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SUPPORTING INFROMATION

Palladium-Catalyzed C(sp^2)-H Aminoimidoylation of Isocyano-Containing Arenes: Synthesis of Amino Substituted N-Heterocycles

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Contents

I. General Information

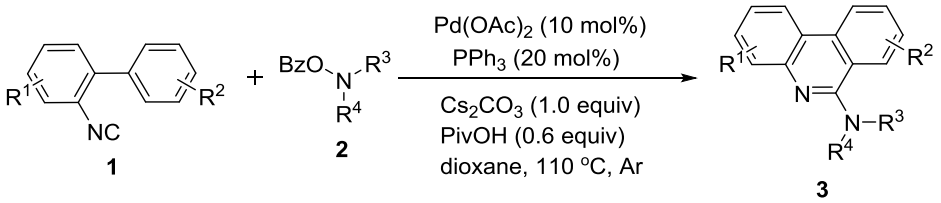
^{1}H NMR (400 MHz) and ^{13}C NMR (125 MHz) were registered on 400 M and 500 M spectrometers. Chemical shifts were reported in units (ppm) by assigning TMS resonance in the ^{1}H spectrum as 0.00 ppm, $CDCl_3$ resonance in the ^{13}C spectrum as 77.0 ppm. All coupling constants (*J* values) were reported in Hertz (Hz). NMR analysis was carried out at 298 K unless noted otherwise. IR spectra were recorded on a Bruker Tensor 27 spectrometer using a diamond comb. Melting points were performed on an X-6 spectrometer. HRMS was obtained on an ESI-LC-MS/MS spectrometer. Isocyanides and *N*-benzoyloxyamines were prepared according to the following literatures:

(1) M. Tobisu, K. Koh, T. Furukawa, N. Chatani, *Angew. Chem., Int. Ed.* 2012, **51**, 11363.

(2) H. Wang, Y. Yu, X. Hong, B. Xu, *Chem. Commun.* 2014, **50**, 13485.

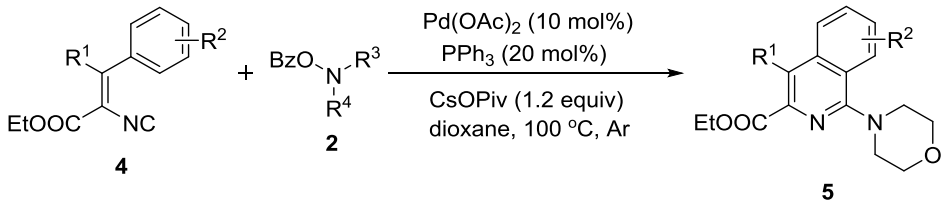
(3) A. M. Berman, J. S. Johnson, *J. Org. Chem.* 2006, **71**, 219.

II. General Procedure



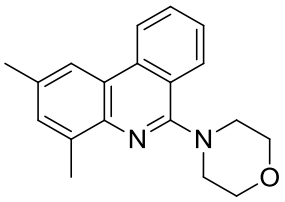
General procedure A: An oven-dried 25 mL Schlenk tube charged with $Pd(OAc)_2$ (0.01 mmol, 2.24 mg), PPh_3 (0.02 mmol, 5.24 mg), Cs_2CO_3 (0.10 mmol, 32.6 mg) and **2** (0.15 mmol) was refilled with Ar for 3 times. Then a solution of pivalic acid (0.06 mmol, 7.0 μL) in 0.5 mL of dioxane was added by syringe and the tube was placed in a 110 °C oil-bath. A solution of **1** (0.1 mmol) in 1.0 mL of dioxane was added dropwise within 1 h by a syringe pump to the reaction mixture. After reacting for another 1-2 h, the reaction was completed. The crude reaction mixture was extracted with EA (20 mL × 3) and washed with brine (20 mL). The organic phase was concentrated in *vacuo* and the residue was purified by silica gel flash column chromatography to afford the corresponding amino-substituted

phenanthridines **3**.



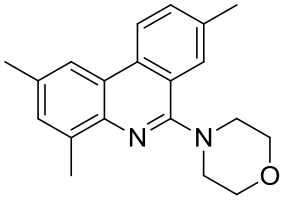
General procedure B: An oven-dried 25 mL Schlenk tube charged with $Pd(OAc)_2$ (0.01 mmol, 2.24 mg), PPh_3 (0.02 mmol, 5.24 mg), CsOPiv (0.12 mmol, 28.08 mg) and **2** (0.15 mmol) was refilled with Ar for 3 times. Then 0.5 mL of dioxane was added by syringe and the tube was placed in a 100 °C oil-bath. A solution of **4** (0.10 mmol) in 1.0 mL of dioxane was added dropwise within 1 h by a syringe pump to the reaction mixture. After reacting for another 1-2 h, the reaction was completed. The crude reaction mixture was extracted with EA (20 mL × 3) and washed with brine (20 mL). The organic phase was concentrated in *vacuo* and the residue was purified by silica gel flash column chromatography to afford the corresponding amino-substituted isoquinolines **5**.

III. Characterization Data



4-(2,4-dimethylphenanthridin-6-yl)morpholine (**3a**)

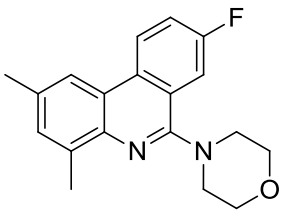
Prepared from 2-isocyano-3,5-dimethyl-1,1'-biphenyl (20.7 mg, 0.10 mmol, 1.0 equiv) and morpholino benzoate (31.1 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether = 1 : 30) furnished the product **3a** as a white solid (23 mg, 0.078 mmol, 78% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.55 (d, *J* = 8.3 Hz, 1H), 8.19 (d, *J* = 8.1 Hz, 1H), 8.09 (s, 1H), 7.74 (t, *J* = 7.2 Hz, 1H), 7.59 (t, *J* = 7.2 Hz, 1H), 7.36 (s, 1H), 4.01 (t, *J* = 4.5 Hz, 4H), 3.51 (t, *J* = 4.6 Hz, 4H), 2.74 (s, 3H), 2.54 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 157.9, 140.4, 136.3, 135.3, 134.0, 131.2, 129.8, 126.4, 126.2, 123.1, 122.3, 121.0, 119.4, 67.2, 51.9, 21.9, 18.2; IR (KBr): 3069, 2967, 2913, 2893, 2857, 1607, 1574, 1522, 1447, 1278, 772 cm^{-1}; HRMS: calcd for $C_{19}H_{20}N_2O$ ($M+H^+$) 293.1648; found 293.1645; mp: 118-120 ^{o}C.



4-(2,4,8-trimethylphenanthridin-6-yl)morpholine (**3b**)

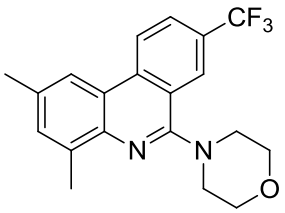
Prepared from 2-isocyano-3,4',5-trimethyl-1,1'-biphenyl (22.1 mg, 0.10 mmol, 1.0 equiv) and morpholino benzoate (31.1 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether = 1 : 16) furnished the product **3b** as a light yellow solid (16 mg, 0.052 mmol, 52% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.44 (d, *J* = 8.4 Hz, 1H), 8.06 (s, 1H), 7.96 (s, 1H), 7.56 (d, *J* = 8.4 Hz, 1H), 7.33 (s, 1H), 4.02 (t, *J* = 4.5 Hz, 4H), 3.50 (t, *J* = 4.6 Hz, 4H), 2.73 (s, 3H), 2.58 (s, 3H), 2.54 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 157.7, 140.0, 136.3, 136.2, 133.9, 133.1, 131.5, 130.8, 125.6, 123.1, 122.4, 121.1, 119.2, 67.2, 51.9, 22.0, 21.9, 18.2; IR (KBr): 3010, 2954, 2919, 2890, 2867, 1605, 1573, 1528, 1451, 1282, 789 cm^{-1}; HRMS: calcd for $C_{20}H_{22}N_2O$ ($M+H^+$) 307.1805; found

307.1805; mp: 88-90 ^{o}C.



4-(8-fluoro-2,4-dimethylphenanthridin-6-yl)morpholine (**3c**)

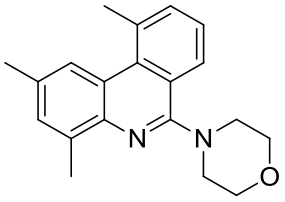
Prepared from 4'-fluoro-2-isocyano-3,5-dimethyl-1,1'-biphenyl (22.5 mg, 0.10 mmol, 1.0 equiv) and morpholino benzoate (31.1 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether = 1 : 8) furnished the product **3c** as a light yellow solid (21 mg, 0.068 mmol, 68% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.55-8.51 (m, 1H), 8.02 (s, 1H), 7.81-7.78 (m, 1H), 7.51-7.45 (m, 1H), 7.35 (s, 1H), 4.01 (t, *J* = 4.5 Hz, 4H), 3.46 (t, *J* = 4.6 Hz, 4H), 2.72 (s, 3H), 2.53 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 162.2 (d, *J* = 246.8 Hz), 157.3 (d, *J* = 4.1 Hz), 140.0, 136.6, 134.6, 131.8 (d, *J* = 1.8 Hz), 131.1, 125.7 (d, *J* = 8.23 Hz), 122.5 (d, *J* = 7.4 Hz), 121.9, 119.2, 119.0 (d, *J* = 23.8 Hz), 110.9 (d, *J* = 21.8 Hz), 67.1, 51.8, 21.9, 18.2; IR (KBr): 3071, 2981, 2916, 2895, 2859, 1617, 1576, 1529, 1450, 1282, 752 cm^{-1}; HRMS: calcd for $C_{19}H_{19}FN_2O$ ($M+H^+$) 311.1554; found 311.1554; mp: 127-129 ^{o}C.



4-(2,4-dimethyl-8-(trifluoromethyl)phenanthridin-6-yl)morpholine (**3d**)

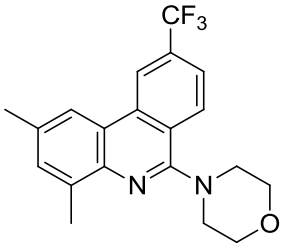
Prepared from 2-isocyano-3,5-dimethyl-4'-(trifluoromethyl)-1,1'-biphenyl (27.5 mg, 0.10 mmol, 1.0 equiv) and morpholino benzoate (31.1 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether = 1 : 16) furnished the product **3d** as a white solid (26 mg, 0.072 mmol, 72% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.63 (d, *J* = 8.7 Hz, 1H), 8.45 (s, 1H), 8.07 (s, 1H), 7.91 (d, *J* = 8.7 Hz, 1H), 7.42 (s, 1H), 4.02 (t, *J* = 4.5 Hz, 4H), 3.49 (t, *J* = 4.6 Hz, 4H), 2.73 (s, 3H), 2.55 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 157.7, 141.2, 137.5, 136.7, 134.7, 132.4, 128.4 (q, *J* = 32.7 Hz), 125.7 (q, *J* = 3.53 Hz),

125.4 (q, J = 272.3 Hz), 124.2, 123.8 (q, J = 4.2 Hz), 121.4, 120.4, 119.7, 66.9, 51.9, 21.9, 18.1; IR (KBr): 3015, 2973, 2920, 2867, 2848, 1625, 1577, 1456, 1435, 1274, 797 cm^{-1}; HRMS: calcd for $C_{20}H_{19}F_3N_2O$ ($M+H^+$) 361.1522; found 361.1521; mp: 127-129 ^oC.



