MHz, CDCl_3): δ 8.23 (d, $J = 7.2$ Hz, 1H), 7.94 (d, $J = 8.0$ Hz, 2H), 7.83 (d, $J = 9.2$ Hz, 1H), 7.64 (s, 1H), 7.55 (d, $J = 8.0$ Hz, 1H), 7.46 (t, $J = 7.6$ Hz, 2H), 7.31-7.28 (m, 2H), 6.77 (dd, $J = 8.8, 6.4$ Hz, 1H), 6.56 (t, $J = 6.8$ Hz, 1H), 2.36 (s, 3H), 2.35 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 138.4, 137.6, 137.5, 135.0, 131.7, 130.1, 129.7, 128.7, 127.5, 126.8, 126.4, 125.5, 121.9, 119.5, 119.1, 113.0, 19.8, 19.7; FT-IR (film): cm^{-1} 3052, 2916, 2858, 1603, 1541, 1518, 1470, 1307, 1017, 835, 771, 744, 698

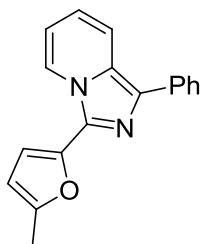
cm⁻¹; HRMS (ESI) calcd for C₂₁H₁₉N₂ [M+H]⁺ 299.1543, found 299.1544; mp: 107-109 °C.

1-phenyl-3-(thiophen-2-yl)imidazo[1,5-*a*]pyridine (**3i**)



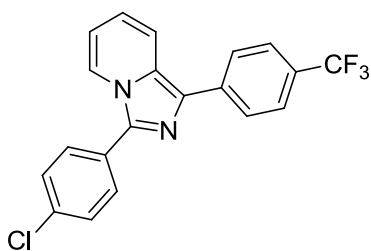
Kelly solid, 45.8 mg, 83% yield, eluent: petroleum ether/ethyl acetate 20/1. ¹H NMR (400 MHz, CDCl₃): δ 8.33 (d, *J* = 7.2 Hz, 1H), 7.94 (d, *J* = 8.0 Hz, 2H), 7.85 (d, *J* = 9.2 Hz, 1H), 7.57 (d, *J* = 3.2 Hz, 1H), 7.49-7.44 (m, 3H), 7.31 (dd, *J* = 7.6, 7.2 Hz, 1H), 7.20 (dd, *J* = 4.8, 4.0 Hz, 1H), 6.81 (dd, *J* = 9.2, 6.4 Hz, 1H), 6.67 (t, *J* = 6.8 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃): δ 134.7, 132.7, 132.3, 132.2, 128.7, 127.9, 127.6, 126.9, 126.7, 126.2, 125.3, 122.0, 119.6, 119.2, 113.8; FT-IR (film): 3069, 2922, 2851, 1602, 1515, 1250, 1074, 849, 770, 1073, 692 cm⁻¹; HRMS (ESI) calcd for C₁₇H₁₂N₂S [M+H]⁺ 277.0794, found 277.0793; mp: 75-77 °C.

3-(5-methylfuran-2-yl)-1-phenylimidazo[1,5-*a*]pyridine (**3j**)



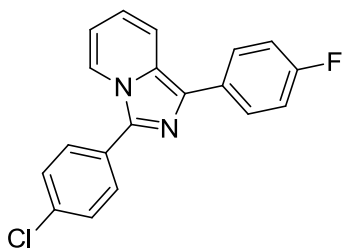
Yellow solid, 29 mg, 53% yield, eluent: petroleum ether/ethyl acetate 20/1. ¹H NMR (400 MHz, CDCl₃): δ 8.59 (d, *J* = 7.2 Hz, 1H), 7.91 (d, *J* = 8.0 Hz, 2H), 7.82 (d, *J* = 9.2 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 7.3 (dd, *J* = 7.6, 7.2 Hz, 1H), 6.94 (d, *J* = 2.8 Hz, 1H), 6.80 (dd, *J* = 9.2, 6.4 Hz, 1H), 6.66 (dd, *J* = 6.8, 6.4 Hz, 1H), 6.19 (d, *J* = 2.8 Hz, 1H), 2.46 (s, 3H); ¹³C NMR (125 MHz, CDCl₃): δ 152.3, 144.4, 134.8, 132.2, 130.5, 128.7, 127.2, 127.0, 126.6, 123.2, 119.6, 119.0, 113.5, 109.8, 107.8, 13.8; FT-IR (film): 3115, 3048, 2919, 2851, 1601, 1571, 1519, 1363, 1333, 1308, 1252, 1016, 957, 904, 803, 770, 696 cm⁻¹; HRMS (ESI) calcd for C₁₈H₁₄N₂O [M+H]⁺ 275.1179, found 275.1178; mp: 99-101 °C.

3-(4-chlorophenyl)-1-(4-(trifluoromethyl)phenyl)imidazo[1,5-*a*]pyridine (**3k**)



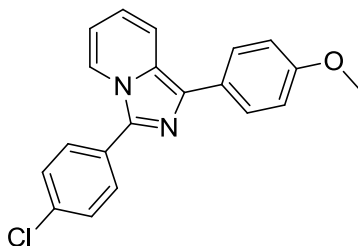
Yellow solid, 33 mg, 44% yield, eluent: petroleum ether/ethyl acetate 20/1. ^1H NMR (400 MHz, CDCl_3): δ 8.23 (d, $J = 7.2$ Hz, 1 H), 8.05 (d, $J = 8.0$ Hz, 2H), 7.87 (d, $J = 9.2$ Hz, 1H), 7.79 (d, $J = 8.4$ Hz, 2H), 7.71 (d, $J = 8.0$ Hz, 2H), 7.53 (d, $J = 8.4$ Hz, 2H), 6.91 (dd, $J = 8.8, 6.4$ Hz, 1H), 6.67 (t, $J = 6.8$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3): 149.9, 138.3, 137.4 (q, $J = 32.0$ Hz), 135.0, 129.6 (q, $J = 134$ Hz), 129.4 (q, $J = 21.6$ Hz), 128.6, 128.3, 126.9, 126.5, 125.6 (q, $J = 3.9$ Hz), 125.3, 121.8, 120.9, 118.8, 113.8; FT-IR (film): 3066, 2917, 2850, 1734, 1604, 1500, 1489, 1476, 1335, 1312, 1239, 1042, 837, 1011, 837, 703 cm^{-1} ; HRMS (ESI) calcd for $\text{C}_{20}\text{H}_{12}\text{ClF}_3\text{N}_2$ $[\text{M}+\text{H}]^+$ 373.0714, found 373.0713; mp: 177-179 $^\circ\text{C}$.

3-(4-chlorophenyl)-1-(4-fluorophenyl)imidazo[1,5-*a*]pyridine (**3l**)



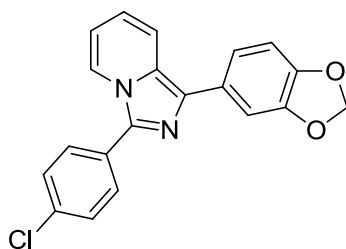
Yellow solid, 31 mg, 48% yield, eluent: petroleum ether/ethyl acetate 20/1. ^1H NMR (400 MHz, CDCl_3): δ 8.19 (d, $J = 6.8$ Hz, 1H), 7.87 (dd, $J = 8.4, 5.6$ Hz, 2H), 7.78 (d, $J = 8.4$ Hz, 3H), 7.51 (d, $J = 8.0$ Hz, 2H), 7.16 (dd, $J = 8.8, 8.4$ Hz, 2H), 6.82 (dd, $J = 8.8, 6.4$ Hz, 1H), 6.62 (t, $J = 6.8$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3): δ 161.9 (d, $J = 245.8$ Hz), 136.9, 134.7, 131.5, 130.9 (d, $J = 3.7$ Hz), 129.4, 129.3, 128.6, 128.4 (d, $J = 7.9$ Hz), 127.7, 121.6, 119.9, 115.6 (d, $J = 21.7$ Hz), 113.6; FT-IR (film): 3069, 2919, 2850, 1597, 1539, 1518, 1500, 1403, 1354, 1223, 1092, 1011, 838, 765, 734, 700, 679, 568 cm^{-1} ; HRMS (ESI) calcd for $\text{C}_{19}\text{H}_{12}\text{ClFN}_2$ $[\text{M}+\text{H}]^+$ 323.0746, found 323.0747; mp: 87-89 $^\circ\text{C}$.

3-(4-chlorophenyl)-1-(4-methoxyphenyl)imidazo[1,5-*a*]pyridine (**3m**)



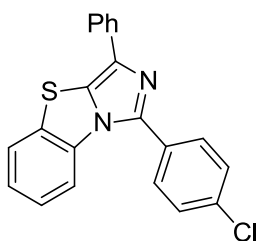
Yellow solid, 46.1 mg, 69% yield, eluent: petroleum ether/ethyl acetate 10/1. ^1H NMR (400 MHz, CDCl_3): δ 8.17 (d, $J = 7.2$ Hz, 1H), 7.84 (d, $J = 8.8$ Hz, 2H), 7.79 (d, $J = 8.4$ Hz, 3H), 7.51 (d, $J = 8.4$ Hz, 2H), 7.02 (d, $J = 8.8$ Hz, 2H), 6.77 (dd, $J = 8.8, 6.4$ Hz, 1H), 6.59 (t, $J = 6.8$ Hz, 1H); 3.87 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 158.7, 136.6, 134.6, 132.3, 129.4, 129.3, 128.7, 128.1, 127.4, 127.3, 121.4, 119.3, 119.3, 114.3, 113.6, 55.4; FT-IR (film): 3067, 3000, 2954, 2834, 1732, 1544, 1501, 1290, 1040, 841, 677 cm^{-1} ; HRMS (ESI) calcd for $\text{C}_{20}\text{H}_{15}\text{ClN}_2\text{O}$ $[\text{M}+\text{H}]^+$ 335.0946, found 335.0945; mp: 107-109 $^\circ\text{C}$.

1-(benzo[*d*]-[1,3]dioxol-5-yl)-3-(4-chlorophenyl)imidazo[1,5-*a*]pyridine (**3n**)



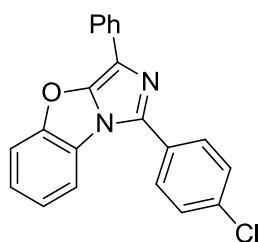
Yellow solid, 43.2 mg, 62% yield, eluent: petroleum ether/ethyl acetate 10/1. ^1H NMR (400 MHz, CDCl_3): δ 8.17 (d, $J = 6.8$ Hz, 1H), 7.78 (d, $J = 8.4$ Hz, 3H), 7.51 (d, $J = 8.4$ Hz, 2H), 7.41 (s, 1H), 7.37 (d, $J = 8.0$ Hz, 1H), 6.92 (d, $J = 8.0$ Hz, 1H), 6.78 (dd, $J = 8.8, 6.4$ Hz, 1H), 6.59 (t, $J = 6.8$ Hz, 1H), 6.0 (s, 2H); ^{13}C NMR (125 MHz, CDCl_3): δ 148.1, 146.6, 136.6, 134.6, 132.3, 129.4, 129.3, 129.1, 128.7, 127.4, 121.5, 120.3, 119.5, 119.2, 113.5, 108.6, 107.6, 101.0, 29.7; FT-IR (film): 3068, 2956, 2898, 2775, 1735, 1538, 1517, 1500, 1489, 1312, 1239, 1010, 985, 764, 603 cm^{-1} ; HRMS (ESI) calcd for $\text{C}_{20}\text{H}_{13}\text{ClN}_2\text{O}_2$ $[\text{M}+\text{H}]^+$ 349.0738, found 349.0737; mp: 92-94 $^\circ\text{C}$.

1-(4-chlorophenyl)-3-phenylbenzo[*d*]imidazo[5,1-*b*]thiazole (**3o**)



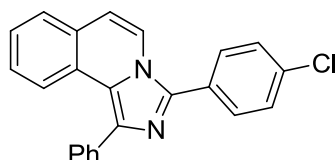
Yellow solid, 44.6 mg, 62% yield, eluent: petroleum ether/ethyl acetate 20/1. ^1H NMR (400 MHz, $\text{DMSO-}d_6$): δ 8.03 (d, $J = 7.2$ Hz, 1H), 7.85 (d, $J = 8.0$ Hz, 2H), 7.74 (d, $J = 7.6$ Hz, 2H), 7.70 (d, $J = 8.0$ Hz, 2H), 7.51 (dd, $J = 7.6, 7.2$ Hz, 2H), 7.46-7.43 (m, 3H), 7.29 (dd, $J = 7.6, 7.2$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3): δ 139.3, 134.4, 132.8, 132.6, 131.4, 131.0, 129.4, 129.0, 128.9, 126.5, 126.2, 125.9, 125.3, 125.1, 124.0, 114.0; FT-IR (film): 3050, 3021, 1604, 1547, 1461, 1402, 1373, 1070, 840, 748, 692 cm^{-1} ; HRMS (ESI) calcd for $\text{C}_{21}\text{H}_{13}\text{ClN}_2\text{S}$ $[\text{M}+\text{H}]^+$ 361.0561, found 361.0562; mp: 211-213 $^\circ\text{C}$.

1-(4-chlorophenyl)-3-phenylbenzo[*d*]imidazo[5,1-*b*]oxazole (**3p**)



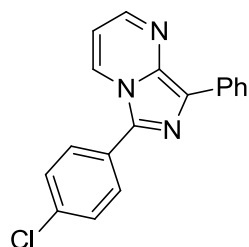
Yellow solid, 46.1 mg, 67% yield, eluent: petroleum ether/ethyl acetate 20/1. ¹H NMR (400 MHz, DMSO-*d*₆): δ 7.96 (d, *J* = 8.0 Hz, 2H), 7.88 (d, *J* = 7.6 Hz, 2H), 7.85-7.80 (t, *J* = 8.4 Hz, 2H), 7.68 (d, *J* = 7.6 Hz, 2H), 7.53-7.40 (m, 4H), 7.25 (dd, *J* = 7.2, 6.8 Hz, 1H); ¹³C NMR (125 MHz, DMSO-*d*₆): δ 153.9, 133.5, 132.1, 131.1, 129.1, 128.8, 128.8, 126.2, 125.9, 125.2, 124.2, 123.8, 113.1, 113.0; FT-IR (film): 3054, 1639, 1607, 1593, 1472, 1457, 1409, 1395, 1197, 1120, 847, 745 cm⁻¹; HRMS (ESI) calcd for C₂₁H₁₃ClN₂O [M+H]⁺ 345.0789, found 345.0787; mp: 215-217 °C.

3-(4-chlorophenyl)-1-phenylimidazo[5,1-*a*]isoquinoline (**3q**)



Yellow solid, 64 mg, 90% yield, eluent: petroleum ether/ethyl acetate 20/1. ¹H NMR (500 MHz, CDCl₃): δ 8.06 (d, *J* = 6.4 Hz, 1H), 7.95 (d, *J* = 6.0 Hz, 1H), 7.79-7.76 (m, 4H), 7.56 (d, *J* = 6.0 Hz, 1H), 7.53-7.50 (m, 4H), 7.44 (dd, *J* = 6.0, 5.6 Hz, 1H), 7.38 (dd, *J* = 6.0, 5.6 Hz, 1H), 7.32-7.29 (t, *J* = 6.0 Hz, 1H); 6.82 (d, *J* = 6.0 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃): δ 139.1, 136.2, 135.8, 135.1, 130.0, 129.8, 129.2, 128.6, 128.4, 128.1, 127.9, 127.6, 127.1, 127.0, 125.7, 124.3, 122.7, 120.4, 114.5; FT-IR (film): 3060, 3026, 2960, 1730, 1604, 1474, 1337, 1193, 836, 765, 747, 704, 508 cm⁻¹; HRMS (ESI) calcd for C₂₃H₁₅ClN₂ [M+H]⁺ 355.0997, found 355.0995; mp: 147-149 °C.