4-(2,4,10-trimethylphenanthridin-6-yl)morpholine (**3e**)

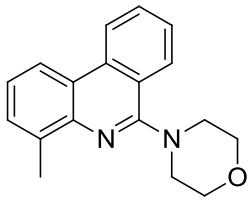
Prepared from 2-isocyano-2',3,5-trimethyl-1,1'-biphenyl (22.1 mg, 0.10 mmol, 1.0 equiv) and morpholino benzoate (31.1 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether = 1 : 16) furnished the product **3e** as a white solid (24 mg, 0.076 mmol, 76% yield). ^1H NMR (400 MHz, $CDCl_3$): δ 8.35 (s, 1H), 8.18 (d, J = 7.9 Hz, 1H), 7.58 (d, J = 7.0 Hz, 1H), 7.49 (t, J = 7.6 Hz, 1H), 7.38 (s, 1H), 4.01 (t, J = 4.5 Hz, 4H), 3.46 (t, J = 4.6 Hz, 4H), 3.09 (s, 3H), 2.76 (s, 3H), 2.55 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 158.6, 141.3, 136.2, 135.9, 134.8, 134.2, 132.9, 130.5, 126.0, 124.8, 124.3, 123.8, 122.4, 67.2, 51.9, 27.1, 22.3, 18.8; IR (KBr): 3018, 2952, 2918, 2884, 2850, 1614, 1583, 1528, 1448, 1279, 750 cm^{-1}; HRMS: calcd for $C_{20}H_{22}N_2O$ ($M+H^+$) 307.1805; found 307.1804; mp: 125-127 ^oC.



4-(2,4-dimethyl-9-(trifluoromethyl)phenanthridin-6-yl)morpholine (**3f**)

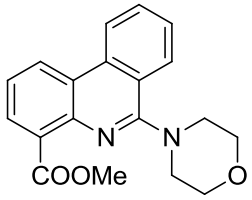
Prepared from 2-isocyano-3,5-dimethyl-3'-(trifluoromethyl)-1,1'-biphenyl (27.5 mg, 0.10 mmol, 1.0 equiv) and morpholino benzoate (31.1 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether = 1 : 16) furnished the product **3f** as a light yellow solid (16 mg, 0.045 mmol, 45 % yield). ^1H NMR (400 MHz, $CDCl_3$): δ 8.80 (s, 1H), 8.29 (d, J = 8.6 Hz, 1H), 8.08 (s, 1H), 7.79 (d, J = 8.6 Hz, 1H), 7.41 (s, 1H), 4.02 (t, J = 4.5 Hz, 4H),

3.49 (t, *J* = 4.7 Hz, 4H), 2.73 (s, 3H), 2.56 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 157.2, 140.7, 136.5, 134.9, 134.7, 132.0, 131.5 (q, *J* = 32.3 Hz), 127.2, 125.2 (q, *J* = 273.0 Hz), 122.5, 122.2 (q, *J* = 3.6 Hz), 121.6, 120.5 (q, *J* = 3.8 Hz), 119.3, 66.9, 51.7, 21.8, 17.9; IR (KBr): 3050, 2960, 2916, 2893, 2857, 1727, 1585, 1517, 1453, 1278, 798 cm^{-1}; HRMS: calcd for $C_{20}H_{19}F_3N_2O$ ($M+H^+$) 361.1522; found 361.1526; mp: 168-170 ^{o}C.



4-(4-methylphenanthridin-6-yl)morpholine (**3g**)

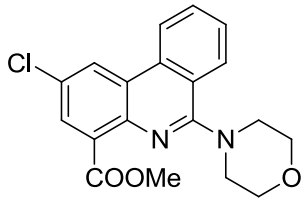
Prepared from 2-isocyano-3-methyl-1, 1'-biphenyl (19.3 mg, 0.10 mmol, 1.0 equiv) and morpholino benzoate (31.1 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether = 1 : 16) furnished the product **3g** as a light yellow solid (16 mg, 0.058 mmol, 58% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.57 (d, *J* = 8.3 Hz, 1H), 8.30 (d, *J* = 8.1 Hz, 1H), 8.20 (d, *J* = 8.0 Hz, 1H), 7.76 (t, *J* = 8.2 Hz, 1H), 7.61 (t, *J* = 8.0 Hz, 1H), 7.52 (d, *J* = 7.1 Hz, 1H), 7.40 (t, *J* = 7.5 Hz, 1H), 4.02 (t, *J* = 4.5 Hz, 4H), 3.54 (t, *J* = 4.7 Hz, 4H), 2.78 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 158.5, 142.2, 136.6, 135.5, 130.0, 129.5, 126.6, 126.3, 124.5, 123.2, 122.4, 120.9, 119.7, 67.1, 51.8, 18.3; IR (KBr): 3021, 2958, 2917, 2884, 2852, 1610, 1576, 1522, 1458, 1273, 786 cm^{-1}; HRMS: calcd for $C_{18}H_{18}N_2O$ ($M+H^+$) 279.1492; found 279.1493; mp: 109-110 ^{o}C.



Methyl 6-morpholinophenanthridine-4-carboxylate (**3h**)

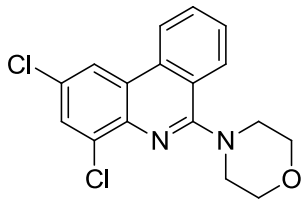
Prepared from methyl 2-isocyano-[1,1'-biphenyl]-3-carboxylate (23.7 mg, 0.10 mmol, 1.0 equiv) and morpholino benzoate (31.1 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether = 1 : 4) furnished the product **3h** as a white solid (21 mg, 0.066 mmol, 66% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.53 (t, *J* = 8.2 Hz, 1H), 8.15 (d, *J* = 8.1

Hz, 1H), 7.86 (d, *J* = 7.3 Hz, 1H), 7.78 (t, *J* = 7.3 Hz, 1H), 7.63 (t, *J* = 7.3 Hz, 1H), 7.48 (t, *J* = 7.8 Hz, 1H), 4.03 (s, 3H), 3.98 (t, *J* = 4.5 Hz, 4H), 3.57 (t, *J* = 4.7 Hz, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 169.7, 159.6, 141.3, 134.8, 130.5, 128.8, 127.2, 126.5, 124.7, 123.8, 123.1, 122.9, 120.9, 67.0, 52.4, 51.5; IR (KBr): 3067, 2998, 2954, 2919, 2829, 1610, 1573, 1523, 1453, 1276, 768 cm^{-1}; HRMS: calcd for $C_{19}H_{18}N_2O_3$ ($M+H^+$) 323.1390; found 323.1391; mp: 110-112 ^oC.



Methyl 2-chloro-6-morpholinophenanthridine-4-carboxylate (**3i**)

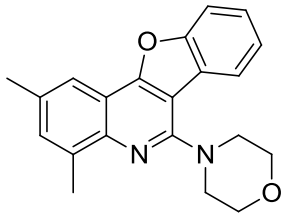
Prepared from methyl 5-chloro-2-isocyano-[1,1'-biphenyl]-3-carboxylate (27.2 mg, 0.10 mmol, 1.0 equiv) and morpholino benzoate (31.1 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether = 1 : 16) furnished the product **3i** as a white solid (24 mg, 0.067 mmol, 67% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.45-8.43 (m, 2H), 8.14 (d, *J* = 8.0 Hz, 1H), 7.81 (s, 1H), 7.78 (d, *J* = 8.3 Hz, 1H), 7.66 (t, *J* = 7.2 Hz, 1H), 4.02 (s, 3H), 3.96 (t, *J* = 4.5 Hz, 4H), 3.57 (t, *J* = 4.7 Hz, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.2, 159.7, 139.9, 133.9, 132.4, 130.8, 129.4, 129.0, 127.9, 126.6, 124.2, 123.1, 121.0, 66.9, 52.6, 51.5; IR (KBr): 3076, 2970, 2948, 2890, 2846, 1613, 1583, 1524, 1448, 1260, 758 cm^{-1}; HRMS: calcd for $C_{19}H_{17}ClN_2O_3$ ($M+H^+$) 357.1000; found 357.1002; mp: 131-133 ^oC.



4-(2,4-dichlorophenanthridin-6-yl)morpholine (**3j**)

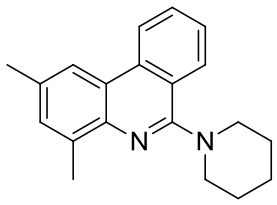
Prepared from 3,5-dichloro-2-isocyano-1,1'-biphenyl (24.8 mg, 0.10 mmol, 1.0 equiv) and morpholino benzoate (31.1 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether = 1 : 16) furnished the product **3j** as a light yellow solid (22 mg, 0.066 mmol, 66% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.45 (d, *J* = 8.2 Hz, 1H), 8.28 (d, *J* = 2.2 Hz,

1H), 8.17 (d, *J* = 7.9 Hz, 1H), 7.80 (t, *J* = 8.2 Hz, 1H), 7.71 (d, *J* = 2.2 Hz, 1H), 7.68 (d, *J* = 8.0 Hz, 1H), 4.00 (t, *J* = 4.5 Hz, 4H), 3.62 (t, *J* = 4.5 Hz, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 159.9, 138.9, 134.2, 133.8, 130.9, 129.6, 129.3, 127.9, 126.7, 124.7, 123.4, 121.3, 120.5, 66.9, 51.7; IR (KBr): 3082, 2970, 2916, 2863, 2848, 1611, 1583, 1517, 1444, 1272, 703 cm^{-1}; HRMS: calcd for $C_{17}H_{14}Cl_2N_2O$ ($M+H^+$) 333.0556; found 333.0557; mp: 204-206 ^{o}C.



2,4-dimethyl-6-morpholinobenzofuro[3,2-*c*]quinolone (**3k**)

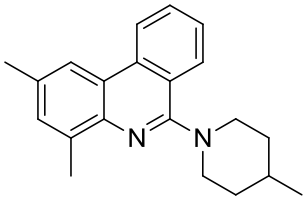
Prepared from 2-(2-isocyano-3,5-dimethylphenyl)benzofuran (24.7 mg, 0.10 mmol, 1.0 equiv) and morpholino benzoate (31.1 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether = 1 : 16) furnished the product **3k** as a light yellow solid (21 mg, 0.061 mmol, 61% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.93 (s, 1H), 7.84 (d, *J* = 7.6 Hz, 1H), 7.72 (d, *J* = 7.9 Hz, 1H), 7.51-7.43 (m, 2H), 7.39 (s, 1H), 4.05 (t, *J* = 4.5 Hz, 4H), 3.64 (t, *J* = 4.5 Hz, 4H), 2.75 (s, 3H), 2.54 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 159.7, 155.8, 154.9, 143.7, 136.0, 133.9, 132.1, 126.2, 123.9, 123.0, 122.2, 117.7, 114.9, 111.9, 108.5, 67.1, 49.9, 21.7, 18.4; IR (KBr): 3030, 2950, 2913, 2857, 2819, 1634, 1594, 1507, 1435, 1275, 752 cm^{-1}; HRMS: calcd for $C_{21}H_{20}N_2O_2$ ($M+H^+$) 333.1598; found 333.1596; mp: 107-109 ^{o}C.



2,4-dimethyl-6-(piperidin-1-yl)phenanthridine (**3l**)

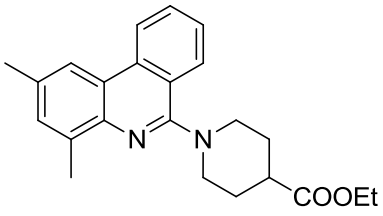
Prepared from 2-isocyano-3,5-dimethyl-1,1'-biphenyl (20.7 mg, 0.10 mmol, 1.0 equiv) and piperidin-1-yl benzoate (30.8 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether = 1 : 60) furnished the product **3l** as a light yellow solid (24 mg, 0.081 mmol, 81%

yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.53 (d, *J* = 8.2 Hz, 1H), 8.19 (d, *J* = 8.0 Hz, 1H), 8.08 (s, 1H), 7.72 (t, *J* = 7.2 Hz, 1H), 7.58 (t, *J* = 7.2 Hz, 1H), 7.35 (s, 1H), 3.46 (t, *J* = 5.1 Hz, 4H), 2.75 (s, 3H), 2.54 (s, 3H), 1.91-1.85 (m, 4H), 1.76-1.71 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 159.2, 140.7, 136.1, 135.2, 133.4, 131.0, 129.6, 126.7, 126.2, 122.9, 122.1, 121.6, 119.4, 52.7, 26.3, 25.2, 21.9, 18.1; IR (KBr): 3068, 2967, 2928, 2854, 1646, 1610, 1584, 1520, 1464, 1264, 774 cm^{-1}; HRMS: calcd for $C_{20}H_{22}N_2$ ($M+H^+$) 291.1856; found 291.1852; mp: 97-99 ^{o}C.



2,4-dimethyl-6-(4-methylpiperidin-1-yl)phenanthridine (**3m**)

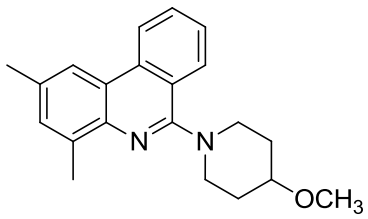
Prepared from 2-isocyano-3, 5-dimethyl-1, 1'-biphenyl (20.7 mg, 0.10 mmol, 1.0 equiv) and 4-methylpiperidin-1-yl benzoate (32.9 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether = 1 : 60) furnished the product **3m** as a light yellow solid (21 mg, 0.070 mmol, 70% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.53 (d, *J* = 8.2 Hz, 1H), 8.18 (d, *J* = 8.2 Hz, 1H), 8.08 (s, 1H), 7.72 (t, *J* = 7.7 Hz, 1H), 7.58 (t, *J* = 7.7 Hz, 1H), 7.35 (s, 1H), 3.94 (d, *J* = 13.2 Hz, 1H), 3.01 (t, *J* = 12.6 Hz, 4H), 2.75 (s, 3H), 2.54 (s, 3H), 1.90-1.80 (m, 2H), 1.74-1.52 (m, 3H), 1.09 (d, *J* = 6.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 158.9, 140.7, 136.1, 135.1, 133.4, 131.0, 129.6, 126.7, 126.2, 122.9, 122.1, 121.6, 119.4, 51.9, 34.7, 31.6, 22.3, 21.9, 18.2; IR (KBr): 3069, 2955, 2922, 2861, 2826, 1609, 1574, 1520, 1453, 1281, 773 cm^{-1}; HRMS: calcd for $C_{21}H_{24}N_2$ ($M+H^+$) 305.2012; found 305.2011; mp: 96-98 ^{o}C.



Ethyl 1-(2,4-dimethylphenanthridin-6-yl)piperidine-4-carboxylate (**3n**)

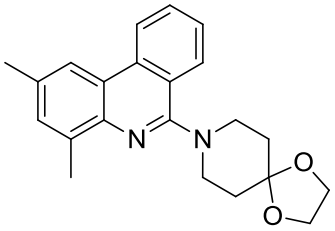
Prepared from 2-isocyano-3, 5-dimethyl-1, 1'-biphenyl (20.7 mg, 0.10 mmol, 1.0 equiv) and ethyl 1-(benzoyloxy)piperidine-4-carboxylate (41.6 mg, 0.15 mmol, 1.5

equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether = 1 : 16) furnished the product **3n** as a light yellow oil (18 mg, 0.050 mmol, 50% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.53 (d, *J* = 8.2 Hz, 1H), 8.17 (d, *J* = 8.2 Hz, 1H), 8.07 (s, 1H), 7.72 (t, *J* = 7.7 Hz, 1H), 7.58 (t, *J* = 7.7 Hz, 1H), 7.34 (s, 1H), 4.21 (q, *J* = 7.1 Hz, 2H), 3.95-3.90 (m, 2H), 3.10-3.03 (m, 2H), 2.73 (s, 3H), 2.63-2.57 (m, 1H), 2.53 (s, 3H), 2.14-2.08 (m, 4H), 1.32 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 175.4, 158.6, 140.5, 136.2, 135.1, 133.7, 131.1, 129.7, 126.4, 126.3, 122.9, 122.2, 121.4, 119.4, 60.5, 51.1, 41.9, 28.6, 21.9, 18.1, 14.4; IR (KBr): 3071, 2955, 2923, 2854, 2820, 1610, 1576, 1521, 1446, 1289, 776 cm^{-1}; HRMS: calcd for $C_{23}H_{26}N_2O_2$ ($M+H^+$) 363.2067; found 363.2070.



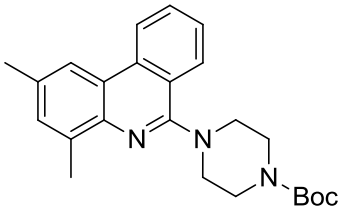
6-(4-methoxypiperidin-1-yl)-2,4-dimethylphenanthridine (**3o**)

Prepared from 2-isocyano-3,5-dimethyl-1,1'-biphenyl (20.7 mg, 0.10 mmol, 1.0 equiv) and 4-methoxypiperidin-1-yl benzoate (35.3 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether = 1 : 16) furnished the product **3o** as a white solid (18 mg, 0.057 mmol, 57% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.53 (d, *J* = 8.2 Hz, 1H), 8.17 (d, *J* = 8.1 Hz, 1H), 8.07 (s, 1H), 7.72 (t, *J* = 7.1 Hz, 1H), 7.58 (t, *J* = 7.1 Hz, 1H), 7.34 (s, 1H), 3.86-3.80 (m, 2H), 3.49-3.47 (m, 1H), 3.44 (s, 3H), 3.22-3.15 (m, 2H), 2.73 (s, 3H), 2.53 (s, 3H), 2.19-2.15 (m, 2H), 1.94-1.84 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 158.5, 140.5, 136.2, 135.1, 133.6, 131.1, 129.7, 126.5, 126.4, 122.9, 122.2, 121.4, 119.4, 55.8, 49.2, 31.3, 21.9, 18.2; IR (KBr): 3019, 2953, 2924, 2854, 1719, 1599, 1576, 1504, 1455, 1272, 768 cm^{-1}; HRMS: calcd for $C_{21}H_{24}N_2O$ ($M+H^+$) 321.1961; found 321.1966; mp: 108-110 ^{o}C.



8-(2,4-dimethylphenanthridin-6-yl)-1,4-dioxa-8-azaspiro[4.5]decane (**3p**)

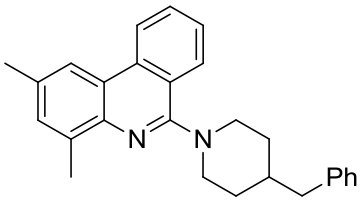
Prepared from 2-isocyano-3,5-dimethyl-1,1'-biphenyl (20.7 mg, 0.10 mmol, 1.0 equiv) and 1,4-dioxa-8-azaspiro[4.5]decan-8-yl benzoate (39.5 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether = 1 : 16) furnished the product **3p** as a light yellow solid (22 mg, 0.064 mmol, 64% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.54 (d, *J* = 8.3 Hz, 1H), 8.18 (d, *J* = 8.2 Hz, 1H), 8.07 (s, 1H), 7.73 (t, *J* = 7.6 Hz, 1H), 7.59 (t, *J* = 7.6 Hz, 1H), 7.34 (s, 1H), 4.04 (s, 4H), 3.63 (t, *J* = 5.4 Hz, 4H), 2.73 (s, 3H), 2.54 (s, 3H), 2.03 (t, *J* = 5.7 Hz, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 158.1, 140.5, 136.2, 135.2, 133.6, 131.1, 129.7, 126.4, 126.3, 122.9, 122.1, 121.3, 119.3, 107.9, 64.5, 49.4, 35.2, 21.9, 18.2; IR (KBr): 3071, 2979, 2918, 2856, 2838, 1611, 1582, 1521, 1454, 1277, 758 cm^{-1}; HRMS: calcd for $C_{22}H_{24}N_2O_2$ ($M+H^+$) 349.1911; found 349.1910; mp: 125-127 ^{o}C.



tert-butyl 4-(2,4-dimethylphenanthridin-6-yl)piperazine-1-carboxylate (**3q**)

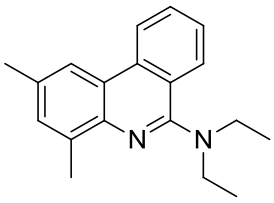
Prepared from 2-isocyano-3,5-dimethyl-1,1'-biphenyl (20.7 mg, 0.10 mmol, 1.0 equiv) and tert-butyl 4-(benzoyloxy)piperazine-1-carboxylate (45.9 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether = 1 : 16) furnished the product **3q** as a light yellow solid (27 mg, 0.070 mmol, 70% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.55 (d, *J* = 8.2 Hz, 1H), 8.19 (d, *J* = 8.1 Hz, 1H), 8.08 (s, 1H), 7.74 (t, *J* = 7.7 Hz, 1H), 7.59 (t, *J* = 7.7 Hz, 1H), 7.35 (s, 1H), 3.74 (t, *J* = 5.1 Hz, 4H), 3.46 (t, *J* = 4.8 Hz, 4H), 2.73 (s, 3H), 2.54 (s, 3H), 1.52 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 157.9, 155.1, 140.3, 136.3, 135.2, 134.1, 131.2, 129.9, 126.5, 126.2, 123.1, 122.3, 121.1, 119.4, 79.9, 51.2, 43.3，28.6,

21.9, 18.2; IR (KBr): 3067, 2961, 2925, 2893, 2845, 1608, 1573, 1520, 1452, 1274, 773 cm^{-1}; HRMS: calcd for $C_{24}H_{29}N_3O_2$ ($M+H^+$) 392.2333; found 392.2332. mp: 139-141 ^oC.



6-(4-benzylpiperidin-1-yl)-2,4-dimethylphenanthridine (**3r**)

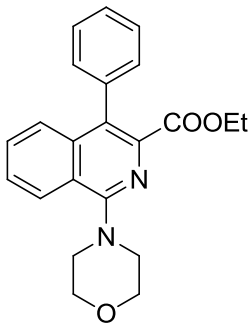
Prepared from 2-isocyano-3,5-dimethyl-1,1'-biphenyl (20.7 mg, 0.10 mmol, 1.0 equiv) and 4-benzylpiperidin-1-yl benzoate (44.3 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether = 1 : 16) furnished the product **3r** as a light yellow solid (26 mg, 0.068 mmol, 68% yield). ^1H NMR (400 MHz, $CDCl_3$): δ 8.53 (d, *J* = 8.2 Hz, 1H), 8.18 (d, *J* = 8.0 Hz, 1H), 8.08 (s, 1H), 7.72 (t, *J* = 7.2 Hz, 1H), 7.58 (t, *J* = 7.2 Hz, 1H), 7.35-7.31 (m, 3H), 7.25-7.23 (m, 3H), 3.95 (d, *J* = 12.9 Hz, 2H), 2.97 (t, *J* = 12.1 Hz, 2H), 2.73 (s, 3H), 2.69 (d, *J* = 6.6 Hz, 2H), 2.54 (s, 3H), 1.87-1.84 (m, 3H), 1.71-1.60 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 158.9, 140.9, 140.6, 136.1, 135.1, 133.5, 131.0, 129.6, 129.3, 128.4, 126.6, 126.2, 125.9, 122.9, 122.1, 121.5, 119.4, 51.9, 43.6, 38.8, 32.6, 21.9, 18.1; IR (KBr): 3070, 2981, 2914, 2849, 2822, 1608, 1573, 1520, 1444, 1284, 774 cm^{-1}; HRMS: calcd for $C_{27}H_{28}N_2$ ($M+H^+$) 381.2325; found 381.2327; mp: 101-103 ^oC.



N,*N*-diethyl-2,4-dimethylphenanthridin-6-amine (**3s**)

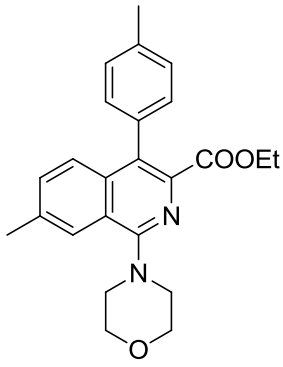
Prepared from 2-isocyano-3,5-dimethyl-1,1'-biphenyl (20.7 mg, 0.10 mmol, 1.0 equiv) and *O*-benzoyl-*N*,*N*-diethylhydroxylamine (28.9 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether = 1 : 60) furnished the product **3s** as a light yellow oil (13 mg, 0.047 mmol, 47% yield). ^1H NMR (400 MHz, $CDCl_3$): δ 8.54 (d, *J* = 8.2 Hz, 1H), 8.21 (d, *J*

= 8.2 Hz, 1H), 8.08 (s, 1H), 7.72 (t, *J* = 8.2 Hz, 1H), 7.57 (t, *J* = 7.1 Hz, 1H), 7.35 (s, 1H), 3.53 (q, *J* = 7.0 Hz, 4H), 2.74 (s, 3H), 2.54 (s, 3H), 1.29 (t, *J* = 7.0 Hz, 6H), ; ^{13}C NMR (125 MHz, $CDCl_3$): δ 157.6, 140.6, 136.0, 135.2, 133.2, 130.9, 129.5, 126.5, 126.1, 122.9, 122.5, 121.8, 119.3, 46.1, 21.9, 18.2, 13.2; IR (KBr): 3072, 2965, 2921, 2852, 1728, 1611, 1574, 1520, 1455, 1288, 773 cm^{-1}; HRMS: calcd for $C_{19}H_{22}N_2$ ($M+H^+$) 279.1856; found 279.1852.