6-(4-chlorophenyl)-8-phenylimidazo[1,5-*a*]pyrimidine (**3r**)



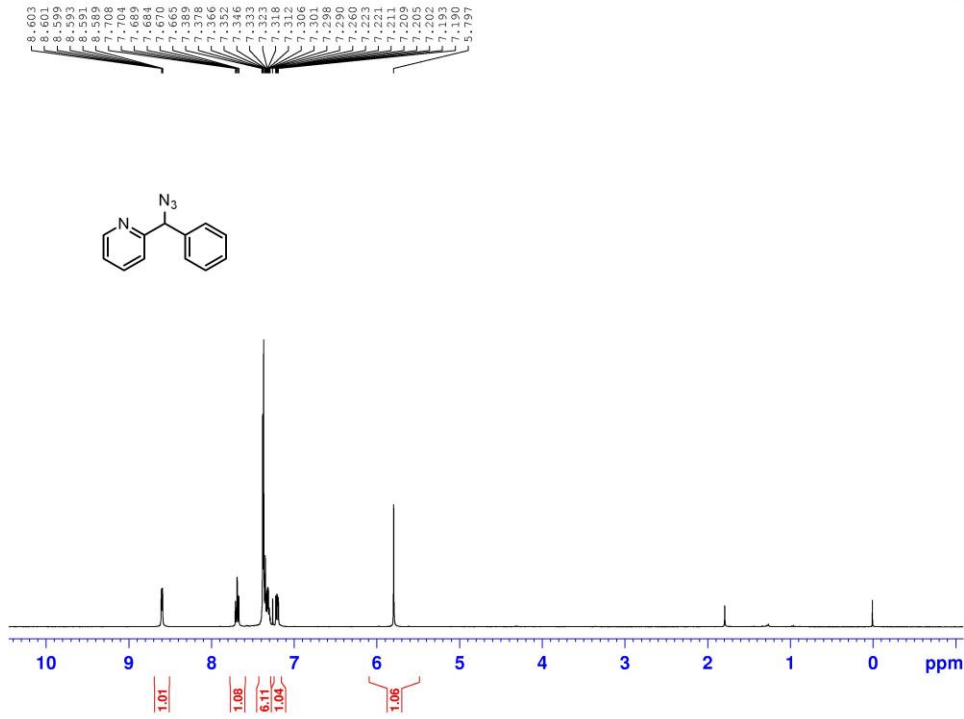
Yellow solid, 36.6 mg, 60% yield, eluent: petroleum ether/ethyl acetate 20/1. ¹H NMR (400 MHz, CDCl₃): δ 8.45-8.41 (m, 3H), 8.23 (s, 1H), 7.78 (d, *J* = 8.0 Hz, 2H), 7.53 (d, *J* = 8.0 Hz, 2H), 7.48 (t, *J* = 7.2 Hz, 2H), 7.30 (t, *J* = 6.8 Hz, 1H), 6.61 (d, *J* = 3.6 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃): δ

145.3, 135.5, 135.2, 133.9, 133.7, 130.3, 129.5, 129.2, 128.5, 128.3, 128.0, 126.9, 126.5, 109.1; FT-IR (film): 3095, 3064, 3026, 2918, 1612, 1601, 1501, 1446, 1403, 1290, 1072, 833, 776, 696 cm^{-1} ; HRMS (ESI) calcd for $\text{C}_{18}\text{H}_{12}\text{ClN}_3$ $[\text{M}+\text{H}]^+$ 306.0793, found 306.0794; mp: 231-233 °C.

V. References

- (1) (a) Shang, S.; Yang, Z. W.; Wang, Y.; Zhang, S. L.; Liu, L. *J. Am. Chem. Soc.* **2010**, *132*, 14391; (b) Desai, L. V.; Stowers, K. J.; Sanford, M. S. *J. Am. Chem. Soc.* **2008**, *130*, 13285; (c) Xu, Z.; Zhang, C.; Jiao, N. *Angew. Chem., Int. Ed.* **2012**, *51*, 11367; (d) Ritzen, B.; Oers, M. C. M.; Delft, F. L.; Rutjes, F. P. J. T. *J. Org. Chem.* **2009**, *74*, 7548.
- (2) Wang, H.; Xu, W.; Wang, Z.; Yu, L.; Xu, K. *J. Org. Chem.* **2015**, *80*, 2431.
- (3) Shibahara, F.; Yamaguchi, E.; Kitagawa, A.; Imai, A.; Murai, T. *Tetrahedron* **2009**, *65*, 5062.

VI. Copies of ^1H NMR and ^{13}C NMR Spectra



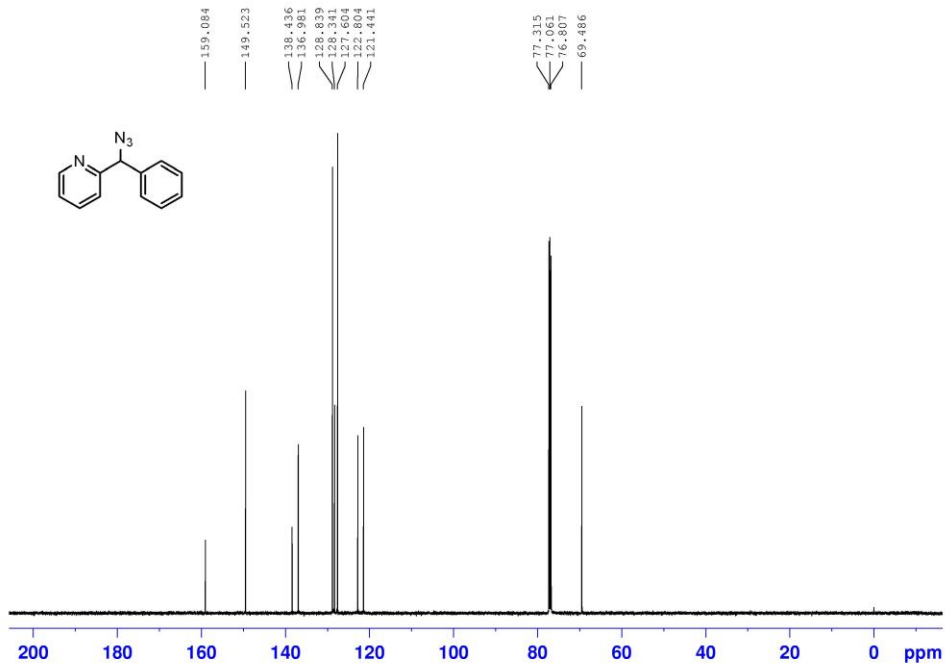
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NAME          24
EXPNO         1
PROCNO        1
Date_         20150908
Time         14.43
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            3
DS            2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG            114
DW            60.400 usec
DE            6.50 usec
TE            299.3 K
D1            1.00000000 sec
TD0           1
    
```

```

----- CHANNEL f1 -----
NUC1          1H
P1            12.58 usec
PL1           0.00 dB
PL12W         10.87646866 W
SFO1          400.1324710 MHz
SI            32768
SF            400.1300038 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

0027



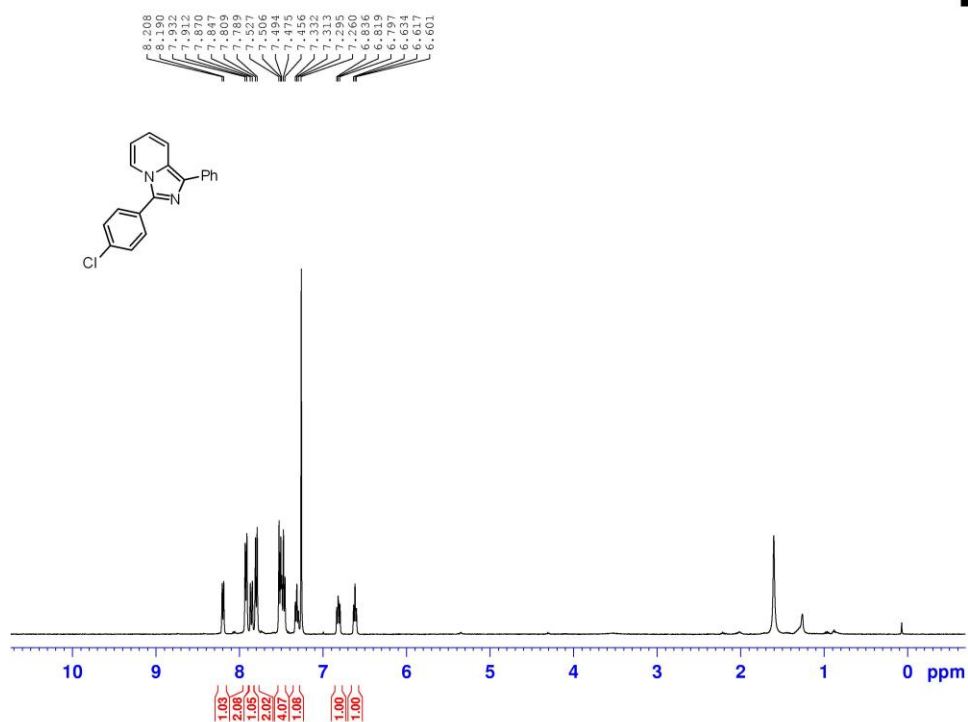
```

NAME          9
EXPNO         1
PROCNO        1
Date_         20150910
Time         2.09
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            1024
DS            4
SWH           29761.994 Hz
FIDRES        0.494331 Hz
AQ            1.1010548 sec
RG            203
DW            16.800 usec
DE            6.50 usec
TE            302.3 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1
    
```

```

----- CHANNEL f1 -----
NUC1          13C
P1            13.84 usec
PL1           2.50 dB
PL12W         46.89624786 W
SFO1          125.7705643 MHz
----- CHANNEL f2 -----
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           2.50 dB
PL12          17.40 dB
PL13          17.40 dB
PL12W         13.02359881 W
SFO2          0.42143536 MHz
SFO3          0.42143536 MHz
SI            32768
SF            125.7577890 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

3a



```

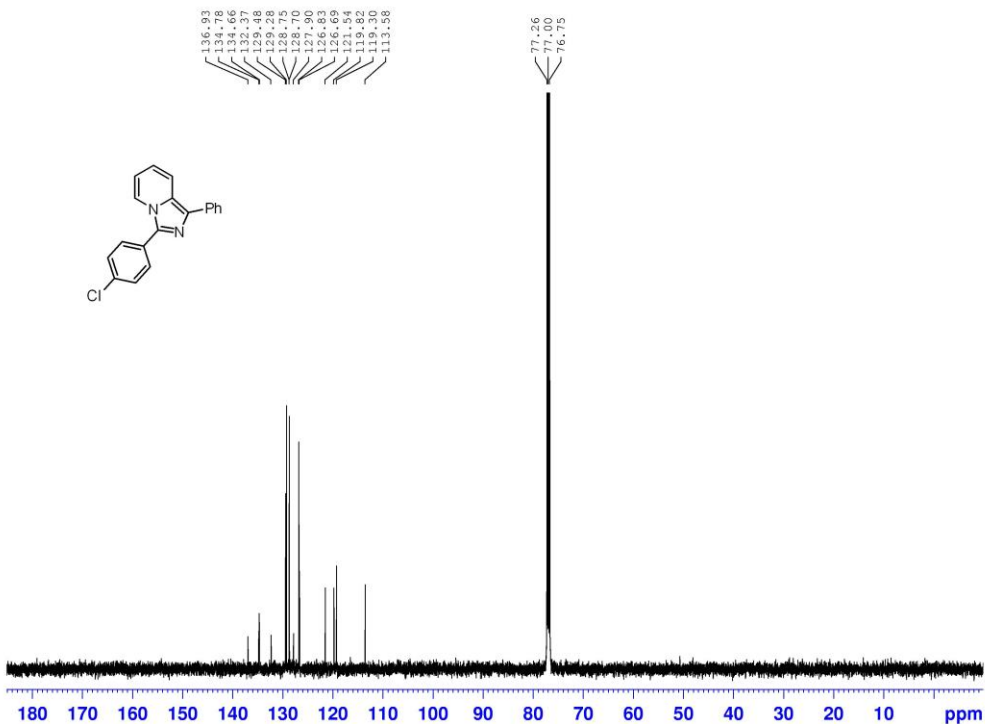
NAME          70196-3H
EXPNO         1
PROCNO        1
Date_         20141221
Time          15.50
INSTRUM       spect
PROBHD        5 mm DUL 13C-1
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8278.146 Hz
FIDRES       0.126514 Hz
AQ           3.9584243 sec
RG            362
RG            362
AQ           60.400 usec
DE            6.50 usec
TE            296.6 K
D1           1.0000000 sec
TD0          1
  
```

----- CHANNEL f1 -----

```

NUC1          1H
P1            12.55 usec
PL1           0.00 dB
PL1W         10.87646866 W
SFO1         400.1324710 MHz
SI            32768
SF           400.1300093 MHz
NEW          EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

70196-3



```

NAME          70196-3C
EXPNO         1
PROCNO        1
Date_         20141221
Time          17.32
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            1632
DS            4
SWH           29761.904 Hz
FIDRES       0.454131 Hz
AQ           1.1010548 sec
RG            525
RG            525
AQ           16.800 usec
DE            6.50 usec
TE            297.8 K
D1           2.0000000 sec
D11          0.0300000 sec
TD0          1
  
```

----- CHANNEL f1 -----

```

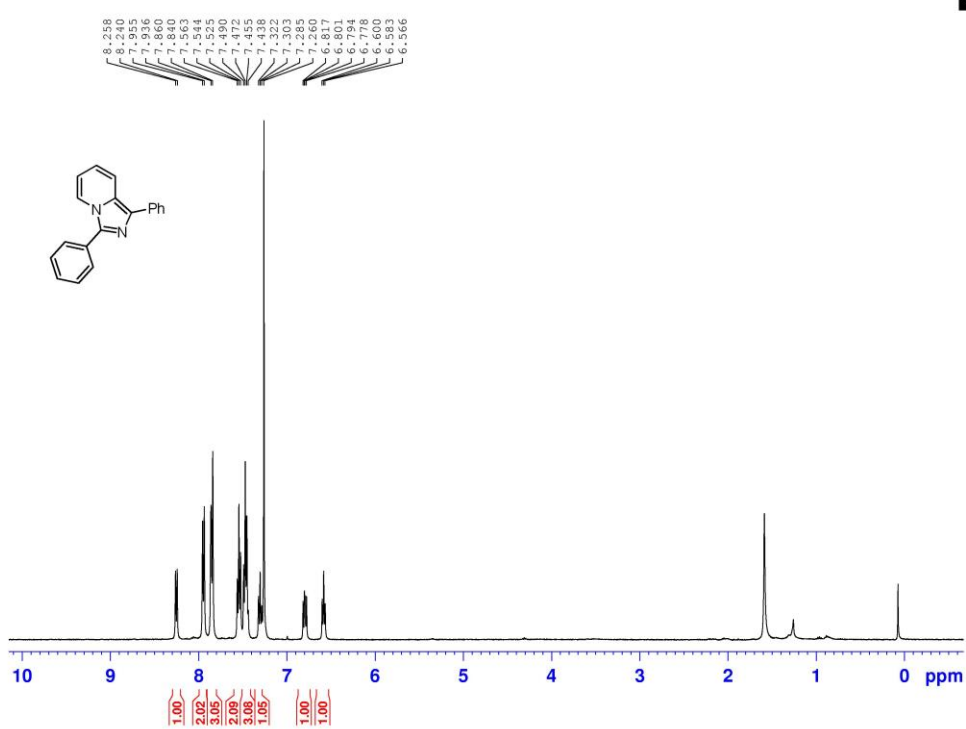
NUC1          13C
P1            13.84 usec
PL1           2.50 dB
PL1W         46.89624786 W
SFO1         125.7703643 MHz
  
```

----- CHANNEL f2 -----

```

CQDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           2.00 dB
PL12         17.40 dB
PL13         17.40 dB
PL2W         13.02359581 W
PL1W         0.42145536 W
PL1W         0.42145536 W
SFO2         500.1320005 MHz
SI            32768
SF           125.7577923 MHz
NEW          EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

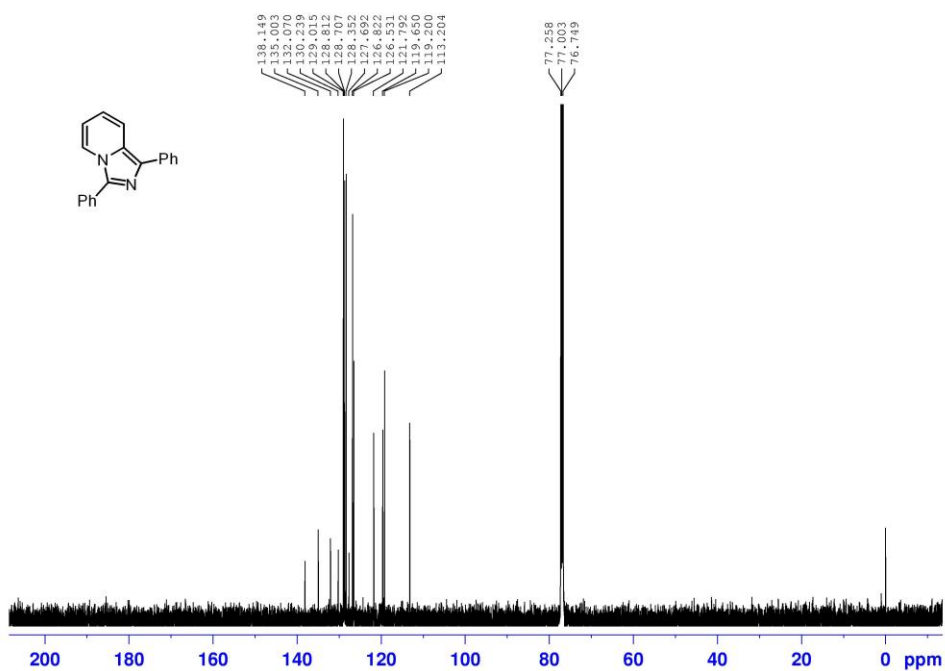
3b



```

NAME          0009-1H
EXPNO         1
PROCNO        1
Date_         20141212
Time          22.31
INSTRUM       spect
PROBHD        5 mm DUL 15C-1
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9594243 sec
RG            512
RC            512
DW            60.400 usec
DE            6.50 usec
TE            300.0 K
D1            1.0000000 sec
TD0
----- CHANNEL f1 -----
NUC1          1H
P1            12.58 usec
PL1           0.00 dB
PL1W          10.87646866 W
SFO1          400.1324710 MHz
SI            32768
SF            400.1300083 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