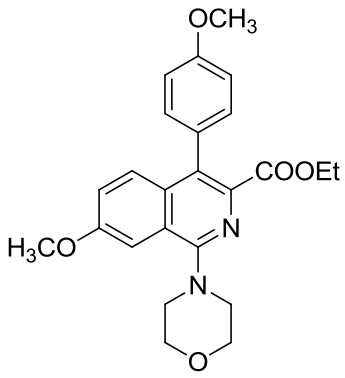
Ethyl 1-morpholino-4-phenylisoquinoline-3-carboxylate (**5a**)

Prepared from ethyl 2-isocyano-3,3-diphenylacrylate (27.7 mg, 0.10 mmol, 1.0 equiv) and morpholino benzoate (31.1 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether = 1 : 8) furnished the product **5a** as a light yellow solid (22 mg, 0.061 mmol, 61% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.17-8.14 (m, 1H), 7.58-7.57 (m, 3H), 7.46-7.42 (m, 3H), 7.34-7.31 (m, 2H), 4.07 (q, *J* = 7.1 Hz, 2H), 3.99 (t, *J* = 4.2 Hz, 4H), 3.53 (t, *J* = 4.2 Hz, 4H), 0.93 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 167.9, 160.4, 140.7, 137.9, 136.7, 130.4, 130.2, 128.3, 128.1, 127.8, 127.3, 127.1, 125.5, 121.9, 67.2, 61.1, 51.9, 13.7; IR (KBr): 3010, 2953, 2924, 2854, 1719, 1599, 1576, 1504, 1455, 1272, 768 cm^{-1}; HRMS: calcd for $C_{22}H_{22}N_2O_3$ ($M+H^+$) 363.1703; found 363.1700; mp: 189-191 ^oC.



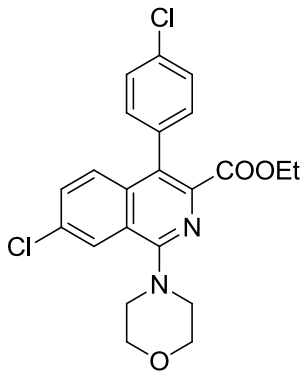
Ethyl 7-methyl-1-morpholino-4-(*p*-tolyl)isoquinoline-3-carboxylate (**5b**)

Prepared from ethyl 2-isocyano-3,3-di-p-tolylacrylate (30.5 mg, 0.10 mmol, 1.0 equiv) and morpholino benzoate (31.1 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether = 1 : 8) furnished the product **5b** as a light yellow solid (21 mg, 0.054 mmol, 54% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 (s, 1H), 7.42 (d, *J* = 8.6 Hz, 1H), 7.30 (d, *J* = 8.6 Hz, 1H), 7.18 (d, *J* = 8.0 Hz, 2H), 7.11 (d, *J* = 8.0 Hz, 2H), 4.01 (q, *J* = 7.2 Hz, 2H), 3.91 (t, *J* = 4.5 Hz, 4H), 3.41 (t, *J* = 4.7 Hz, 4H), 2.46 (s, 3H), 2.35 (s, 3H), 0.89 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.1, 159.7, 139.9, 137.4, 137.3, 136.2, 133.8, 132.2, 130.1, 128.9, 128.5, 127.1, 124.4, 122.2, 67.2, 61.1, 51.9, 22.1, 21.4, 13.8; IR (KBr): 3025, 2977, 2921, 2882, 2850, 1646, 1573, 1518, 1441, 1276, 739 cm^{-1}; HRMS: calcd for $C_{24}H_{26}N_2O_3$ ($M+H^+$) 391.2016; found 391.2019; mp: 105-107 ^{o}C.



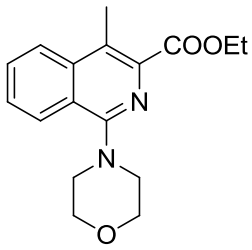
Ethyl 7-methoxy-4-(4-methoxyphenyl)-1-morpholinoisoquinoline-3-carboxylate (**5c**)

Prepared from ethyl 3,3-bis(4-chlorophenyl)-2-isocyanoacrylate (33.7 mg, 0.10 mmol, 1.0 equiv) and morpholino benzoate (31.05 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether = 1 : 8) furnished the product **5c** as a light yellow solid (26 mg, 0.062 mmol, 62% yield).^{1}H NMR (400 MHz, $CDCl_3$): δ 7.53 (d, *J* = 9.2 Hz, 1H), 7.45 (s, 1H), 7.22 (d, *J* = 8.5 Hz, 2H), 6.98 (d, *J* = 8.7 Hz, 2H), 4.10 (q, *J* = 7.1 Hz, 2H), 3.99 (t, *J* = 4.4 Hz, 4H), 3.95 (s, 3H), 3.87 (s, 3H), 3.48 (t, *J* = 4.6 Hz, 4H), 1.00 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.2, 159.4, 159.1, 158.8, 139.0, 133.2, 131.3, 128.9, 128.6, 123.6, 121.9, 113.8, 104.4, 67.2, 61.0, 55.6, 55.5, 51.6, 13.9; IR (KBr): 3030, 2982, 2966, 2920, 2846, 1646, 1582, 1517, 1444, 1272, 835 cm^{-1}; HRMS: calcd for $C_{24}H_{26}N_2O_5$ ($M+H^+$) 423.1914; found 423.1912; mp: 133-135 ^{o}C.



Ethyl 7-chloro-4-(4-chlorophenyl)-1-morpholinoisoquinoline-3-carboxylate (**5d**)

Prepared from ethyl 3,3-bis(4-chlorophenyl)-2-isocyanoacrylate (34.6 mg, 0.10 mmol, 1.0 equiv) and morpholino benzoate (31.1 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether = 1 : 8) furnished the product **5d** as a light yellow solid (16 mg, 0.037 mmol, 37% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.10 (s, 1H), 7.52 (d, *J* = 9.0 Hz, 1H), 7.48-7.43 (m, 3H), 7.24 (d, *J* = 8.4 Hz, 2H), 4.11 (q, *J* = 7.2 Hz, 2H), 3.99 (t, *J* = 4.5 Hz, 4H), 3.51 (t, *J* = 4.6 Hz, 4H), 1.02 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 167.3, 159.7, 140.9, 136.2, 134.7, 134.2, 133.7, 131.6, 131.3, 128.7, 128.6, 126.7, 124.7, 122.7, 67.0, 61.4, 51.8, 13.9; IR (KBr): 3020, 2961, 2920, 2851, 1736, 1573, 1562, 1543, 1445, 1267, 782 cm^{-1}; HRMS: calcd for $C_{22}H_{20}Cl_2N_2O_3$ ($M+H^+$) 431.0924; found 431.0928; mp: 89-91 ^{o}C.



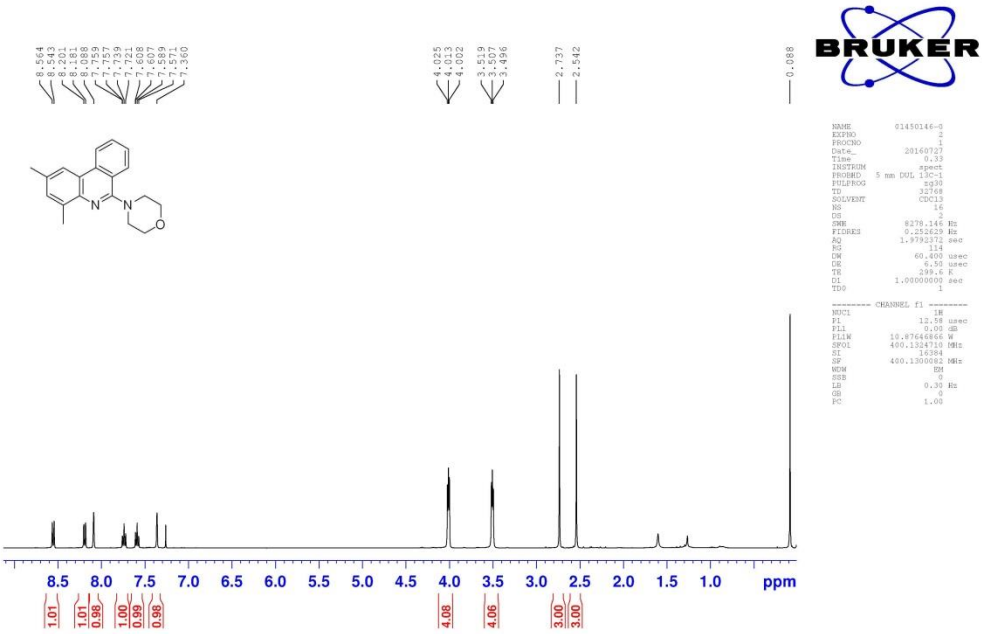
Ethyl 4-methyl-1-morpholinoisoquinoline-3-carboxylate (**5e**)

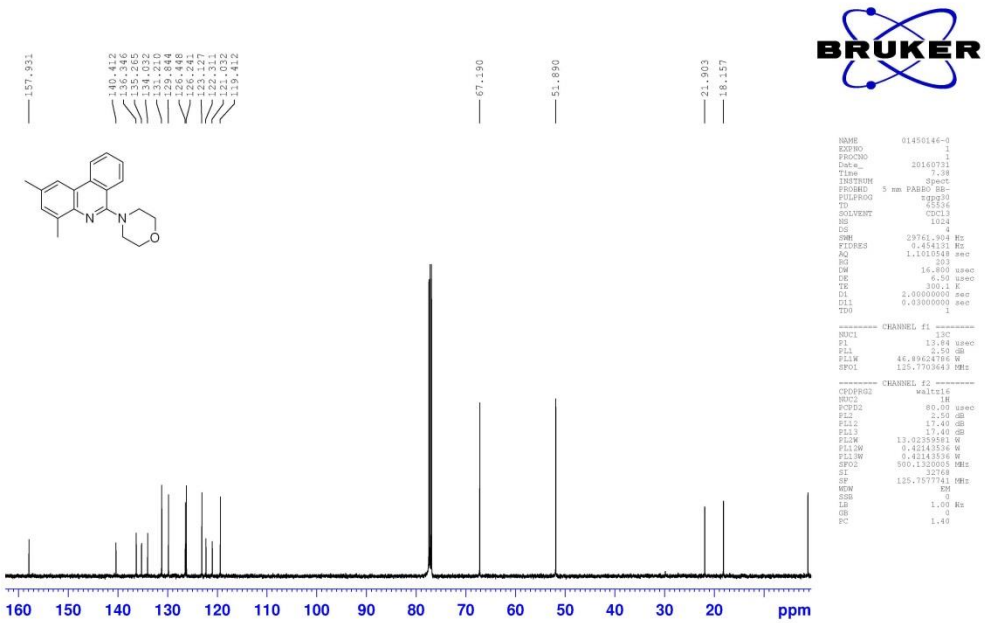
Prepared from (*Z*)-ethyl 2-isocyano-3-phenylbut-2-enoate (21.5 mg, 0.10 mmol, 1.0 equiv) and morpholino benzoate (31.05 mg, 0.15 mmol, 1.5 equiv) according to the general procedure. Column chromatography purification (EtOAc : petroleum ether = 1 : 8) furnished the product **5e** as a light yellow oil (10 mg, 0.031 mmol, 31% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.14 (d, *J* = 8.1 Hz, 1H), 8.04 (d, *J* = 8.4 Hz, 1H), 7.71 (t, *J* = 8.4 Hz, 1H), 7.59 (t, *J* = 8.1 Hz, 1H), 4.46 (q, *J* = 7.1 Hz, 2H), 3.96 (t, *J* = 4.5 Hz, 4H), 3.42 (t, *J* = 4.7 Hz, 4H), 2.69 (s, 3H), 1.44 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR

(125 MHz, $CDCl_3$): δ 168.3, 159.2, 140.3, 138.2, 130.1, 127.2, 125.8, 124.9, 122.9, 122.1, 67.2, 61.4, 51.9, 14.4, 14.0; IR (KBr): 3073, 2959, 2921, 2850, 1721, 1615, 1579, 1504, 1441, 1273, 767 cm^{-1}; HRMS: calcd for $C_{17}H_{20}N_2O_3$ ($M+H^+$) 301.1547; found 301.1549.

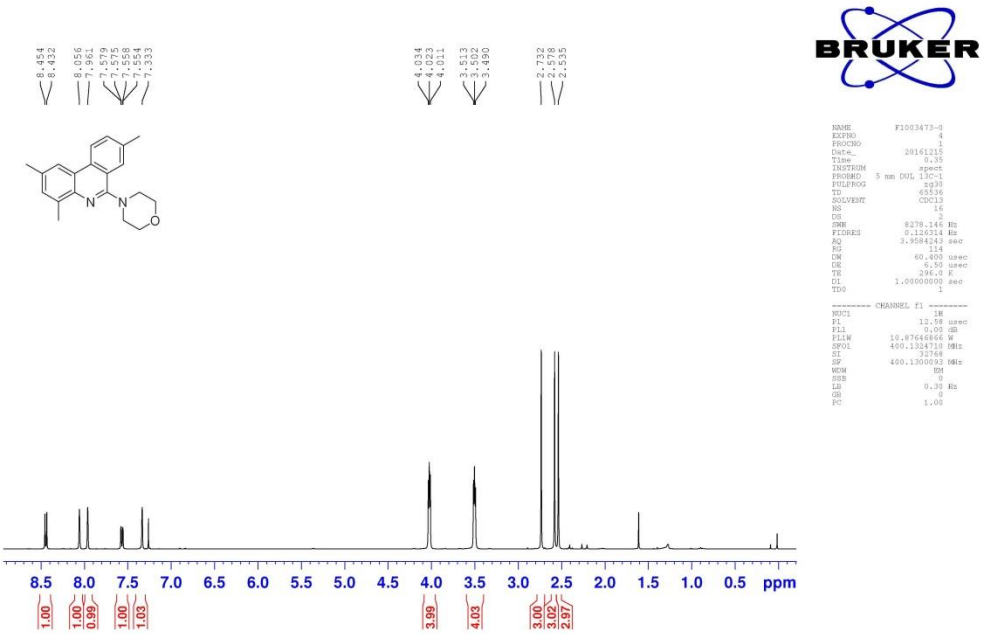
IV. Copies of ^{1}H and ^{13}C NMR Spectra

3a





3b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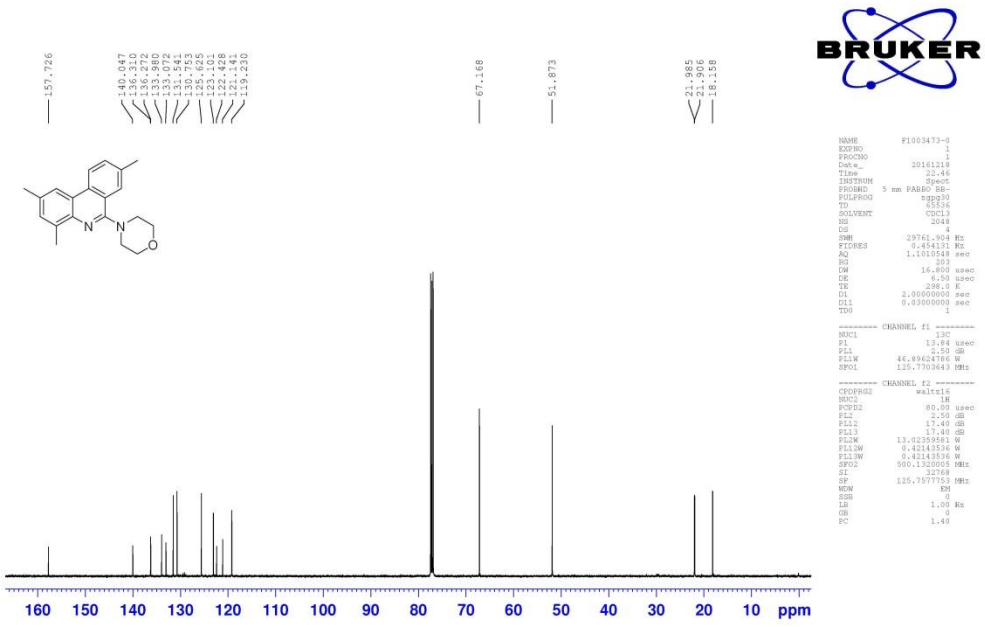
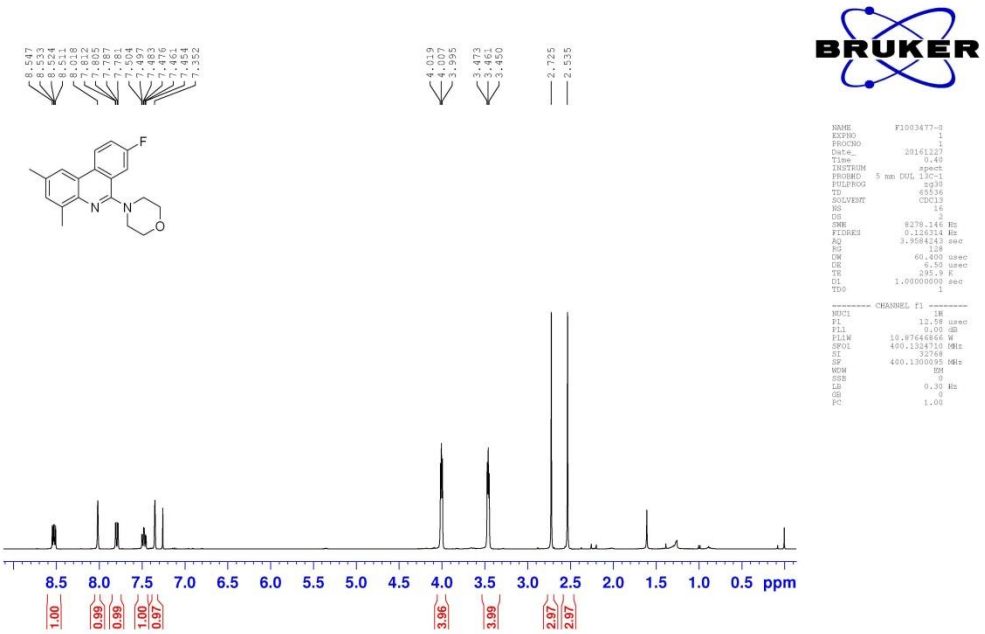
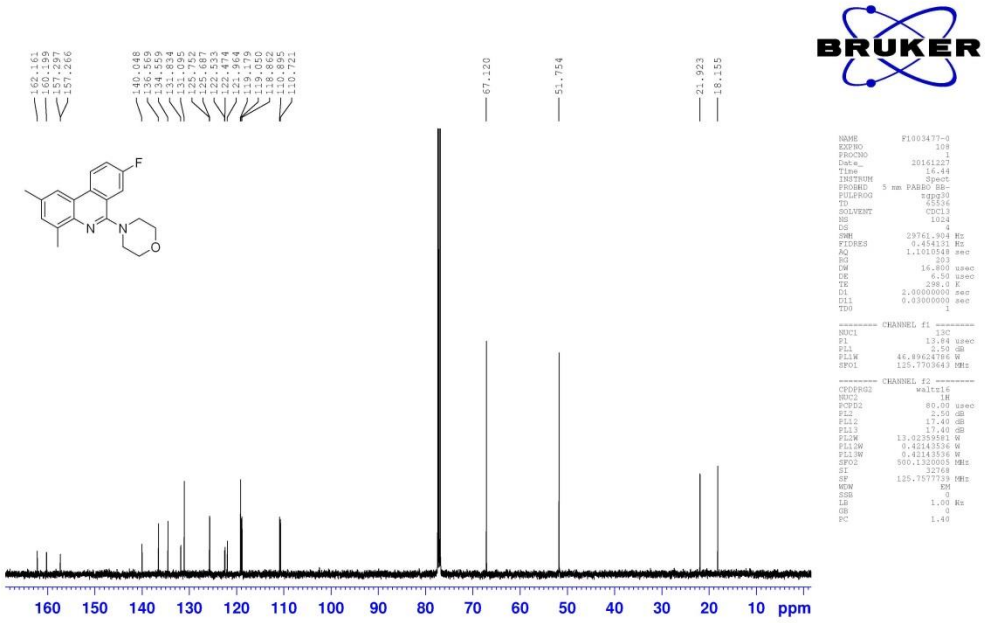
8.432
8.056
7.961
7.579
7.575
7.558
7.554
7.333
4.034
4.023
4.011
3.513
3.502
3.490
2.732
2.578
2.535
BRUKER
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
1.00
0.99
1.00
1.03
3.99
4.03
3.00
3.02
2.97

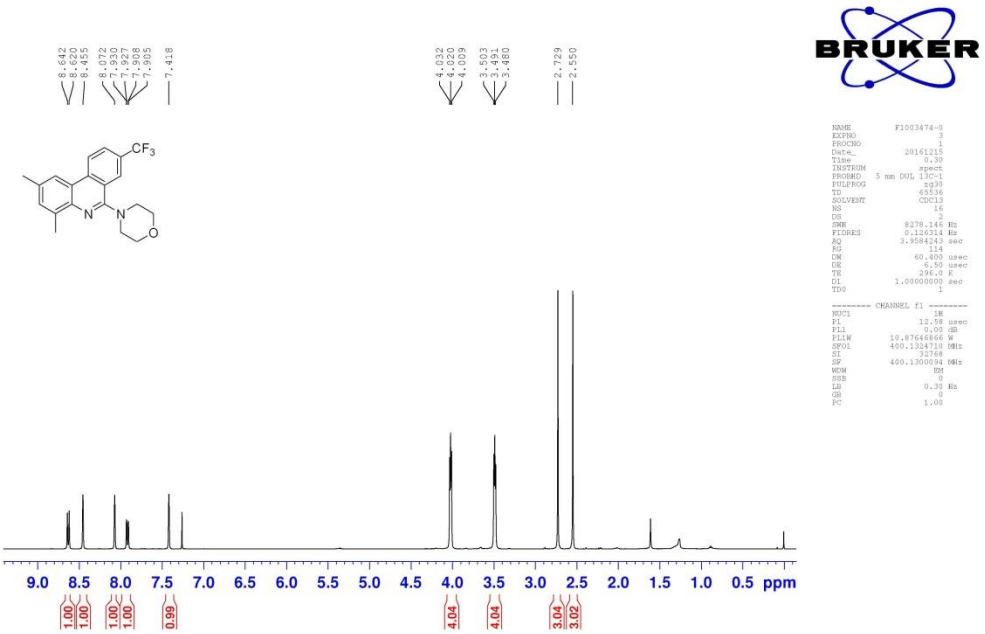

157.726
140.047
136.310
136.272
133.980
133.872
131.541
130.753
125.625
123.101
122.428
121.141
119.230
67.168
51.873
21.985
21.906
18.158
BRUKER
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