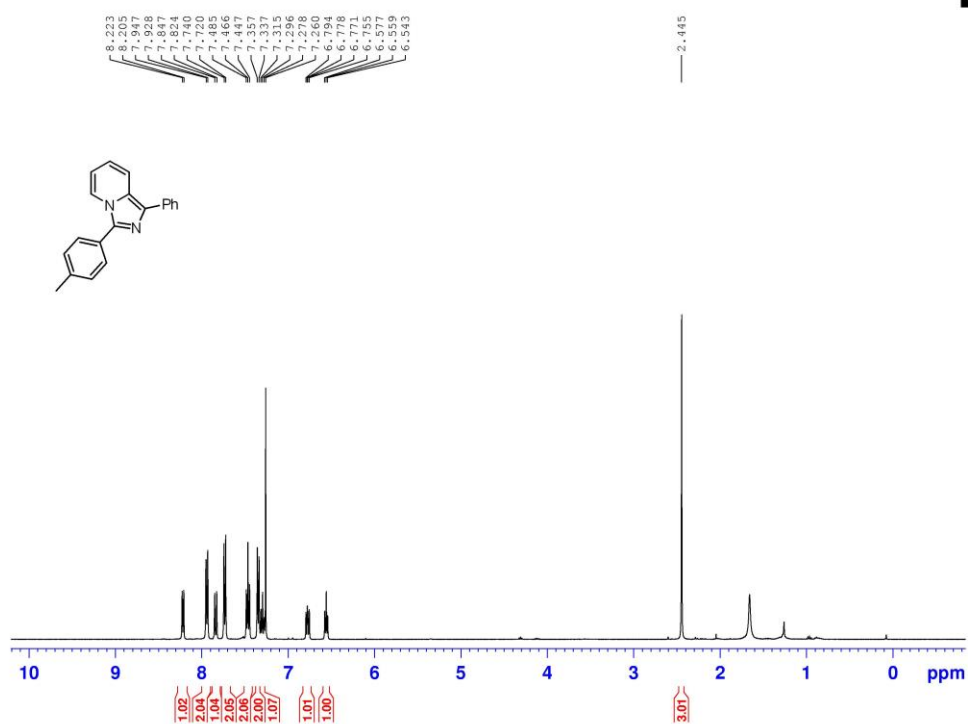
3b



```

NAME          3b
EXPNO         1
PROCNO        1
Date_         20151104
Time          12.29
INSTRUM       spect
PROBHD        5 mm PABBO 5B
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            2048
DS            4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010548 sec
RG            203
RC            203
DW            16.400 usec
DE            6.50 usec
TE            299.9 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0
----- CHANNEL f1 -----
NUC1          13C
P1            12.84 usec
PL1           2.50 dB
PL1W          46.89624786 W
SFO1          125.7703643 MHz
----- CHANNEL f2 -----
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           2.50 dB
PL12          17.40 dB
PL13          17.40 dB
PL1W          13.02359884 W
PL1W          0.42145536 W
SFO2          500.1360925 MHz
SFO3          0.42145536 MHz
SI            32768
SF            125.7577913 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

3c



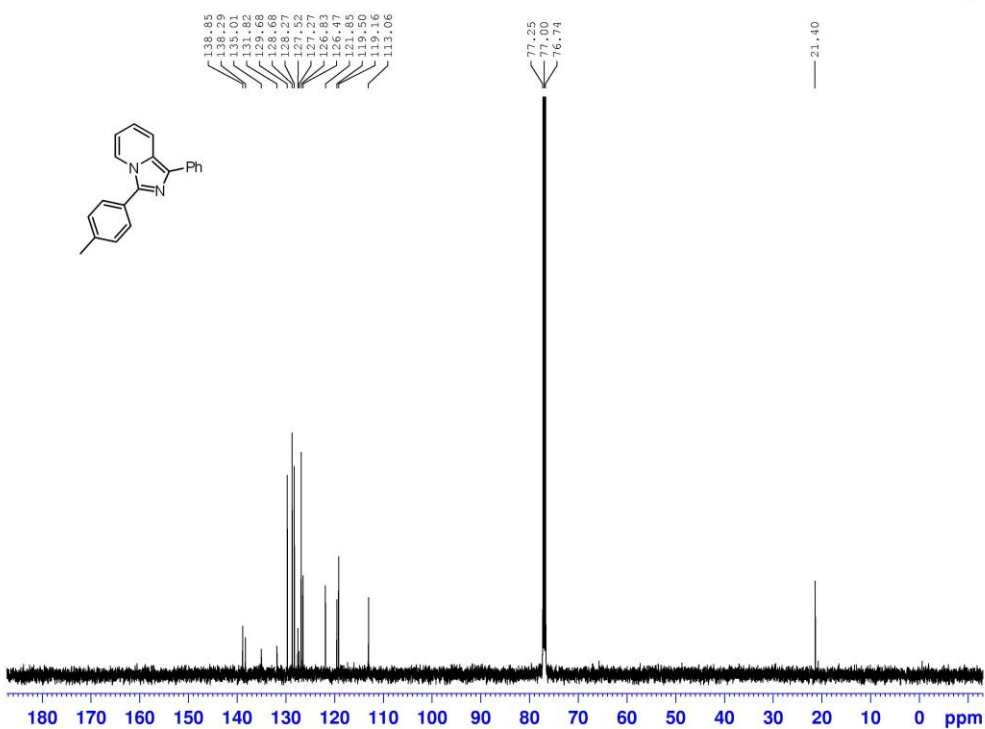
```

NAME          70196-4H
EXPNO         1
PROCNO        1
Date_         20141130
Time          23.06
INSTRUM       spect
PROBHD        5 mm DUL 15C-1
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            8
DS            2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9594243 sec
RG            322.3
RC            322.3
DW            60.400 usec
DE            6.50 usec
TE            299.3 K
D1            1.00000000 sec
TD0
  
```

```

----- CHANNEL f1 -----
NUC1          1H
P1            12.58 usec
PL1           0.00 dB
PL1W          10.87646866 W
SFO1          400.1324710 MHz
SI            32768
SF            400.1300082 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

70196-4



```

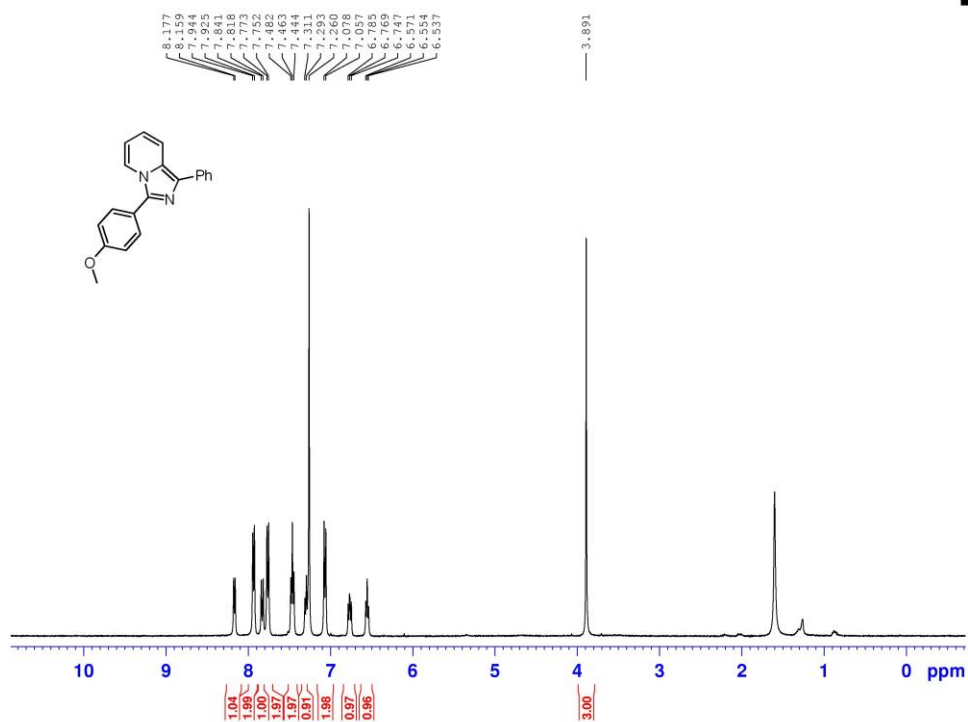
NAME          70196-4C
EXPNO         1
PROCNO        1
Date_         20141201
Time          23.59
INSTRUM       spect
PROBHD        5 mm PABBO 30C
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            4
DS            4
SWH           29761.994 Hz
FIDRES        0.454131 Hz
AQ            1.1010548 sec
RG            203
RC            16.800 usec
DE            6.50 usec
TE            301.3 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1
  
```

```

----- CHANNEL f1 -----
NUC1          13C
P1            13.84 usec
PL1           2.50 dB
PL1W          46.89624786 W
SFO1          125.7703643 MHz

----- CHANNEL f2 -----
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           2.50 dB
PL2W          37.40 dB
PL13          37.40 dB
PL2W          13.02339381 W
PL12W         0.42145536 W
PL1W          0.42145536 W
SFO2          500.1320005 MHz
SI            32768
SF            125.7577912 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

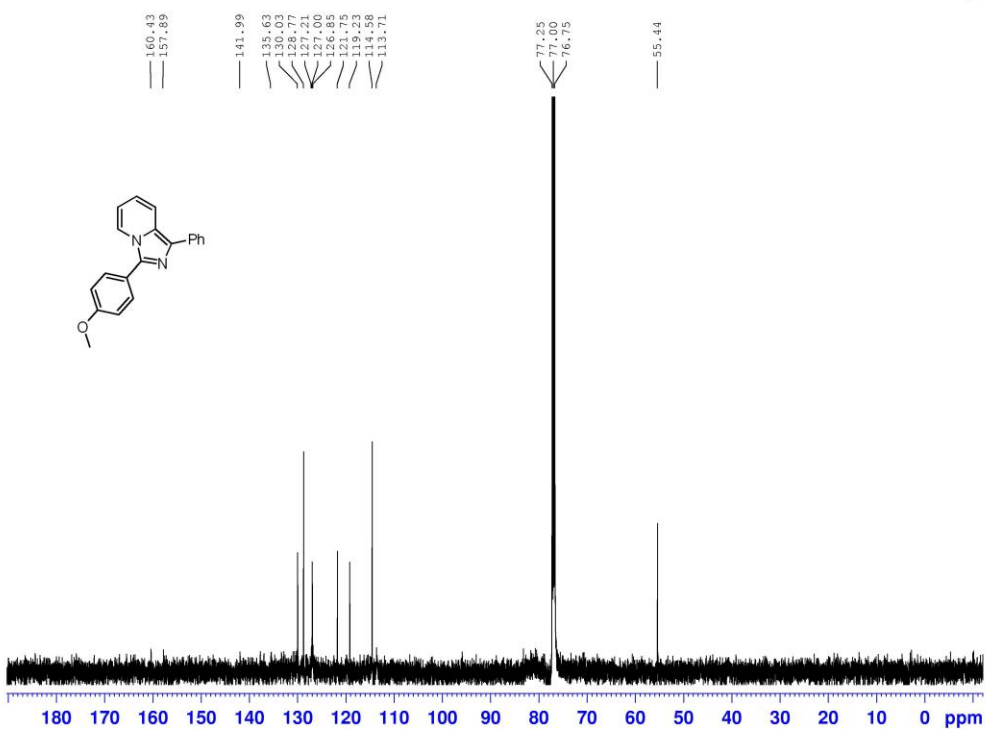
3d



```

NAME          70198-3H
EXPNO         1
PROCNO        1
Date_         20141202
Time          21.05
INSTRUM       spect
PROBHD        5 mm DUL 15c-1
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            8
DS            2
SWH           8278.146 Hz
FIDRES       0.126314 Hz
AQ           3.9594243 sec
RG           574.7
RC           574.7
DW           60.400 usec
DE           6.50 usec
TE           300.2 K
D1           1.00000000 sec
TD0
----- CHANNEL f1 -----
NUC1          1H
P1            12.58 usec
PL1           0.00 dB
PL1W         10.87646866 W
SFO1         400.1324710 MHz
SI            32768
SF           400.1300080 MHz
WDW           EM
SSB           0
LB           0.30 Hz
GB           0
PC           1.00
    
```

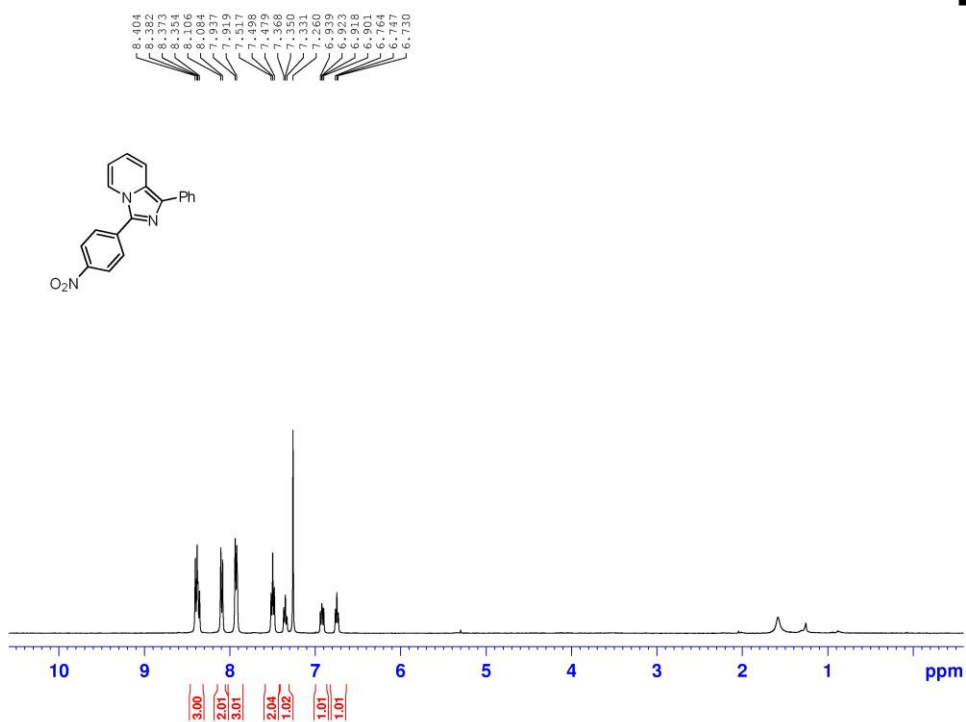
70198-3



```

NAME          70198-3C
EXPNO         1
PROCNO        1
Date_         20141214
Time          17.45
INSTRUM       spect
PROBHD        5 mm PABBO 80c
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            4
DS            4
SWH           29761.904 Hz
FIDRES       0.454131 Hz
AQ           1.1010548 sec
RG           203
RC           16.800 usec
DE           6.50 usec
TE           299.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0          1
----- CHANNEL f1 -----
NUC1          13C
P1            13.84 usec
PL1           2.50 dB
PL1W         46.89624786 W
SFO1         125.7703643 MHz
----- CHANNEL f2 -----
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           2.50 dB
PL12         17.40 dB
PL13         17.40 dB
PL1W         13.02339381 W
PL2W         0.42145536 W
PL1W         0.42145536 W
SFO2         500.1320005 MHz
SI            32768
SF           125.7577913 MHz
WDW           EM
SSB           0
LB           1.00 Hz
GB           0
PC           1.40
    
```


3e



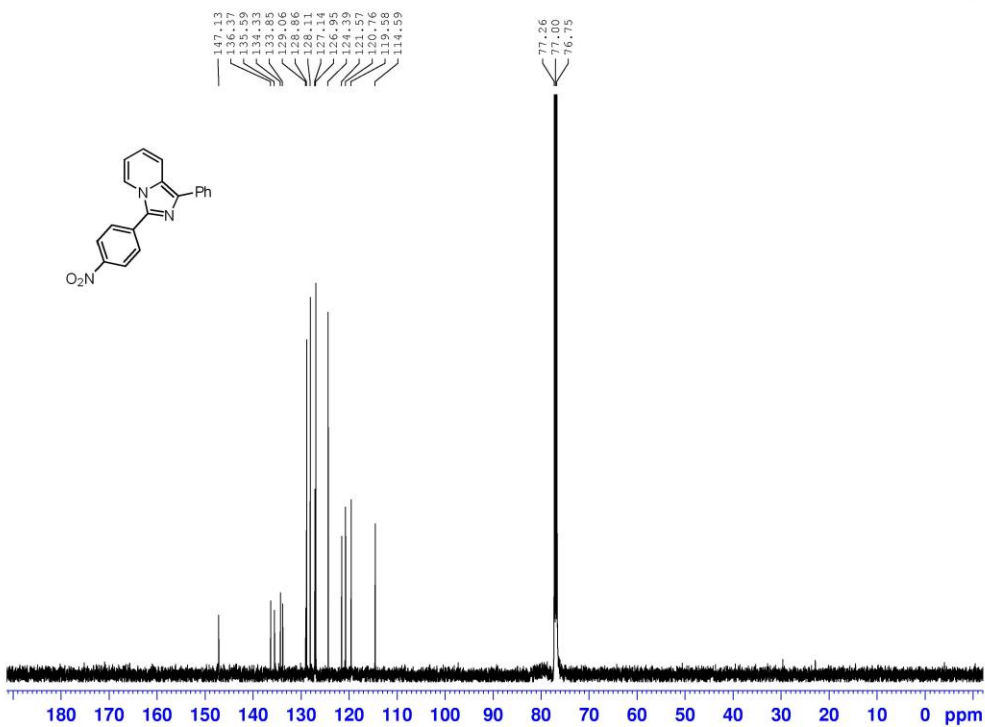
```

NAME          0010-1H
EXPNO         1
PROCNO        1
Date_         20141213
Time          16.09
INSTRUM       spect
PROBHD        5 mm DUL 15c-1
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9594243 sec
RG            456.1
RC            456.1
DW            60.400 usec
DE            6.50 usec
TE            298.0 K
D1            1.0000000 sec
TD0
  
```