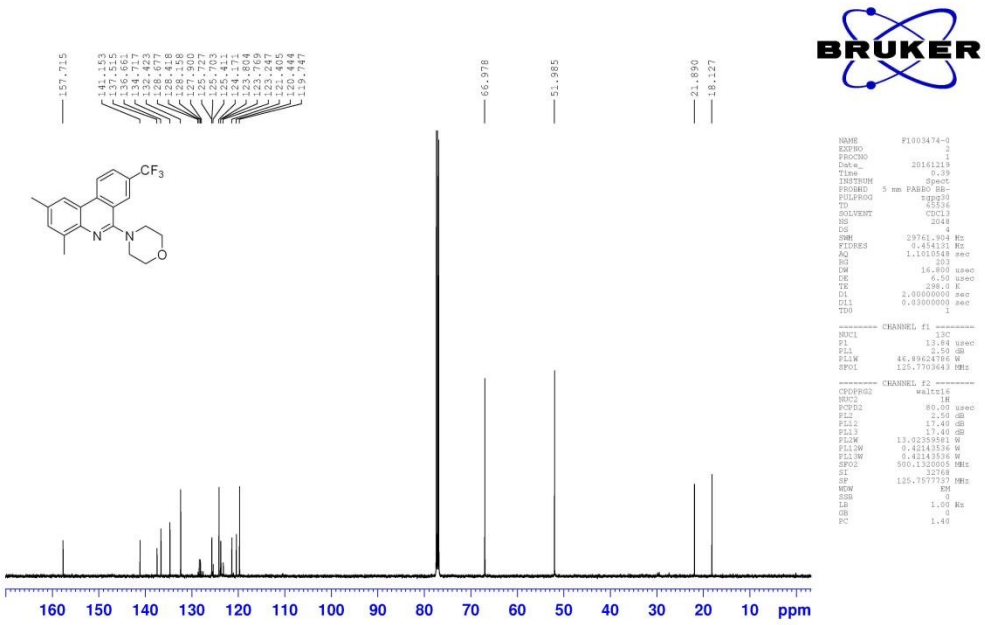
3c


8.547
8.543
8.524
8.511
8.018
7.802
7.795
7.787
7.781
7.504
7.493
7.483
7.476
7.461
7.454
7.352
4.019
4.007
3.995
3.473
3.461
3.450
2.725
2.535
F
N
O
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
0.99
0.99
1.00
0.97
3.96
3.99
2.97
2.97

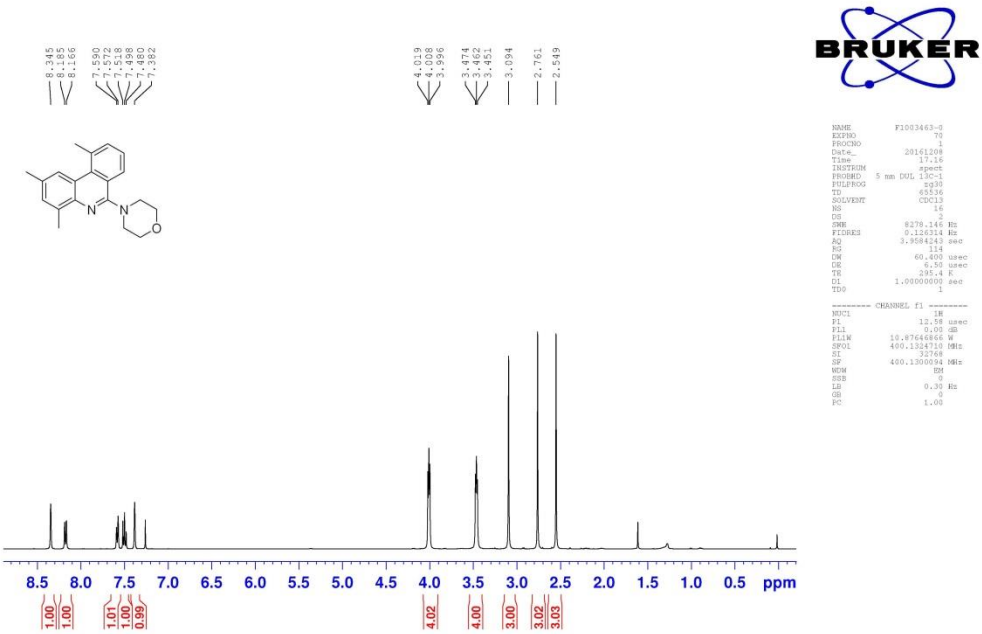

162.161
160.199
157.297
157.266
140.048
136.569
134.559
131.034
130.885
125.752
125.687
122.533
122.474
121.464
119.179
119.050
118.862
110.995
110.721
67.120
51.754
21.923
18.155
F
N
O
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

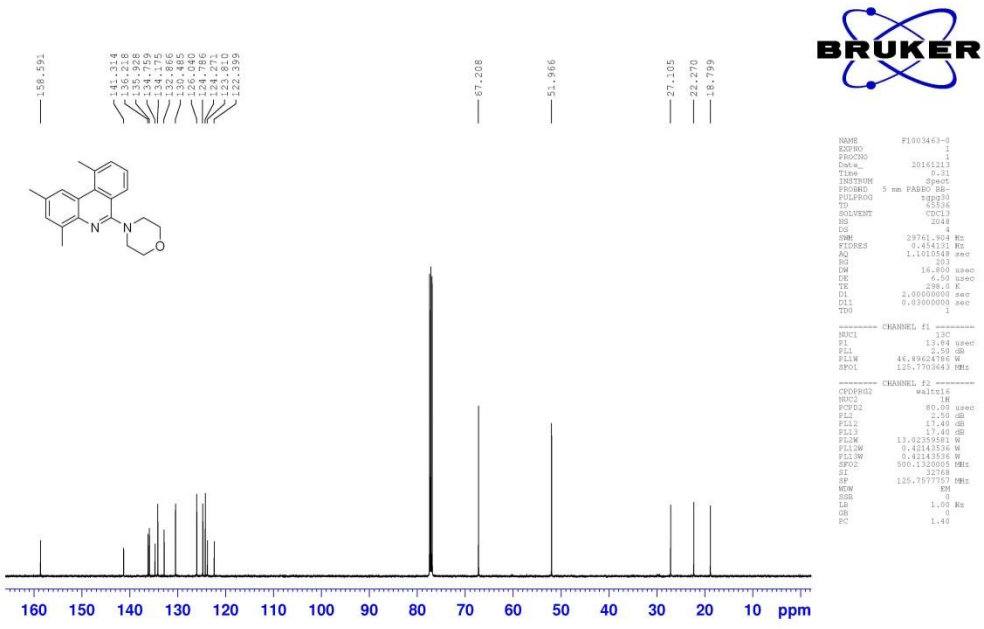
3d


8.642
8.620
8.455
8.072
7.930
7.927
7.908
7.905
7.418
4.032
4.020
4.009
3.503
3.491
3.480
2.729
2.550
CF3
N
N
O
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
1.00
1.00
1.00
0.99
4.04
4.04
3.04
3.02

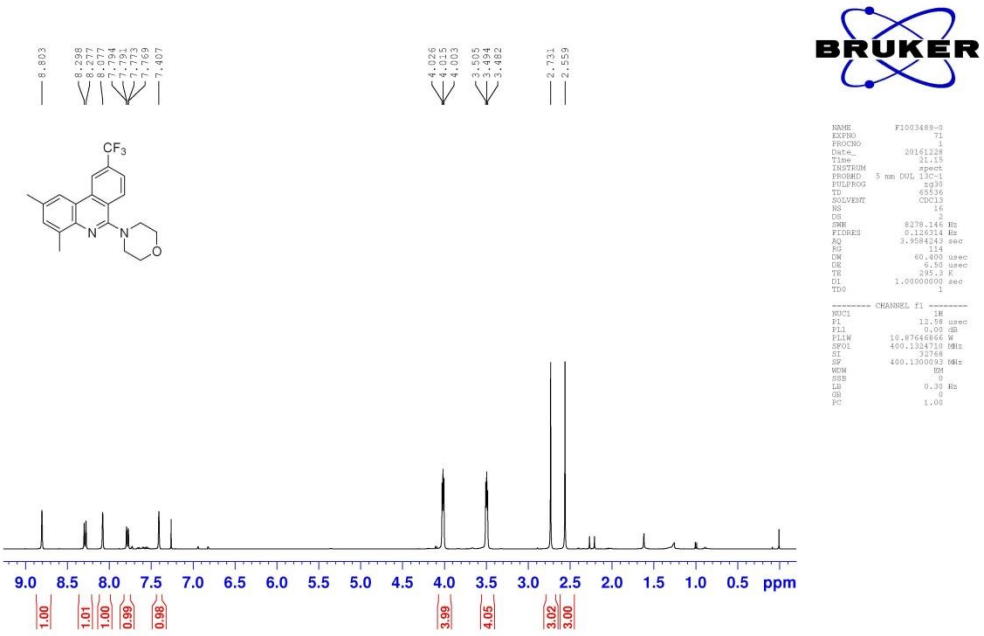

157.715
141.153
137.615
136.161
134.717
132.423
128.677
128.418
128.158
127.900
125.727
125.703
124.471
123.804
123.769
123.247
120.405
120.404
119.747
66.978
51.985
21.890
18.127
CF3
N
N
O
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

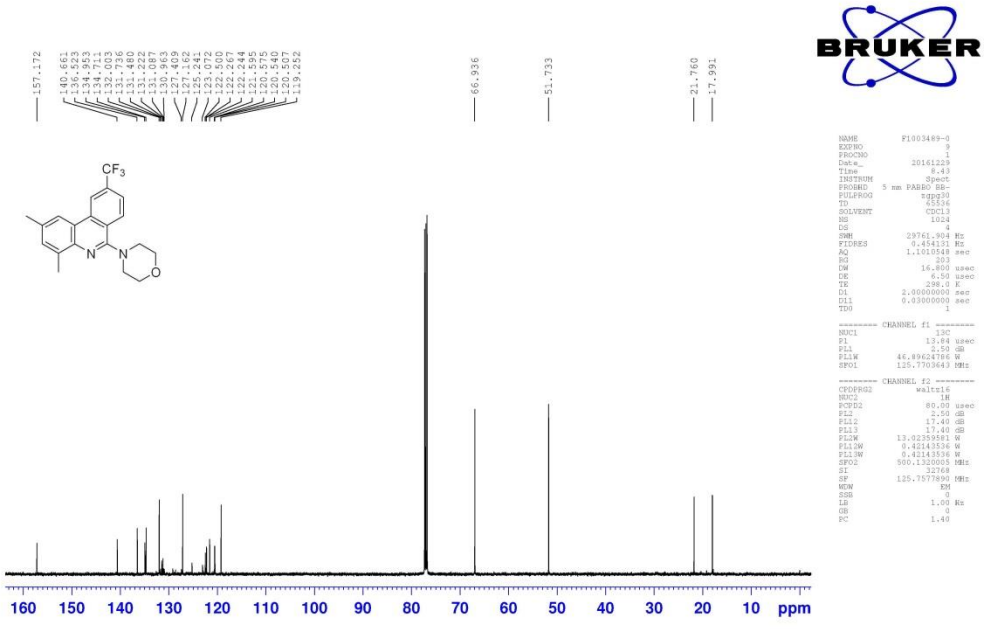
3e


8.345
8.185
8.166
7.590
7.572
7.518
7.498
7.480
7.382
4.019
4.008
3.996
3.474
3.462
3.451
3.094
2.761
2.549
BRUKER
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
1.00
1.01
1.00
0.99
4.02
4.00
3.00
3.02
3.03