```

----- CHANNEL f1 -----
NUC1          1H
P1            12.58 usec
PL1           0.00 dB
PL1W          10.87646866 W
SFO1          400.1324710 MHz
SI            32768
SF            400.1300080 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

0010-1C



```

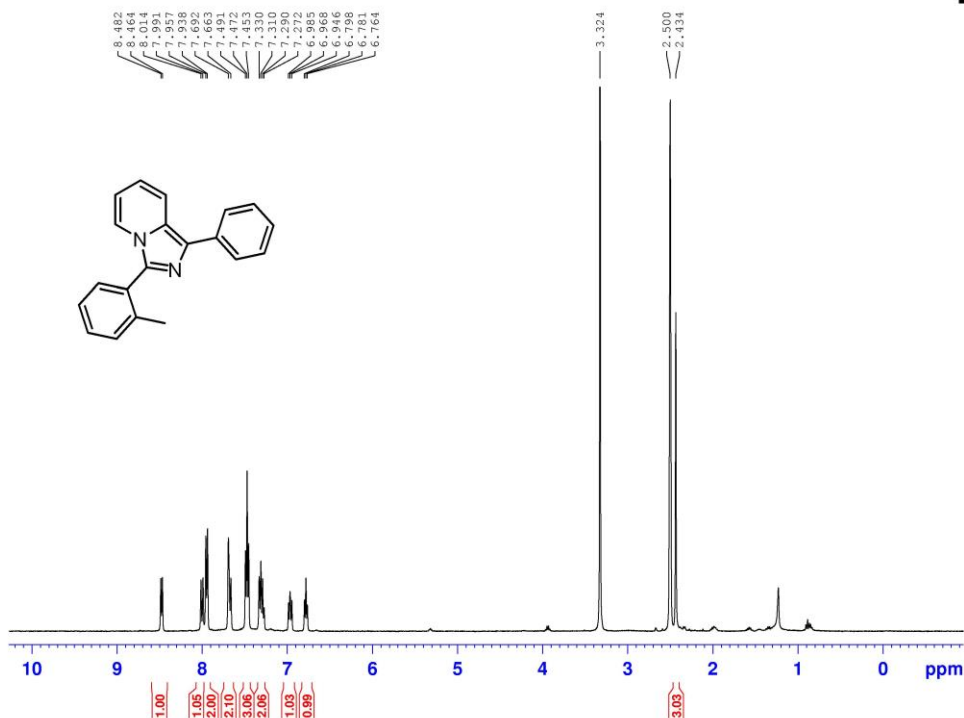
NAME          0010-1C
EXPNO         1
PROCNO        1
Date_         20141214
Time          21.29
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            3753
DS            4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010548 sec
RG            203
RC            16.890 usec
DE            6.50 usec
TE            299.5 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0           1
  
```

```

----- CHANNEL f1 -----
NUC1          13C
P1            13.84 usec
PL1           2.50 dB
PL1W          46.89624786 W
SFO1          125.7703643 MHz

----- CHANNEL f2 -----
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           2.00 dB
PL12          37.40 dB
PL13          37.40 dB
PL2W          13.02359581 W
PL1W          0.42143536 W
PL1LW        0.42143536 W
SFO2          500.1320095 MHz
SI            32768
SF            125.7577913 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

3f

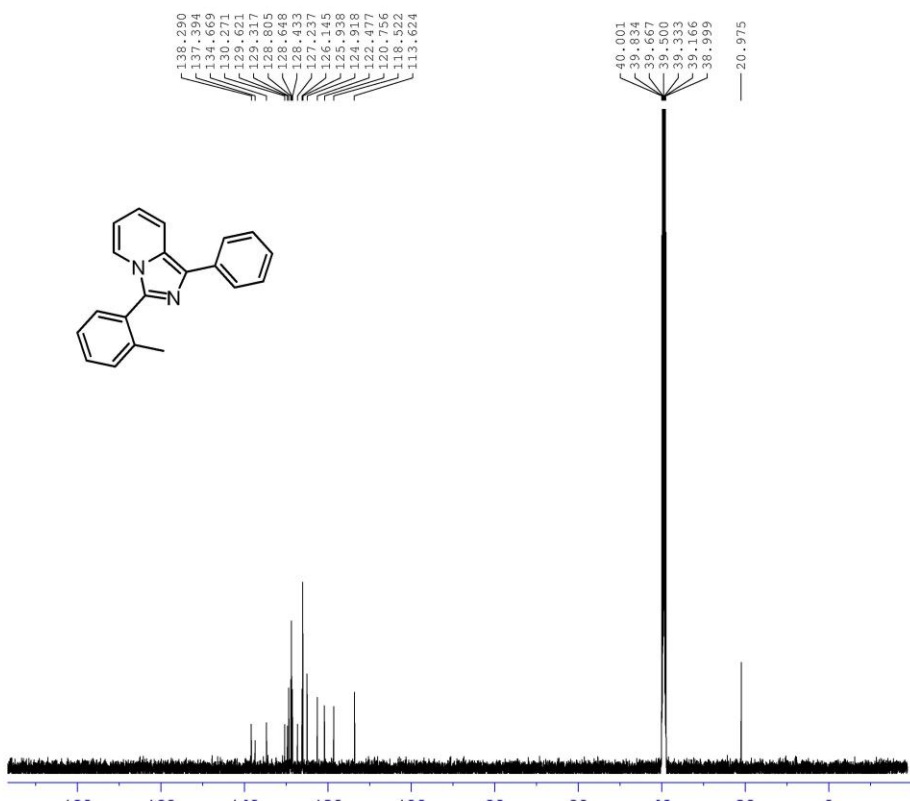


```

NAME      0019-1H
EXPNO    1
PROCNO   1
Date_    20150129
Time     2.54
INSTRUM  spect
PROBHD   5 mm DUL 13C-1
PULPROG  zg30
TD        65536
SOLVENT  DMSO
NS        16
DS        2
SWH      8278.146 Hz
FIDRES   0.126314 Hz
AQ        3.9584243 sec
RG        256
DM        60.400 usec
DE        6.50 usec
TE        297.3 K
D1        1.00000000 sec
TD0       1
  
```

```

----- CHANNEL f1 -----
NUC1      1H
P1        12.58 usec
P11       0.00 dB
PL1W      10.87646866 W
SFO1      400.1324710 MHz
SI        32768
SF        400.1300027 MHz
KW        EM
SSB       0
LB        0.50 Hz
GB        0
PC        1.00
  
```



```

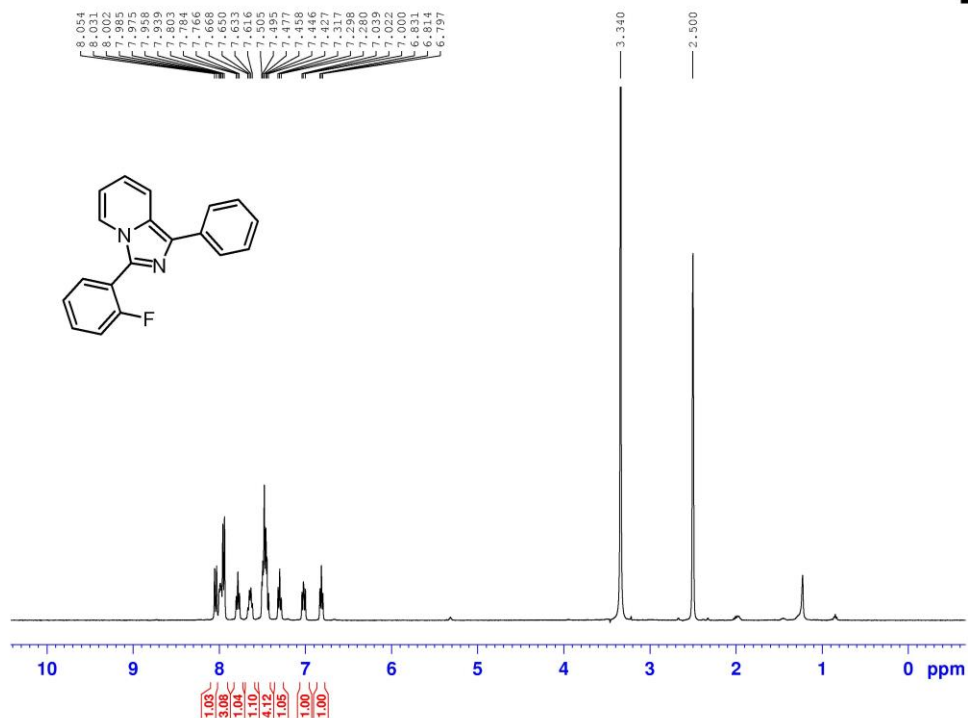
NAME      0019-C
EXPNO    1
PROCNO   1
Date_    20150130
Time     11.09
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD        65536
SOLVENT  DMSO
NS        1024
DS        4
SWH      29761.904 Hz
FIDRES   0.454131 Hz
AQ        1.1010548 sec
RG        203
DM        16.000 usec
DE        6.50 usec
TE        301.7 K
D1        2.00000000 sec
D11      0.03000000 sec
TD0       1
  
```

```

----- CHANNEL f1 -----
NUC1      13C
P1        13.84 usec
P11       2.50 dB
PL1W      46.89624786 W
SFO1      125.7703643 MHz

----- CHANNEL f2 -----
NAME      Waltz16
NUC2      1H
PCPD2    80.00 usec
P12      2.50 dB
P112     17.40 dB
P113     17.40 dB
P12W     13.02359501 W
P132W    0.42143536 W
P133W    0.42143536 W
SFO2     500.1320005 MHz
SF2      32768
SI        32768
SF        125.7578578 MHz
KW        EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
  
```

3g



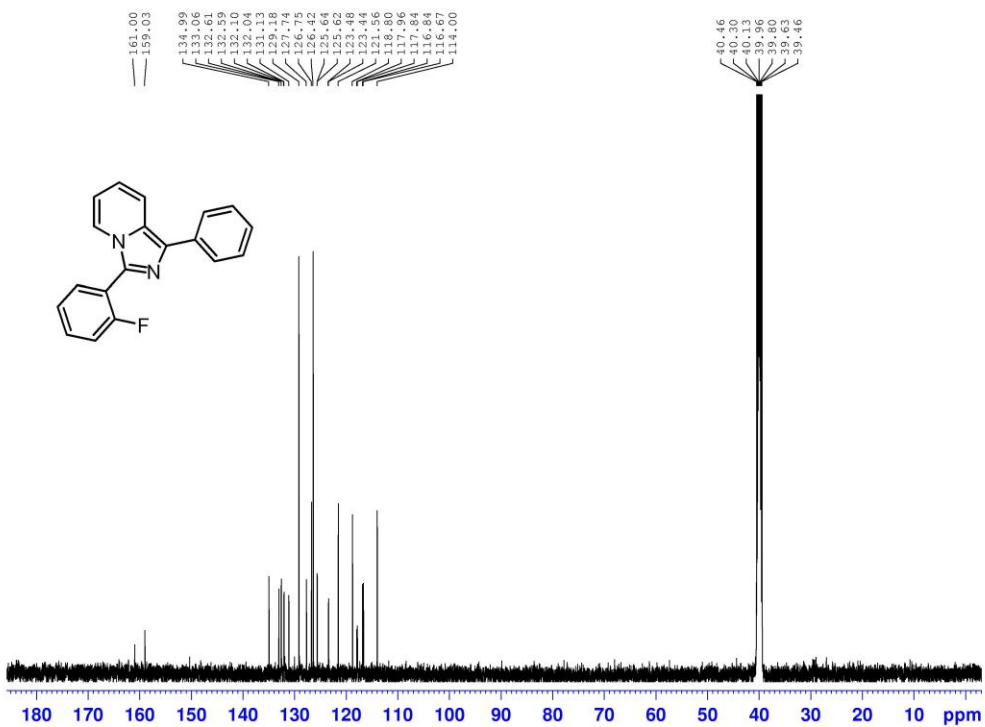
```

NAME          0023-H
EXPNO         1
PROCNO        1
Date_         20150127
Time          9.27
INSTRUM       spect
PROBHD        5 mm DUL 13C-1
PULPROG       zg30
TD            65536
SOLVENT       DMSO
NS            16
DS            2
SWS          8278.144 Hz
FIDRES       0.126314 Hz
AQ           3.9584243 sec
RG           256
RW           60.400 usec
DE           6.50 usec
TE           298.0 K
D1           1.00000000 sec
TD0          1
  
```

```

----- CHANNEL f1 -----
NUC1          1H
P1            12.00 usec
PL1           0.00 dB
PL1W         10.87646866 W
SFO1         400.1324710 MHz
SI            32768
SF           400.1300028 MHz
WCM          0
SSB           0
LB           0.30 Hz
GB            0
PC            1.00
  
```

0023



```

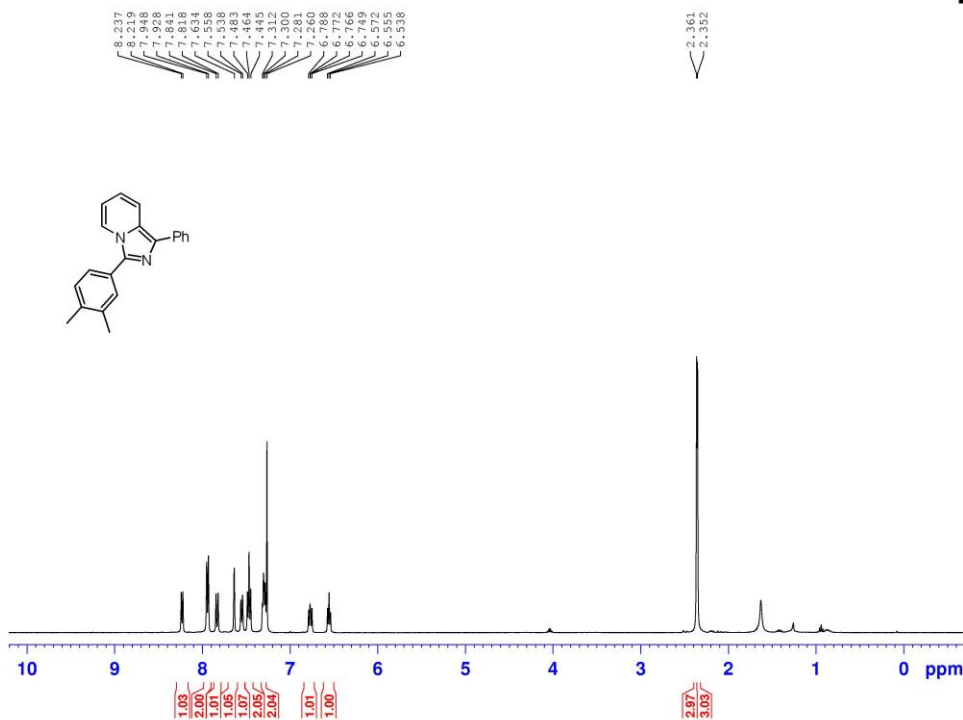
NAME          0023-C
EXPNO         1
PROCNO        1
Date_         20150127
Time          19.24
INSTRUM       spect
PROBHD        5 mm PABBO BB
PULPROG       zgpg30
TD            65536
SOLVENT       DMSO
NS            2048
DS            4
SWS          29761.904 Hz
FIDRES       0.454151 Hz
AQ           1.1101545 sec
RG           203
RW           16.800 usec
DE           6.50 usec
TE           298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0          1
  
```

```

----- CHANNEL f1 -----
NUC1          13C
P1            13.84 usec
PL1           2.15 dB
PL1W         46.89624786 W
SFO1         125.7703643 MHz

----- CHANNEL f2 -----
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           2.50 dB
PL12         37.40 dB
PL13         37.40 dB
PL2W         13.02399581 W
PL1W         0.42145336 W
PL13W        0.42145336 W
SFO2         500.1320005 MHz
SI            32768
SF           125.7577966 MHz
WCM          0
SSB           0
LB           1.00 Hz
GB            0
PC            1.40
  
```

3h