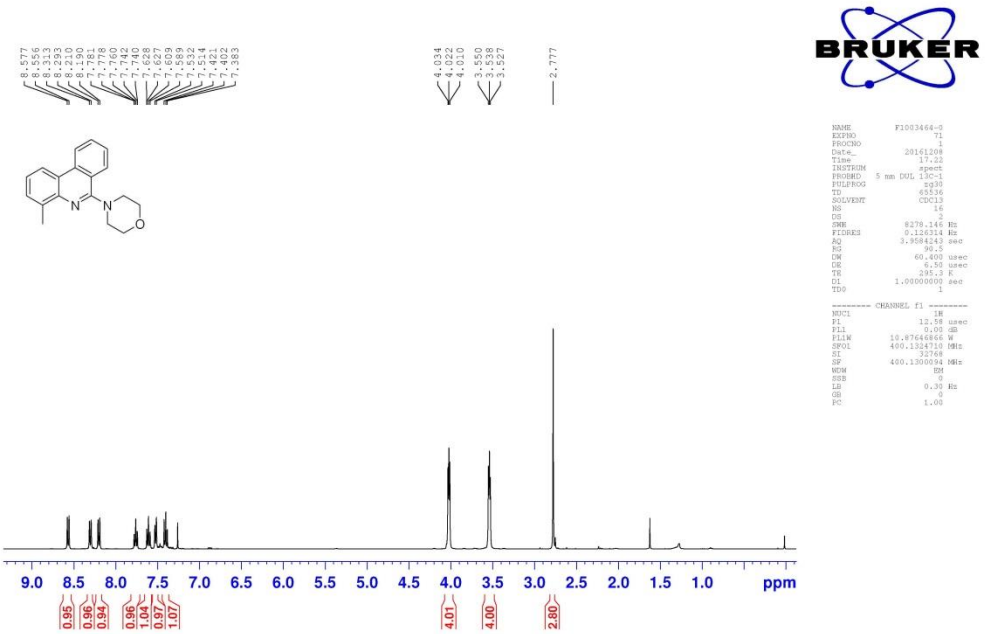

158.591
145.314
139.218
135.928
134.759
134.175
132.666
130.685
126.040
124.786
124.271
123.610
122.399
67.208
51.966
27.105
22.270
18.799
BRUKER
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

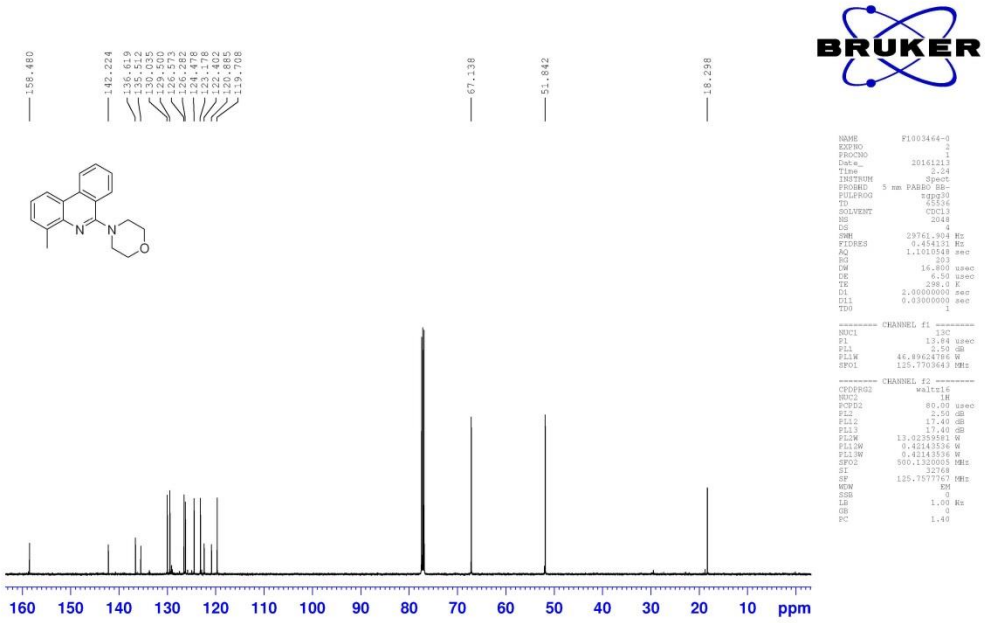
3f


8.803
8.298
8.277
8.077
7.794
7.781
7.773
7.769
7.407
4.026
4.015
4.003
3.505
3.494
3.482
2.731
2.559
CF3
N
O
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
1.01
1.00
0.99
0.98
3.99
4.05
3.02
3.00

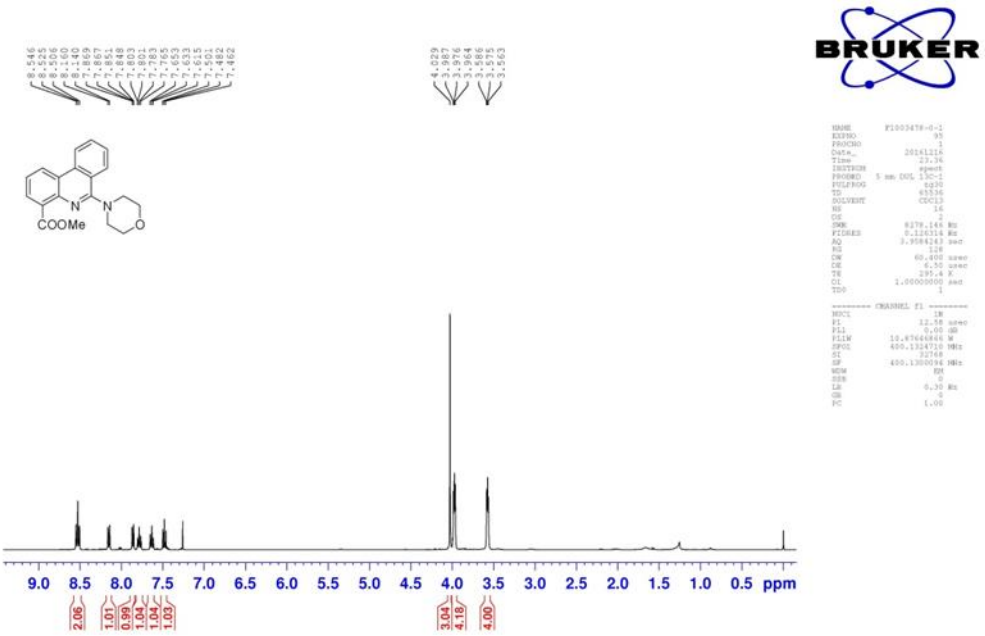

157.172
140.661
136.523
134.953
134.711
132.003
131.736
131.480
131.222
131.087
130.963
127.409
127.162
125.841
125.572
122.500
122.267
122.244
121.595
120.955
120.540
120.507
119.252
66.936
51.733
21.760
17.991
CF3
N
O
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

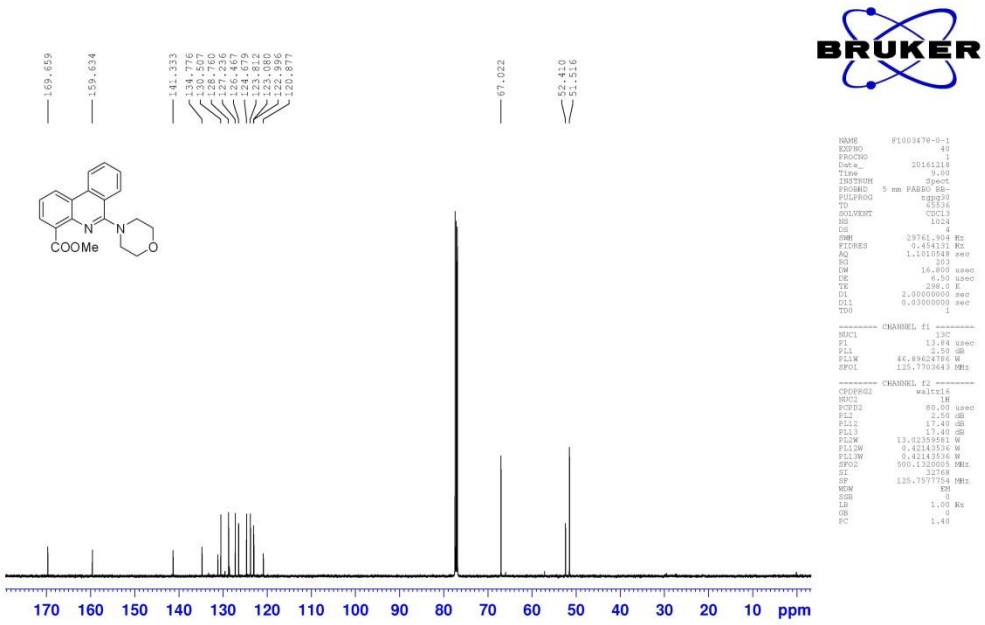
3g


8.577
8.556
8.313
8.293
8.210
8.190
7.781
7.778
7.760
7.742
7.740
7.628
7.627
7.609
7.589
7.532
7.514
7.421
7.402
7.383
4.034
4.022
4.010
3.550
3.538
3.527
2.777
BRUKER
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
0.95
0.96
0.94
0.96
1.04
0.97
1.07
4.01
4.00
2.80

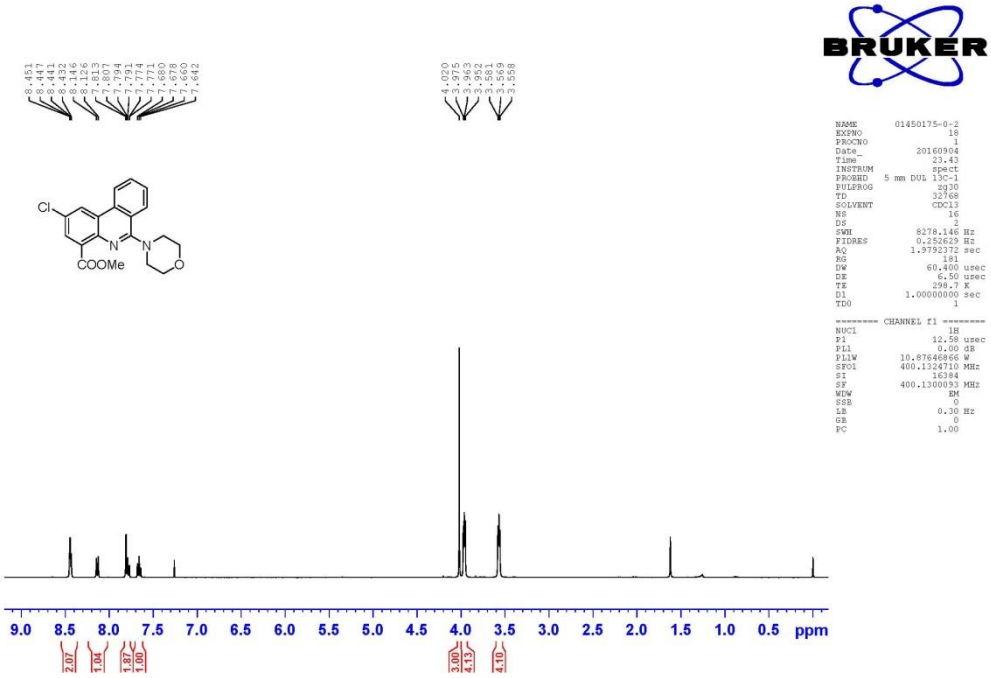

158.480
142.224
136.619
135.112
130.035
129.500
126.573
126.482
124.478
123.178
122.402
120.885
119.708
67.138
51.842
18.298
BRUKER
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