```

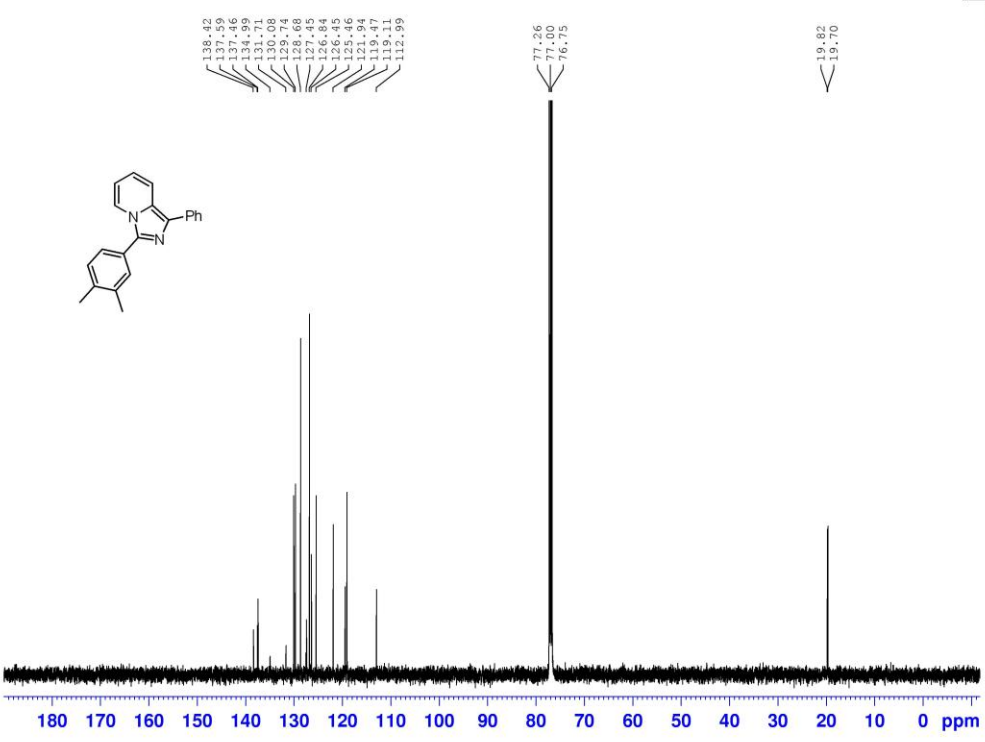
NAME          0019-2H
EXPNO         1
PROCNO        1
Date_         20141219
Time          14.00
INSTRUM       spect
PROBHD        5 mm DUL 13C-1
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8278.144 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG            327.5
RW           60.400 usec
DE            6.50 usec
TE            297.2 K
D1            1.00000000 sec
TD0           1
  
```

----- CHANNEL f1 -----

```

NUC1          1H
P1            12.00 usec
P11           0.00 dB
PL1W         10.87646886 W
SFO1         400.1324710 MHz
SI            32768
SF           400.1300093 MHz
WCM          0
SSB           0
LB            0.50 Hz
GB            0
PC            1.00
  
```

0019-2



```

NAME          0019-2C
EXPNO         1
PROCNO        1
Date_         20141221
Time          16.02
INSTRUM       spect
PROBHD        5 mm PABBO-BB
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            1081
DS            4
SWH           29761.904 Hz
FIDRES        0.454151 Hz
AQ            1.11010345 sec
RG            203
RW           16.800 usec
DE            6.50 usec
TE            297.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1
  
```

----- CHANNEL f1 -----

```

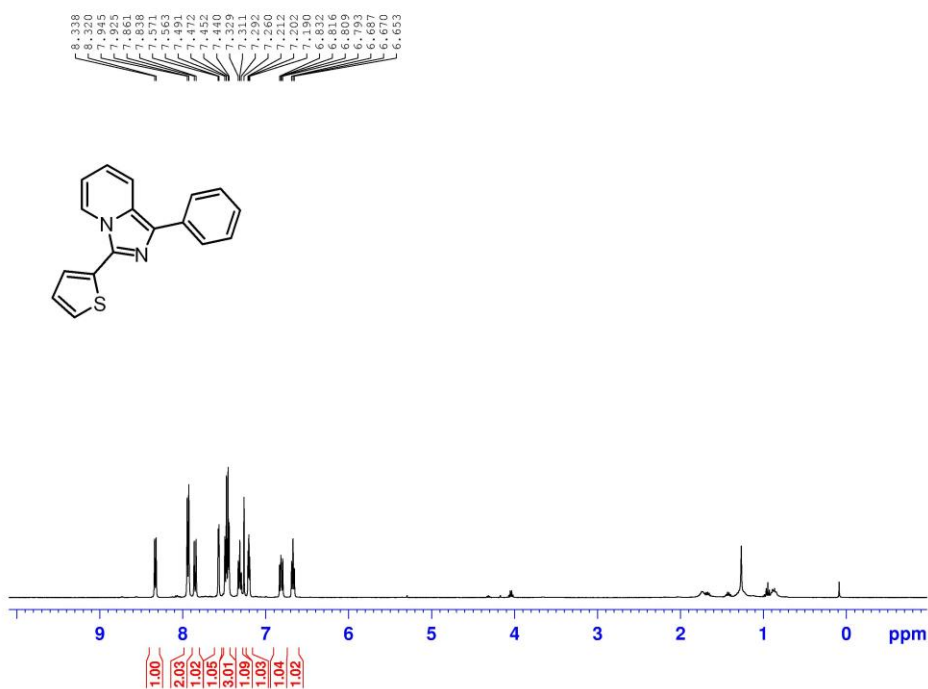
NUC1          13C
P1            13.84 usec
P11           2.15 dB
PL1W         46.89624786 W
SFO1         125.7703643 MHz
  
```

----- CHANNEL f2 -----

```

CPDPRG2       waltz16
NUC2          1H
PCPD02        80.00 usec
P12           2.50 dB
PL12         37.40 dB
P113         37.40 dB
PL13         37.40 dB
P12W         13.0239581 W
SFO2         0.42145536 MHz
P113W        0.42145536 W
SFO2         500.1320003 MHz
SI            32768
SF           125.7577933 MHz
WCM          0
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

0010-2



```

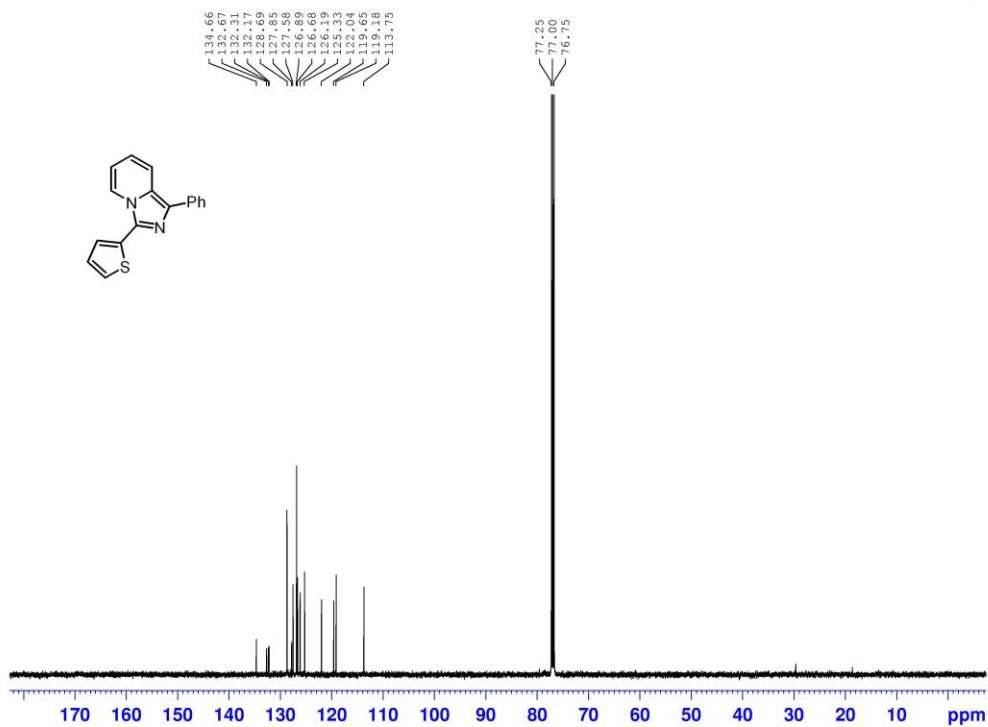
NAME          0010-2H
EXPNO         1
PROCNO        1
Date_         20141212
Time          22.44
INSTRUM       spect
PROBHD        5 mm DUL 13C-1
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWS           3278.144 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG            181
DM            60.400 usec
DE            6.50 usec
TE            299.5 K
D1            1.00000000 sec
TD0
  
```

----- CHANNEL f1 -----

```

NUC1          1H
P1            12.00 usec
PL1           0.00 dB
PL1W          10.87646866 W
SFO1          400.1324710 MHz
SI            32768
SF            400.13000000 MHz
WDW           EM
SSB            0
LB            0.30 Hz
GB            0
PC            1.00
  
```

0010-2



```

NAME          0010-2C
EXPNO         1
PROCNO        1
Date_         20141216
Time          6.07
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            4
DS            4
SWS           29761.904 Hz
FIDRES        0.454151 Hz
AQ            1.11010345 sec
RG            203
DM            16.000 usec
DE            6.50 usec
TE            300.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0
  
```

----- CHANNEL f1 -----

```

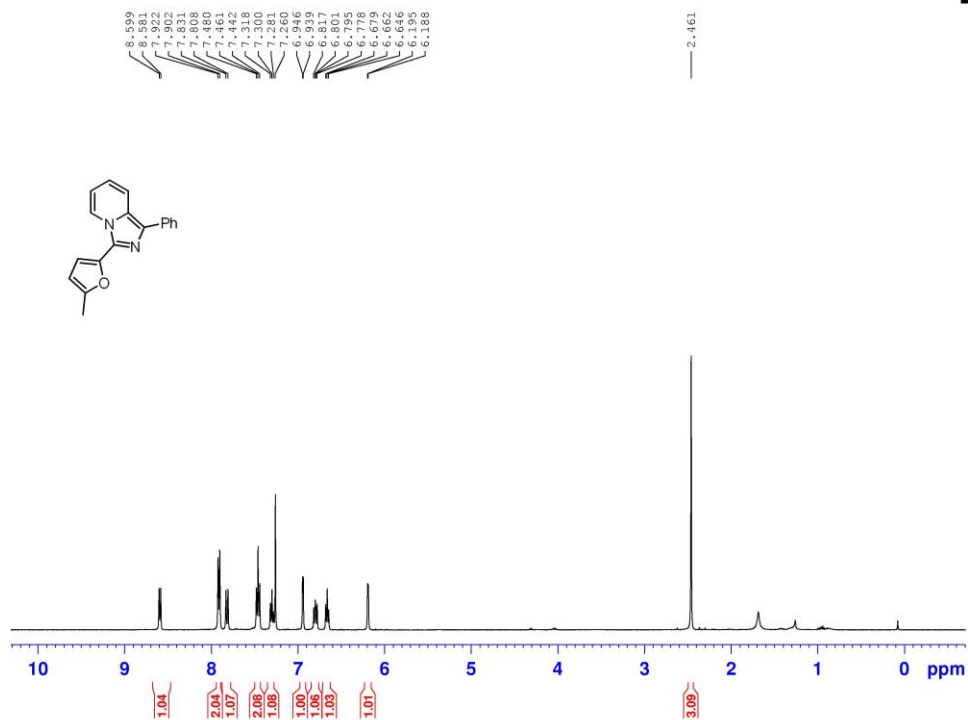
NUC1          13C
P1            13.84 usec
PL1           2.15 dB
PL1W          46.89624786 W
SFO1          125.7703643 MHz
  
```

----- CHANNEL f2 -----

```

CPDPRG2      waltz16
NUC2          1H
PCPD02       80.00 usec
PL2           2.50 dB
PL12         37.40 dB
PL13         37.40 dB
PL2W         13.0239581 W
PL1W         0.42145336 W
PL13W        0.42145336 W
SFO2         500.13200000 MHz
SI            32768
SF            125.7577950 MHz
WDW           EM
SSB            0
LB            1.00 Hz
GB            0
PC            1.40
  
```

3j



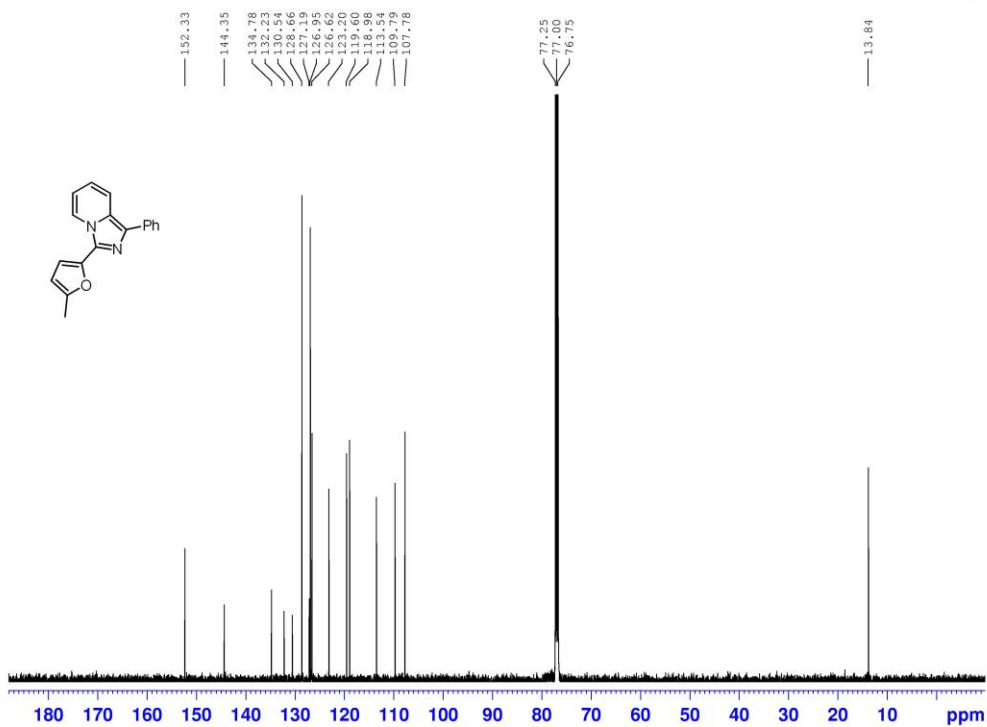
```

NAME          0016-2H
EXPNO         1
PROCNO        1
Date_         20141216
Time          23.48
INSTRUM       spect
PROBHD        5 mm DUL 13C-1
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            8
DS            2
SWS           8278.144 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG            362
RGW           60.400 usec
DE            6.50 usec
TE            298.2 K
D1            1.0000000 sec
TD0           1
  
```

```

----- CHANNEL f1 -----
NUC1          1H
P1            12.00 usec
P11           0.00 dB
PL1W         10.87646886 W
SFO1         400.1324710 MHz
SI            32768
SF           400.1300082 MHz
WCMW         0
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

0016-2



```

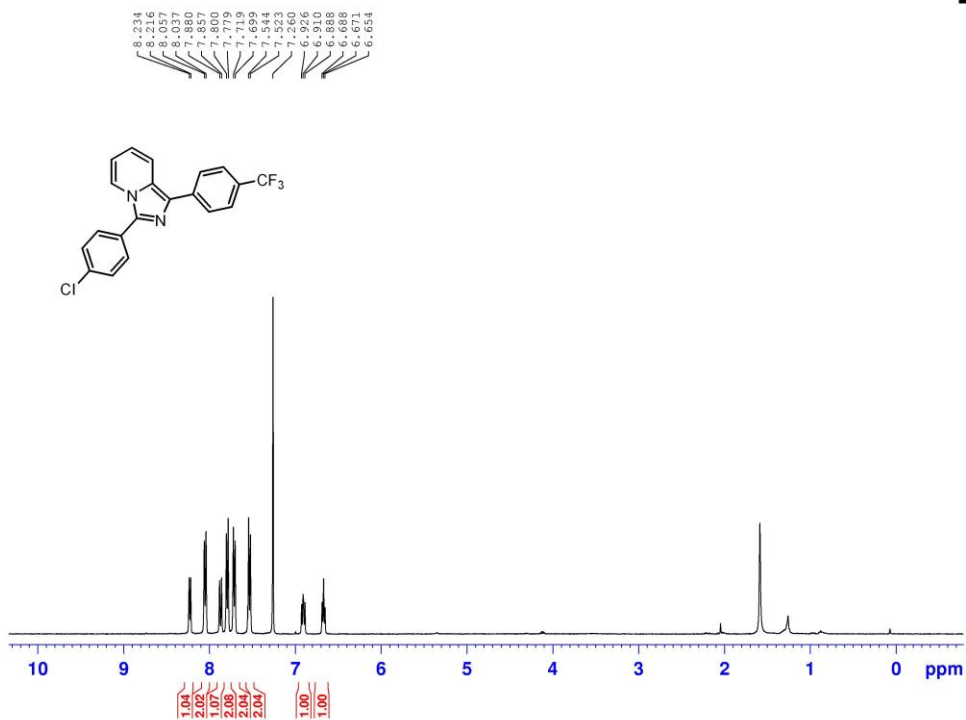
NAME          0016-2C
EXPNO         1
PROCNO        1
Date_         20141217
Time          15.49
INSTRUM       spect
PROBHD        5 mm PABBO-BB
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            8
DS            4
SWS           29761.904 Hz
FIDRES        0.454151 Hz
AQ            1.1010346 sec
RG            203
RGW           16.800 usec
DE            6.50 usec
TE            299.5 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0           1
  
```

```

----- CHANNEL f1 -----
NUC1          13C
P1            13.84 usec
P11           2.15 dB
PL1W         46.89624786 W
SFO1         125.7703643 MHz

----- CHANNEL f2 -----
CPDPRG2       waltz16
NUC2          1H
PCPD02        80.00 usec
P12           2.50 dB
PL12         37.40 dB
P113          37.40 dB
PL13         13.0239581 W
PL1W         0.42145336 W
SFO2         500.1320005 MHz
SI            32768
SF           125.7577922 MHz
WCMW         0
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

3k



```

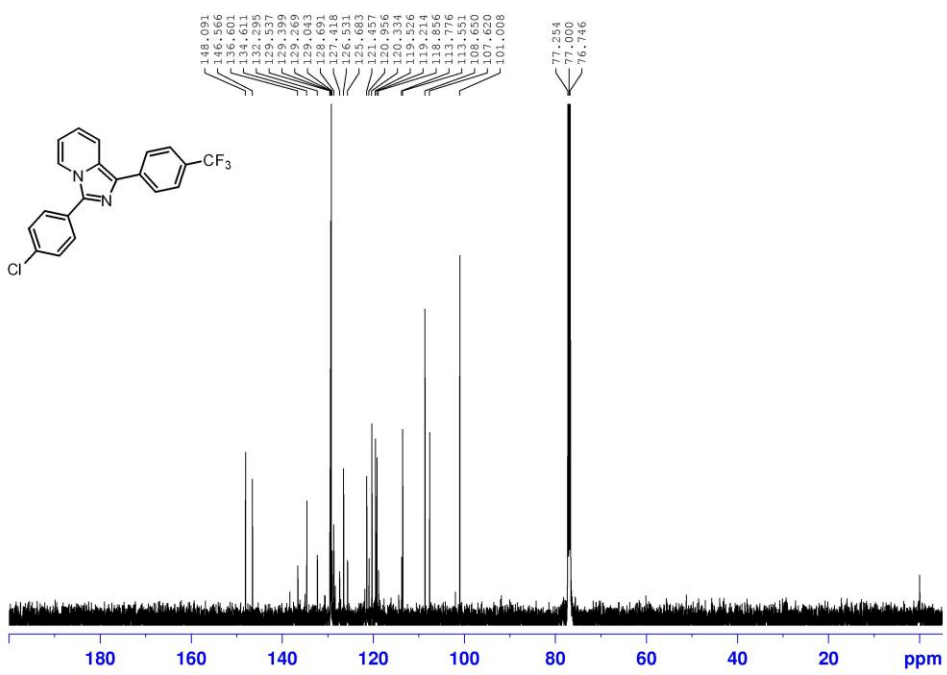
NAME      0029-1h
EXPNO    1
PROCNO   1
Date_    20141231
Time     4:13
INSTRUM spect
PROBHD   5 mm DUL 13C-1
PULPROG zgpg30
TD        65536
SOLVENT  CDCl3
NS        16
DS        2
SWH      8278.144 Hz
FIDRES   0.126314 Hz
AQ        3.9584243 sec
RG        362
RW        60.400 usec
DE        6.50 usec
TE        296.3 K
D1        1.00000000 sec
TD0       1
  
```

----- CHANNEL f1 -----

```

NUC1      1H
P1        12.55 usec
PL1       0.00 dB
PL1W     10.87646886 W
SFO1     400.1324710 MHz
SI        32768
SF        400.1300095 MHz
RGW       2 M
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
  
```

3k'



```

NAME      3k'
EXPNO    1
PROCNO   1
Date_    20151104
Time     20:40
INSTRUM spect
PROBHD   5 mm PABBO BB-
PULPROG zgpg30
TD        65536
SOLVENT  CDCl3
NS        3200
DS        4
SWH      29761.904 Hz
FIDRES   0.454131 Hz
AQ        1.1010548 sec
RG        293
DW        16.800 usec
DE        6.50 usec
TE        300.2 K
D1        2.00000000 sec
D11      0.03000000 sec
TD0       1
  
```

----- CHANNEL f1 -----

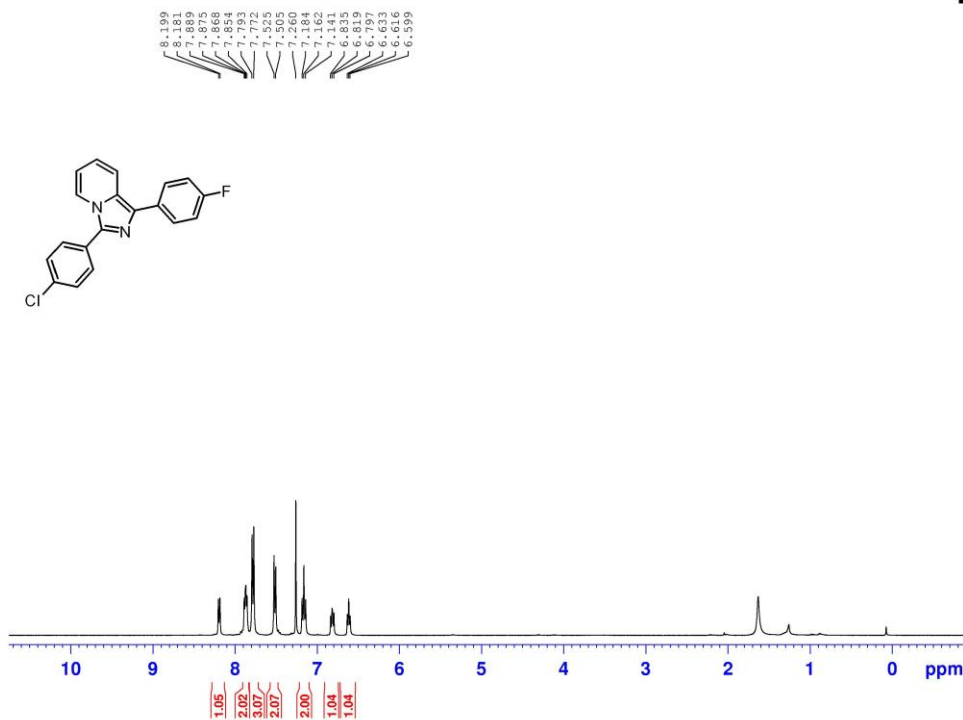
```

NUC1      13C
P1        13.84 usec
PL1       2.50 dB
PL1W     46.89624786 W
SFO1     125.7703643 MHz
  
```

----- CHANNEL f2 -----

```

CPDPRG2  waltz16
NUC2      1H
PCPD2    80.00 usec
PL2      2.50 dB
PL12    17.40 dB
PL13    17.40 dB
PL14    17.40 dB
PL15    17.40 dB
PL16    17.40 dB
PL17    17.40 dB
PL18    17.40 dB
PL19    17.40 dB
PL20    17.40 dB
PL21    17.40 dB
PL22    17.40 dB
PL23    17.40 dB
PL24    17.40 dB
PL25    17.40 dB
PL26    17.40 dB
PL27    17.40 dB
PL28    17.40 dB
PL29    17.40 dB
PL30    17.40 dB
PL31    17.40 dB
PL32    17.40 dB
PL33    17.40 dB
PL34    17.40 dB
PL35    17.40 dB
PL36    17.40 dB
PL37    17.40 dB
PL38    17.40 dB
PL39    17.40 dB
PL40    17.40 dB
PL41    17.40 dB
PL42    17.40 dB
PL43    17.40 dB
PL44    17.40 dB
PL45    17.40 dB
PL46    17.40 dB
PL47    17.40 dB
PL48    17.40 dB
PL49    17.40 dB
PL50    17.40 dB
PL51    17.40 dB
PL52    17.40 dB
PL53    17.40 dB
PL54    17.40 dB
PL55    17.40 dB
PL56    17.40 dB
PL57    17.40 dB
PL58    17.40 dB
PL59    17.40 dB
PL60    17.40 dB
PL61    17.40 dB
PL62    17.40 dB
PL63    17.40 dB
PL64    17.40 dB
PL65    17.40 dB
PL66    17.40 dB
PL67    17.40 dB
PL68    17.40 dB
PL69    17.40 dB
PL70    17.40 dB
PL71    17.40 dB
PL72    17.40 dB
PL73    17.40 dB
PL74    17.40 dB
PL75    17.40 dB
PL76    17.40 dB
PL77    17.40 dB
PL78    17.40 dB
PL79    17.40 dB
PL80    17.40 dB
PL81    17.40 dB
PL82    17.40 dB
PL83    17.40 dB
PL84    17.40 dB
PL85    17.40 dB
PL86    17.40 dB
PL87    17.40 dB
PL88    17.40 dB
PL89    17.40 dB
PL90    17.40 dB
PL91    17.40 dB
PL92    17.40 dB
PL93    17.40 dB
PL94    17.40 dB
PL95    17.40 dB
PL96    17.40 dB
PL97    17.40 dB
PL98    17.40 dB
PL99    17.40 dB
PL100   17.40 dB
  
```



```

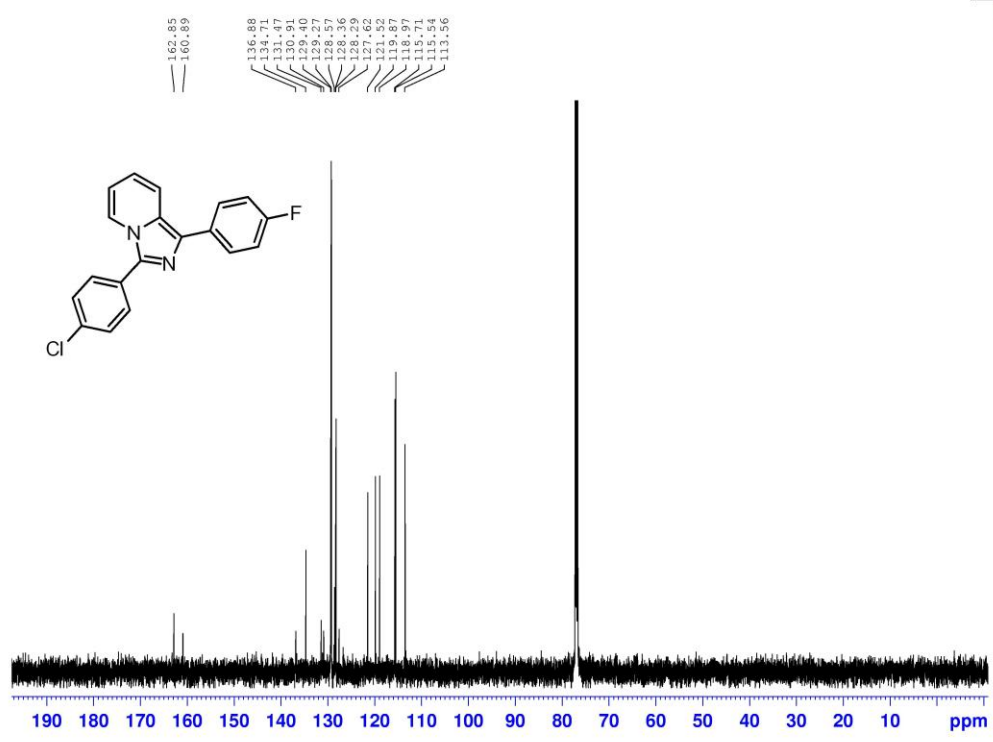
NAME          0024-2H
EXPNO         1
PROCNO        1
Date_         20141224
Time          12.31
INSTRUM       spect
PROBHD        5 mm DUL 13C-1
PULPROG       zgpg
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWS           8278.144 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG            256
RW           60.400 usec
DE            6.50 usec
TE            297.3 K
D1            1.00000000 sec
TD0           1
    
```

----- CHANNEL f1 -----

```

NUC1          1H
P1            12.00 usec
P11           0.00 dB
PL1W         10.87646886 W
SFO1         400.1324710 MHz
SI            32768
SF           400.1300095 MHz
WCM          0
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

0024-2



```

NAME          0024-2C
EXPNO         1
PROCNO        1
Date_         20141225
Time          6.33
INSTRUM       spect
PROBHD        5 mm PABBO-BB
PULPROG       zgpg
TD            65536
SOLVENT       CDCl3
NS            835
DS            4
SWS           29761.904 Hz
FIDRES        0.454151 Hz
AQ            1.1010546 sec
RG            203
RW           16.800 usec
DE            6.50 usec
TE            300.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1
    
```

----- CHANNEL f1 -----

```

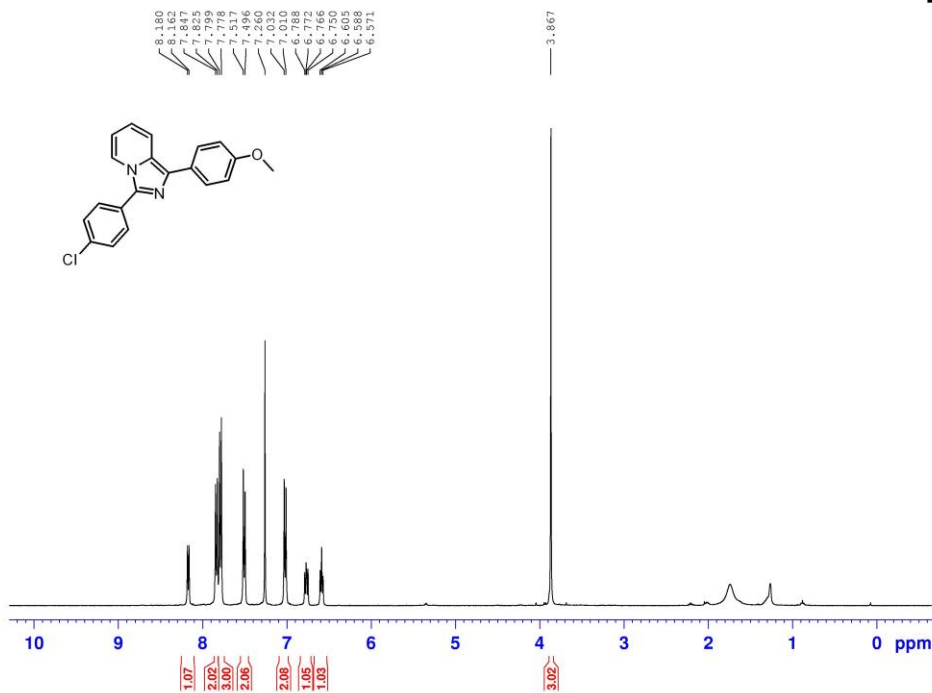
NUC1          13C
P1            13.84 usec
P11           2.10 dB
PL1W         46.89624786 W
SFO1         125.7703643 MHz
    
```

----- CHANNEL f2 -----

```

CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
P12           2.50 dB
PL12         37.40 dB
P13           37.40 dB
PL13         37.40 dB
PL2W         13.0239581 W
SFO2         0.42145336 MHz
PL13W        0.42145336 W
SFO2         500.1320005 MHz
SI            32768
SF           125.7577966 MHz
WCM          0
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```


3m

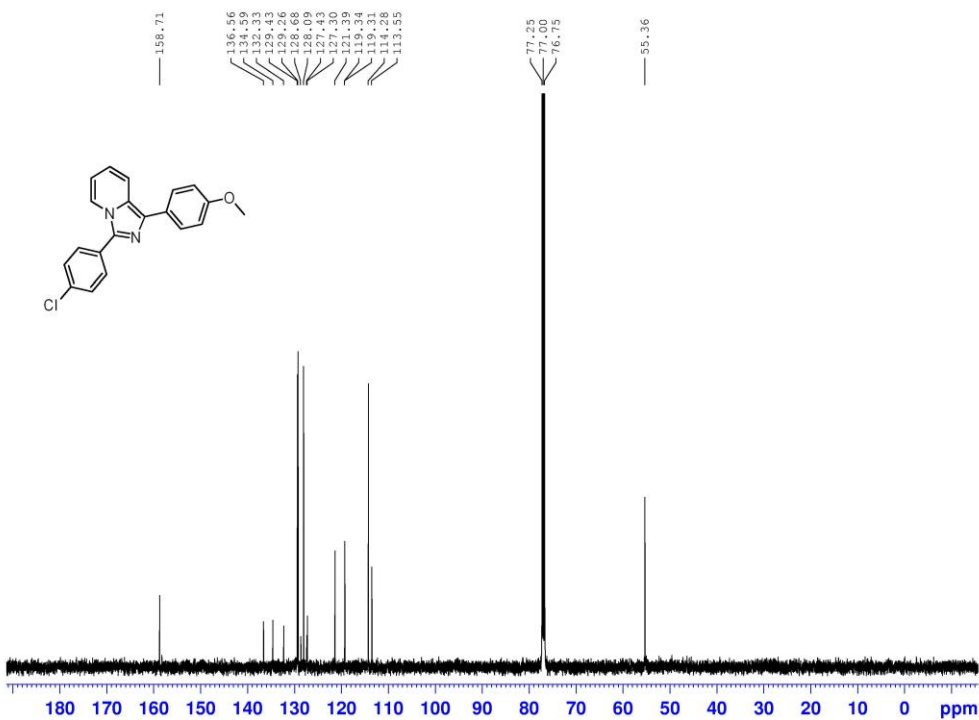


```

NAME      0024-1'H
EXPNO     1
PROCNO    1
Date_     20141225
Time      14.46
INSTRUM   spect
PROBHD    5 mm DUL 13C-1
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         2
DS         4
SWH        8278.146 Hz
FIDRES     0.126314 Hz
AQ         3.9584243 sec
RG         256
WDW        60.400 usec
DE         6.50 usec
TE         299.1 K
D1         1.00000000 sec
D11        1
D12        1
D13        1
D14        1
D15        1
D16        1
D17        1
D18        1
D19        1
D20        1
D21        1
D22        1
D23        1
D24        1
D25        1
D26        1
D27        1
D28        1
D29        1
D30        1
D31        1
D32        1
D33        1
D34        1
D35        1
D36        1
D37        1
D38        1
D39        1
D40        1
D41        1
D42        1
D43        1
D44        1
D45        1
D46        1
D47        1
D48        1
D49        1
D50        1
D51        1
D52        1
D53        1
D54        1
D55        1
D56        1
D57        1
D58        1
D59        1
D60        1
D61        1
D62        1
D63        1
D64        1
D65        1
D66        1
D67        1
D68        1
D69        1
D70        1
D71        1
D72        1
D73        1
D74        1
D75        1
D76        1
D77        1
D78        1
D79        1
D80        1
D81        1
D82        1
D83        1
D84        1
D85        1
D86        1
D87        1
D88        1
D89        1
D90        1
D91        1
D92        1
D93        1
D94        1
D95        1
D96        1
D97        1
D98        1
D99        1
D100       1
===== CHANNEL f1 =====
NUC1       1H
P1         12.58 usec
PL1        0.00 dB
PL12       10.8764686 W
SFO1       400.1324719 MHz
SI         32768
SF         400.1300029 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
CB         0
PC         1.00

```

0024-1

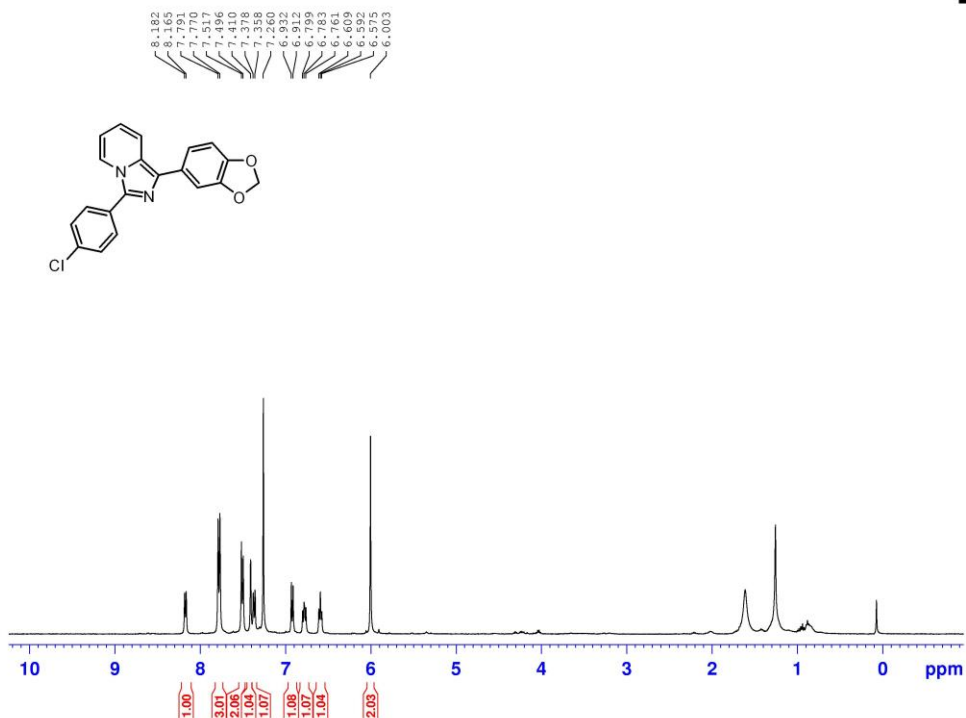


```

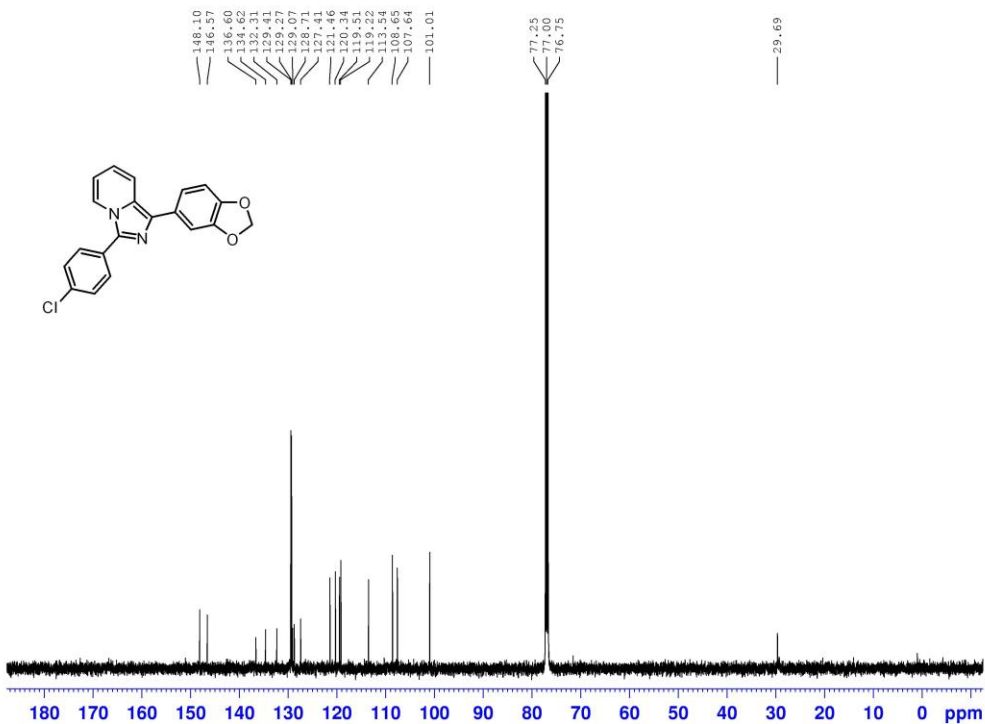
NAME      0024-1C
EXPNO     1
PROCNO    1
Date_     20141225
Time      7.23
INSTRUM   spect
PROBHD    5 mm DABBO 13C
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         4
DS         4
SWH        29761.994 Hz
FIDRES     0.454331 Hz
AQ         1.1010548 sec
RG         256
WDW        16.800 usec
DE         6.50 usec
TE         300.8 K
D1         2.00000000 sec
D11        0.93000000 sec
D12        1
D13        1
D14        1
D15        1
D16        1
D17        1
D18        1
D19        1
D20        1
D21        1
D22        1
D23        1
D24        1
D25        1
D26        1
D27        1
D28        1
D29        1
D30        1
D31        1
D32        1
D33        1
D34        1
D35        1
D36        1
D37        1
D38        1
D39        1
D40        1
D41        1
D42        1
D43        1
D44        1
D45        1
D46        1
D47        1
D48        1
D49        1
D50        1
D51        1
D52        1
D53        1
D54        1
D55        1
D56        1
D57        1
D58        1
D59        1
D60        1
D61        1
D62        1
D63        1
D64        1
D65        1
D66        1
D67        1
D68        1
D69        1
D70        1
D71        1
D72        1
D73        1
D74        1
D75        1
D76        1
D77        1
D78        1
D79        1
D80        1
D81        1
D82        1
D83        1
D84        1
D85        1
D86        1
D87        1
D88        1
D89        1
D90        1
D91        1
D92        1
D93        1
D94        1
D95        1
D96        1
D97        1
D98        1
D99        1
D100       1
===== CHANNEL f1 =====
NUC1       13C
P1         13.84 usec
PL1        2.50 dB
PL12       46.89624786 W
SFO1       125.7703643 MHz
===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2     80.00 usec
P12       2.50 dB
PL12     17.40 dB
PL13     17.40 dB
PL14     17.40 dB
PL15     17.40 dB
PL16     17.40 dB
PL17     17.40 dB
PL18     17.40 dB
PL19     17.40 dB
PL20     17.40 dB
PL21     17.40 dB
PL22     17.40 dB
PL23     17.40 dB
PL24     17.40 dB
PL25     17.40 dB
PL26     17.40 dB
PL27     17.40 dB
PL28     17.40 dB
PL29     17.40 dB
PL30     17.40 dB
PL31     17.40 dB
PL32     17.40 dB
PL33     17.40 dB
PL34     17.40 dB
PL35     17.40 dB
PL36     17.40 dB
PL37     17.40 dB
PL38     17.40 dB
PL39     17.40 dB
PL40     17.40 dB
PL41     17.40 dB
PL42     17.40 dB
PL43     17.40 dB
PL44     17.40 dB
PL45     17.40 dB
PL46     17.40 dB
PL47     17.40 dB
PL48     17.40 dB
PL49     17.40 dB
PL50     17.40 dB
PL51     17.40 dB
PL52     17.40 dB
PL53     17.40 dB
PL54     17.40 dB
PL55     17.40 dB
PL56     17.40 dB
PL57     17.40 dB
PL58     17.40 dB
PL59     17.40 dB
PL60     17.40 dB
PL61     17.40 dB
PL62     17.40 dB
PL63     17.40 dB
PL64     17.40 dB
PL65     17.40 dB
PL66     17.40 dB
PL67     17.40 dB
PL68     17.40 dB
PL69     17.40 dB
PL70     17.40 dB
PL71     17.40 dB
PL72     17.40 dB
PL73     17.40 dB
PL74     17.40 dB
PL75     17.40 dB
PL76     17.40 dB
PL77     17.40 dB
PL78     17.40 dB
PL79     17.40 dB
PL80     17.40 dB
PL81     17.40 dB
PL82     17.40 dB
PL83     17.40 dB
PL84     17.40 dB
PL85     17.40 dB
PL86     17.40 dB
PL87     17.40 dB
PL88     17.40 dB
PL89     17.40 dB
PL90     17.40 dB
PL91     17.40 dB
PL92     17.40 dB
PL93     17.40 dB
PL94     17.40 dB
PL95     17.40 dB
PL96     17.40 dB
PL97     17.40 dB
PL98     17.40 dB
PL99     17.40 dB
PL100     17.40 dB
SFO1       125.7703643 MHz
SFO2       400.1300029 MHz
SI         32768
SF         125.7577922 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
CB         0
PC         1.00

```

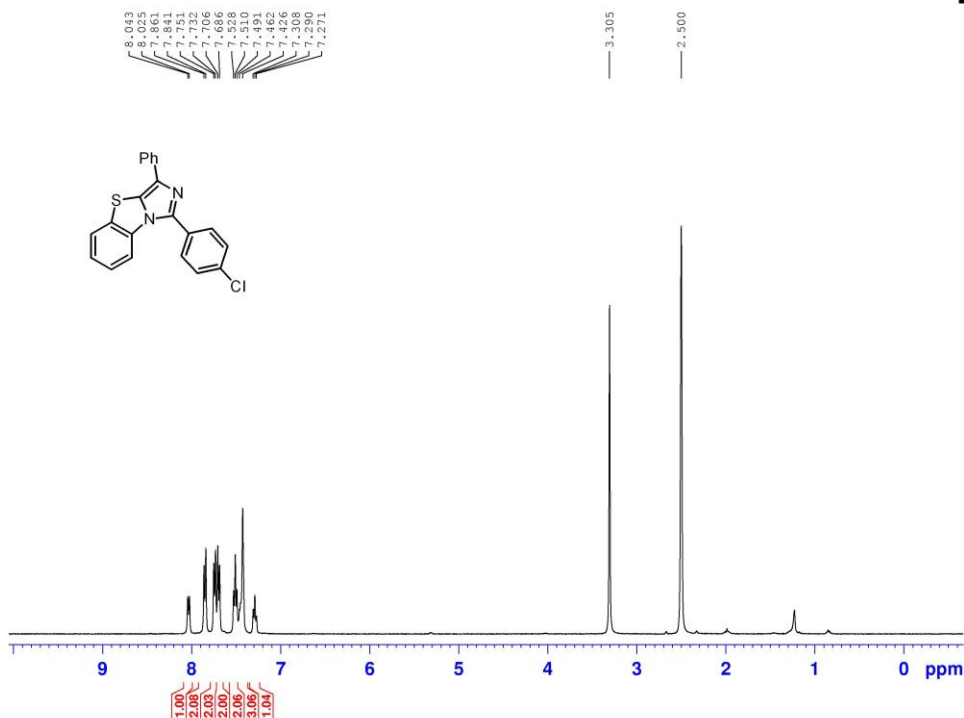
3n



0027-1



30



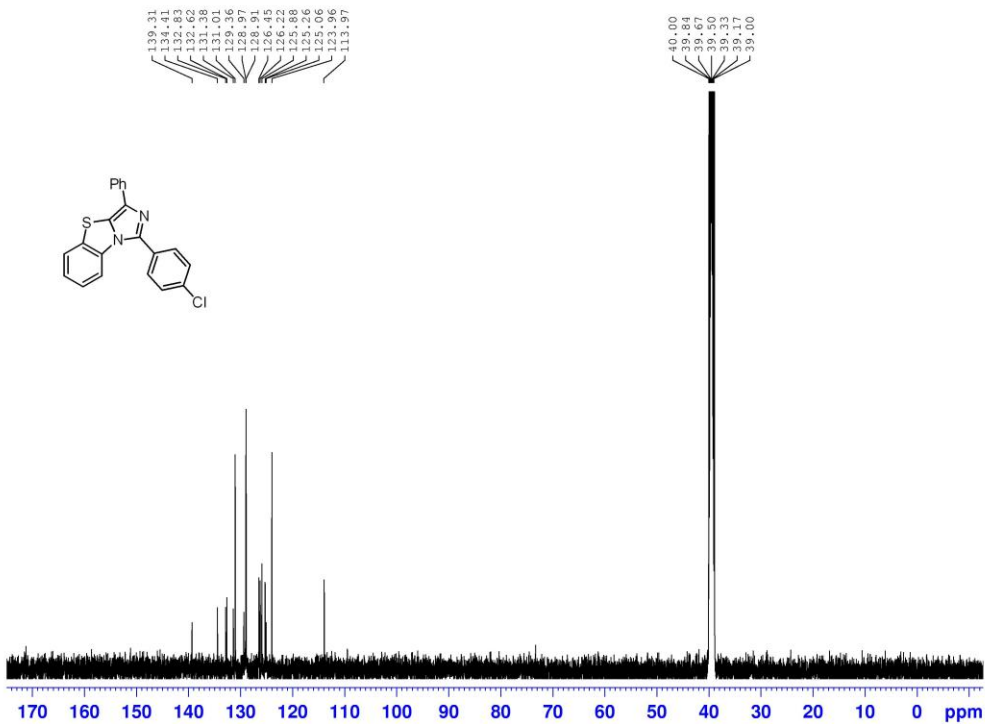
```

NAME          0037-1H
EXPNO         1
PROCNO        1
Date_         20150113
Time          14.24
INSTRUM       spect
PROBHD        5 mm DUL 13C-1
PULPROG       zg30
TD            65536
SOLVENT       DMSO
NS            16
DS            2
SWH           8278.144 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG            362
AQ            60.400 usec
DE            6.50 usec
TE            298.0 K
D1            1.00000000 sec
D11           1
D10           1
  
```

```

----- CHANNEL f1 -----
NUC1          1H
P1            12.00 usec
P11           0.00 dB
P1LW          10.87646886 W
SFO1          400.1324710 MHz
SI            32768
SF            400.1300028 MHz
WCMW         EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

0037-1



```

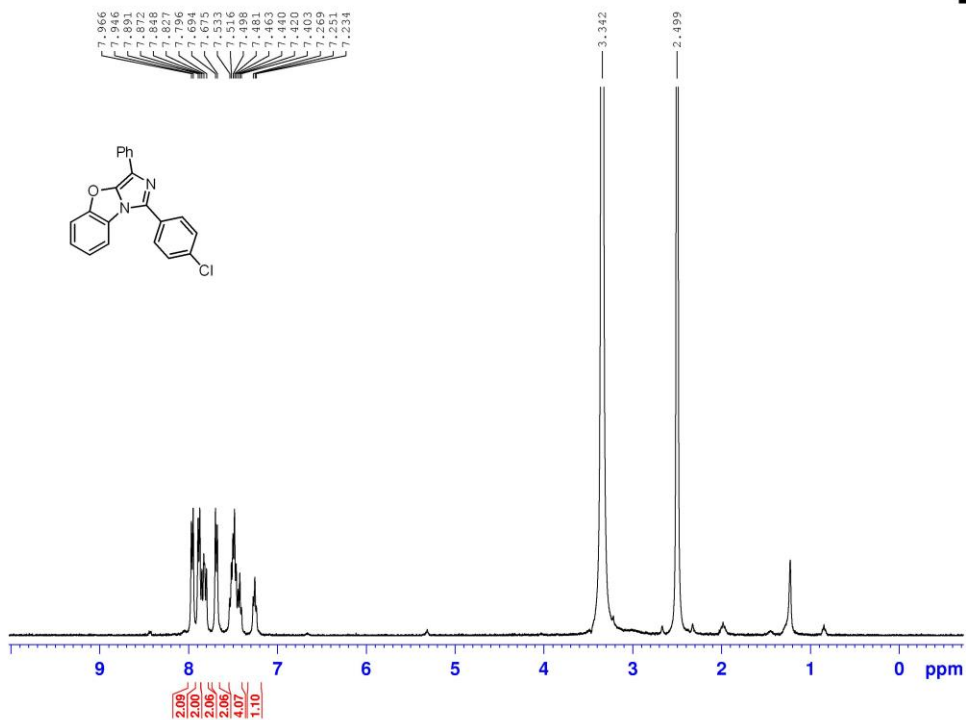
NAME          0037-1C
EXPNO         2
PROCNO        1
Date_         20150122
Time          17.59
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       DMSO
NS            1713
DS            4
SWH           29761.904 Hz
FIDRES        0.454151 Hz
AQ            1.1010345 sec
RG            203
AQ            16.800 usec
DE            6.50 usec
TE            298.4 K
D1            2.00000000 sec
D11           0.03000000 sec
D10           1
  
```

```

----- CHANNEL f1 -----
NUC1          13C
P1            13.84 usec
P11           2.15 dB
P1LW          46.89624786 W
SFO1          125.7703643 MHz

----- CHANNEL f2 -----
CPDPRG2      waltz16
NUC2          1H
PCPD2         80.00 usec
P12           2.50 dB
P1L2          37.40 dB
P1L3          37.40 dB
P1LW          13.0239581 W
SFO2          0.42145536 W
P1L3W         0.42145536 W
SFO2          500.1320005 MHz
SI            32768
SF            125.7578553 MHz
WCMW         EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

3p

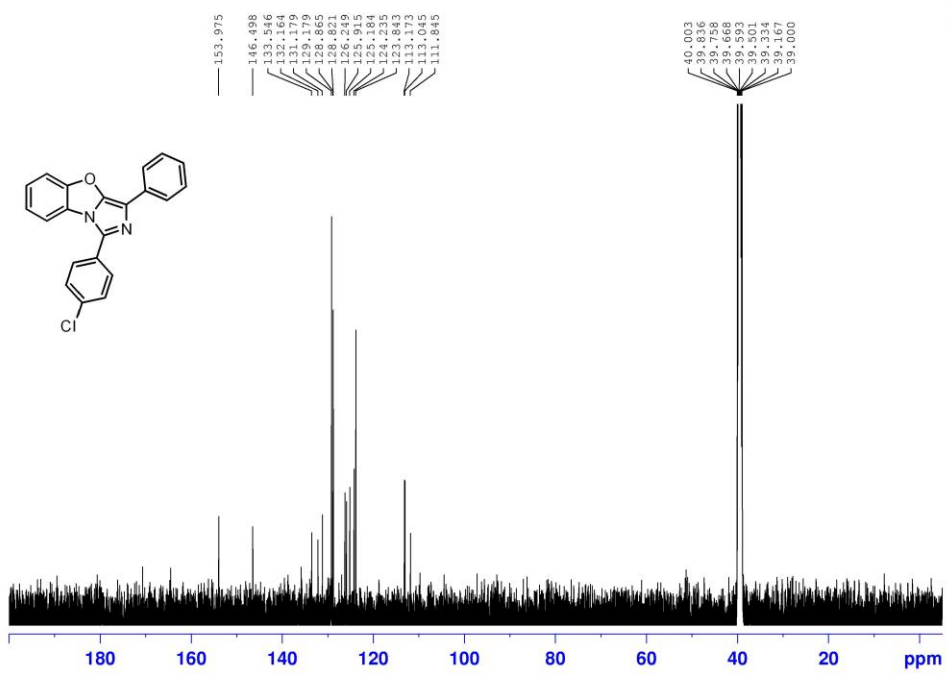


```

NAME          0040h
EXPNO         1
PROCNO        1
Date_         20150128
Time          2.24
INSTRUM       spect
PROBHD        5 mm DUL 13C-1
PULPROG       zg30
TD            65536
SOLVENT       DMSO
NS            16
DS            2
SWH           8278.144 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG            321.5
FW           60.400 usec
DE            6.50 usec
TE            296.2 K
D1            1.00000000 sec
D11           1
TD0           1

----- CHANNEL f1 -----
NUC1          1H
P1            12.55 usec
PL1           0.00 dB
PL1W          10.87646886 W
SFO1          400.1324710 MHz
SI            32768
SF            400.1300031 MHz
WOW           0
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

3p



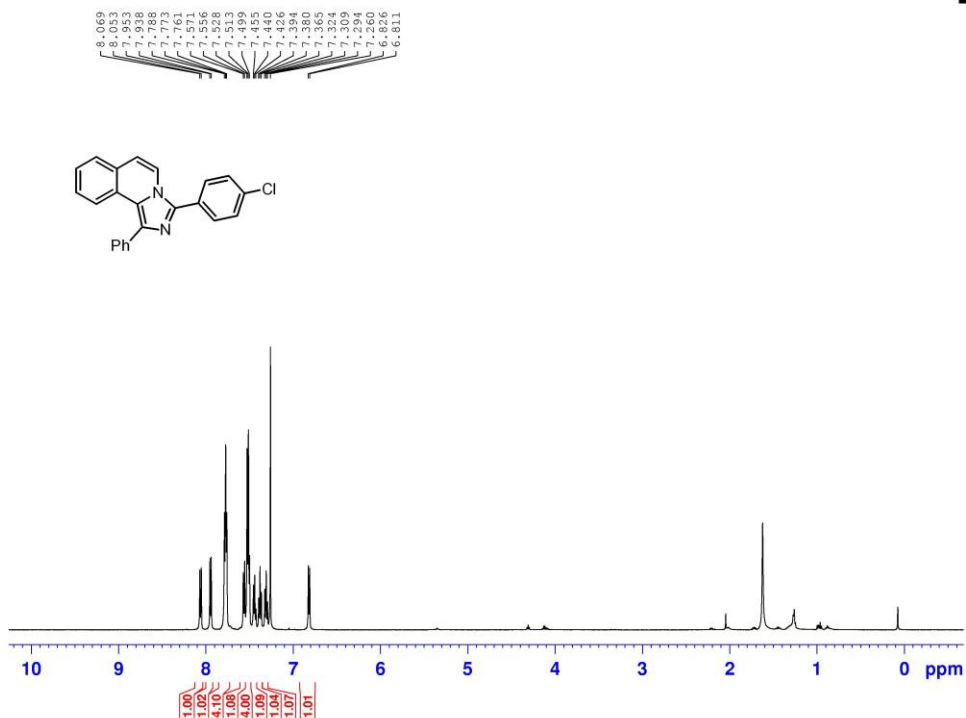
```

NAME          3p
EXPNO         1
PROCNO        1
Date_         20151104
Time          6.55
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       DMSO
NS            2368
DS            4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010548 sec
RG            203
DW            16.900 usec
DE            6.50 usec
TE            300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

----- CHANNEL f1 -----
NUC1          13C
P1            13.84 usec
PL1           2.50 dB
PL1W          46.89624786 W
SFO1          125.7703643 MHz

----- CHANNEL f2 -----
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           2.50 dB
PL12          17.40 dB
PL13          17.40 dB
PL1W          13.02359581 W
PL1W          0.42145336 W
PL1W          0.42145336 W
SFO2          500.1320005 MHz
SI            32768
SF            125.7578908 MHz
WOW           0
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

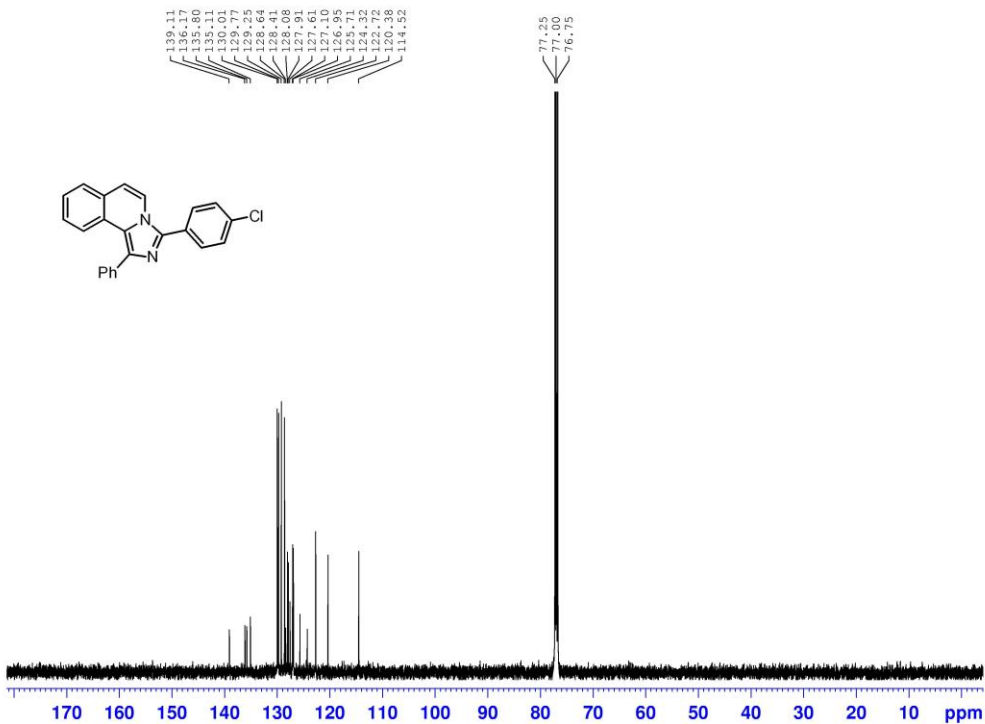
3q



NAME 0049-3
EXPNO 1
PROCNO 1
Date_ 20150117
Time 6.39
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.187632 Hz
AQ 3.1719923 sec
RG 203
RW 48.400 usec
DE 6.50 usec
TE 298.3 K
D1 1.0000000 sec
TD0 1

----- CHANNEL f1 -----
NUC1 1H
P1 14.00 usec
PL1 2.50 dB
PL1W 13.0239581 W
SFO1 500.1330883 MHz
SI 32768
SF 500.1300233 MHz
WVW F2
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

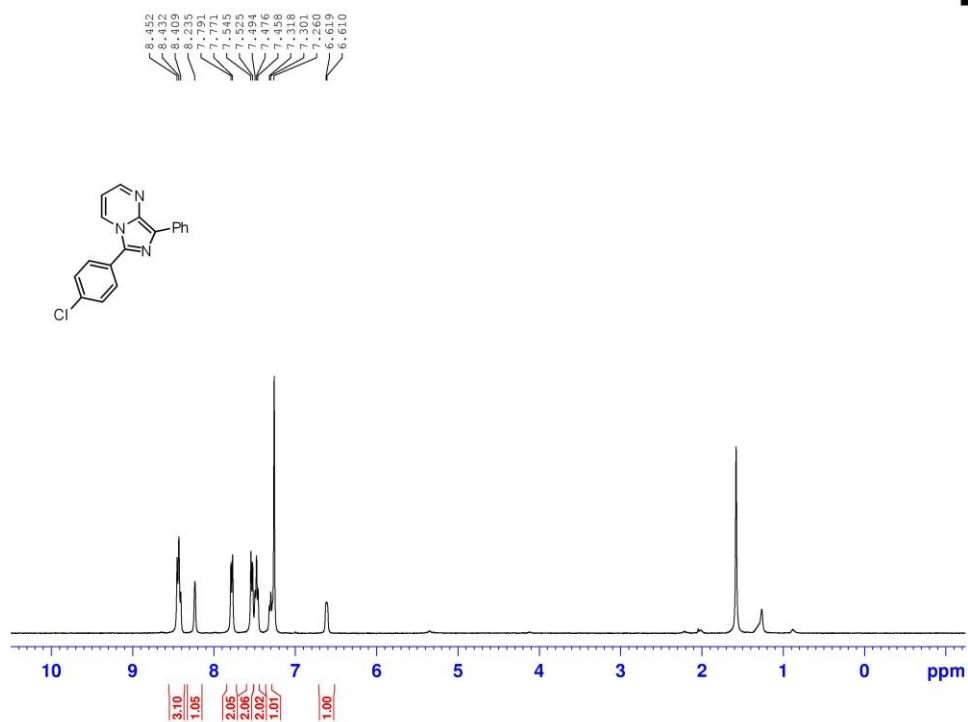
0049-3C



NAME 0049-3
EXPNO 2
PROCNO 2
Date_ 20150124
Time 18.46
INSTRUM Spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1054
DS 2
SWH 29761.904 Hz
FIDRES 0.434131 Hz
AQ 1.1101048 sec
RG 203
RW 16.500 usec
DE 302.2 K
TE 2.0000000 sec
D1 0.0300000 sec
D11 1
TD0 1

----- CHANNEL f1 -----
NUC1 13C
P1 13.84 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7703643 MHz
----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL2 17.40 dB
PL13 17.40 dB
PL2W 13.0239581 W
PL13W 0.42143536 W
PL2W 0.42143536 W
SFO2 500.1330883 MHz
SI 32768
SF 125.7577921 MHz
WVW F2
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

3r



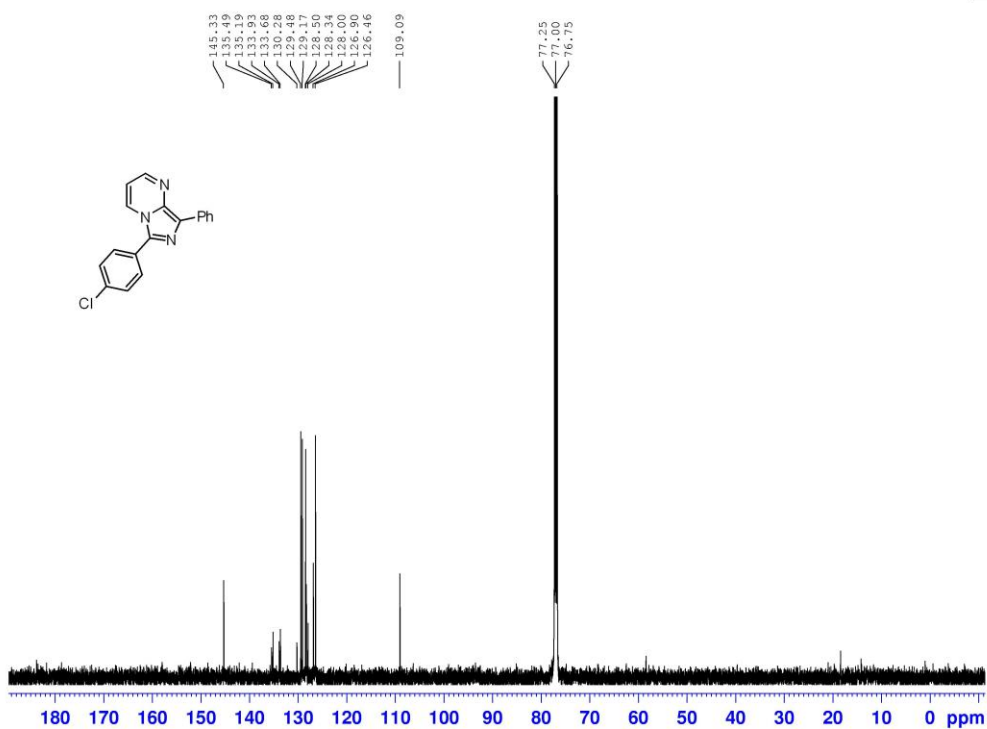
```

NAME          0049-2H
EXPNO         1
PROCNO        1
Date_         20150120
Time          13.39
INSTRUM       spect
PROBHD        5 mm DUL 15C-1
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9594243 sec
RG            456.1
RC            456.1
DW            60.400 usec
DE            6.50 usec
TE            297.3 K
D1            1.0000000 sec
TD0
  
```

```

----- CHANNEL f1 -----
NUC1          1H
P1            12.58 usec
PL1           0.00 dB
PL1W          10.87646866 W
SFO1          400.1324710 MHz
SI            32768
SF            400.1300096 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

0049-2C



```

NAME          0049-2
EXPNO         2
PROCNO        2
Date_         20150124
Time          16.38
INSTRUM       spect
PROBHD        5 mm PABBO 8B
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            1812
DS            4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010546 sec
RG            203
RC            203
DW            16.800 usec
DE            6.50 usec
TE            301.1 K
D1            2.0000000 sec
D11           0.03000000 sec
TD0           1
  
```

```

----- CHANNEL f1 -----
NUC1          13C
P1            13.84 usec
PL1           2.50 dB
PL1W          46.89624786 W
SFO1          125.7703643 MHz

----- CHANNEL f2 -----
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           2.50 dB
PL12          17.40 dB
PL13          17.40 dB
PL2W          13.02339381 W
PL12W         0.42145536 W
PL13W         0.42145536 W
SFO2          500.1320005 MHz
SI            32768
SF            125.7577912 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```