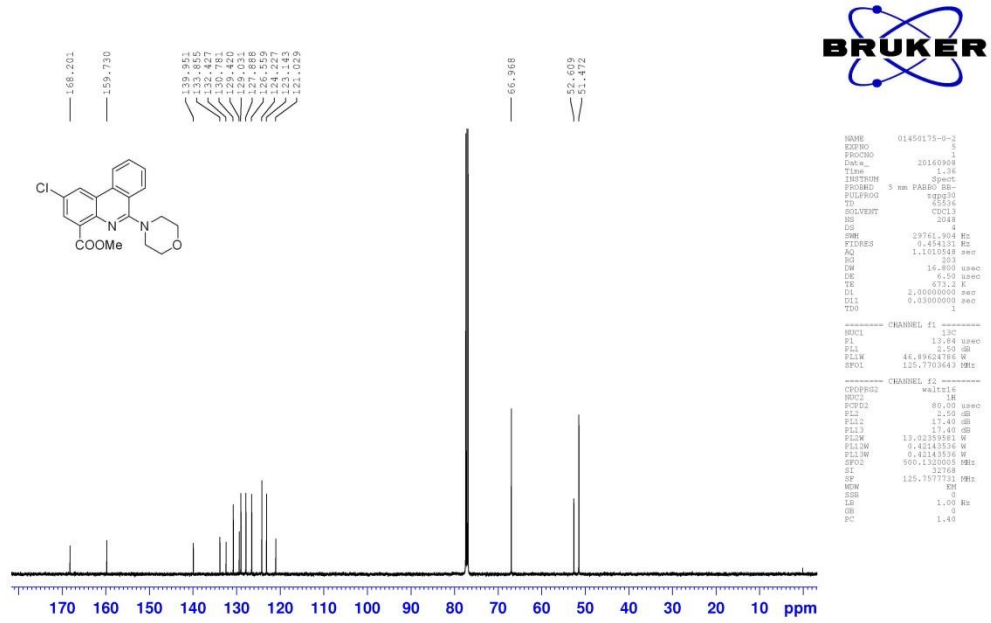
3h


BRUKER
COOMe
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

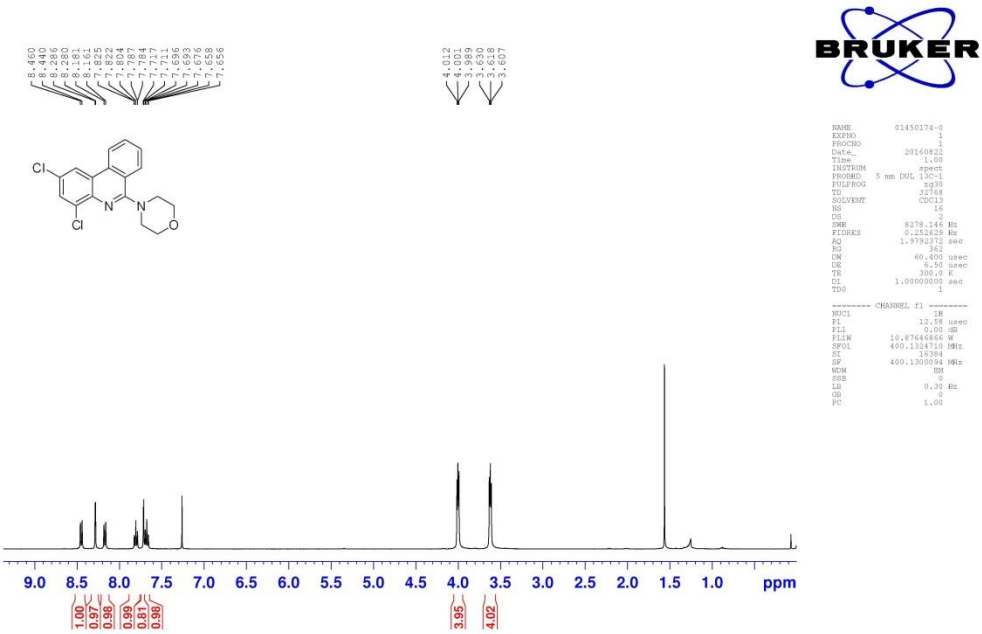

BRUKER
COOMe
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

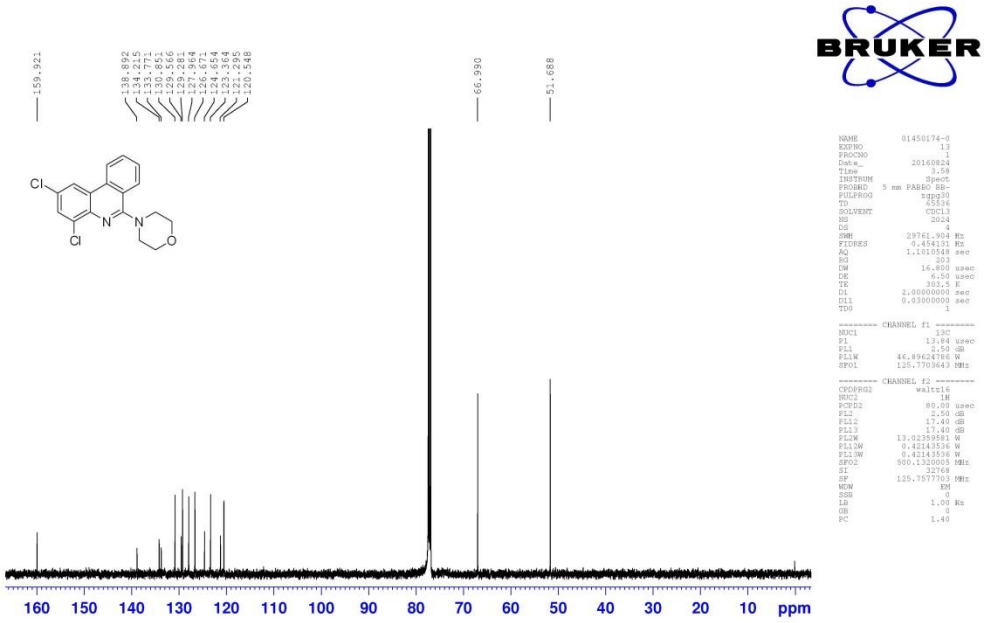
3i


8.451
8.447
8.441
8.432
8.416
8.126
7.813
7.807
7.794
7.791
7.774
7.771
7.680
7.678
7.660
7.642
4.020
3.975
3.963
3.952
3.581
3.569
3.558
Cl
N
N
O
COOMe
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.07
1.04
1.87
1.00
3.00
4.13
4.10

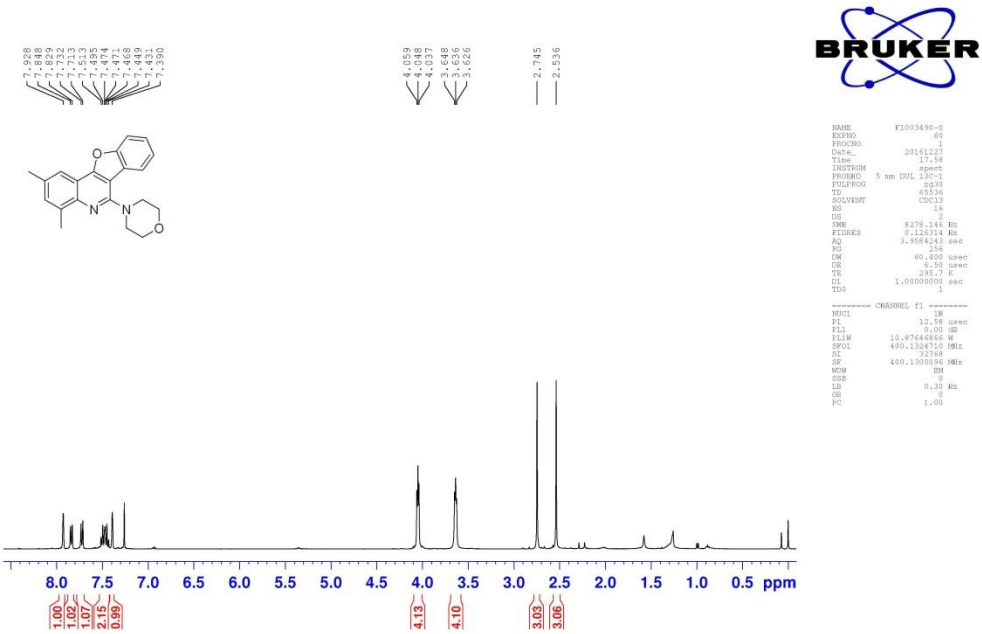

168.201
159.730
139.851
133.855
132.427
130.781
129.421
128.810
127.888
126.559
124.227
123.483
121.089
66.988
52.609
51.472
Cl
N
N
O
COOMe
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

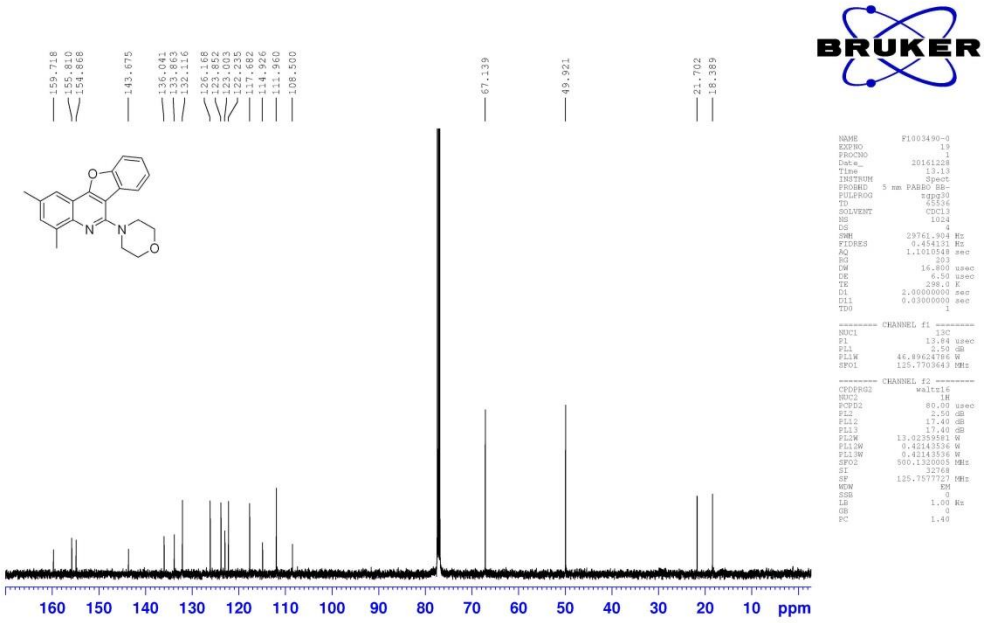
3j


8.460
8.440
8.286
8.280
8.161
8.156
7.825
7.804
7.787
7.784
7.711
7.696
7.693
7.676
7.668
7.668
4.012
4.001
3.989
3.630
3.618
3.607
Cl
N
O
Cl
BRUKER
NAME 01450174-0
EXPNO 1
PROCNO 1
Date_ 20160822
Time 1.00
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 362
DW 60.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 16384
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.00
0.97
0.98
0.99
0.81
0.98
3.95
4.02

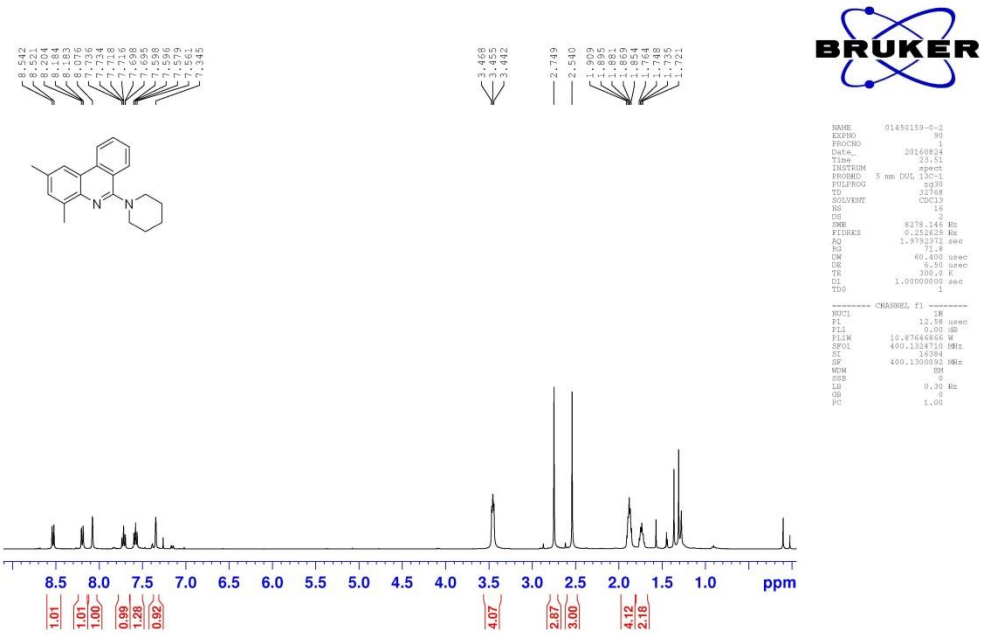

159.921
138.892
134.215
133.771
130.851
129.566
129.081
127.964
126.671
124.654
123.364
121.245
120.548
66.990
51.688
Cl
N
O
Cl
BRUKER
NAME 01450174-0
EXPNO 13
PROCNO 1
Date_ 20160824
Time 3.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 303.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 13.04 usec
PL1 2.59 dB
PL1W 46.89624786 W
SFO1 125.7709643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577703 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

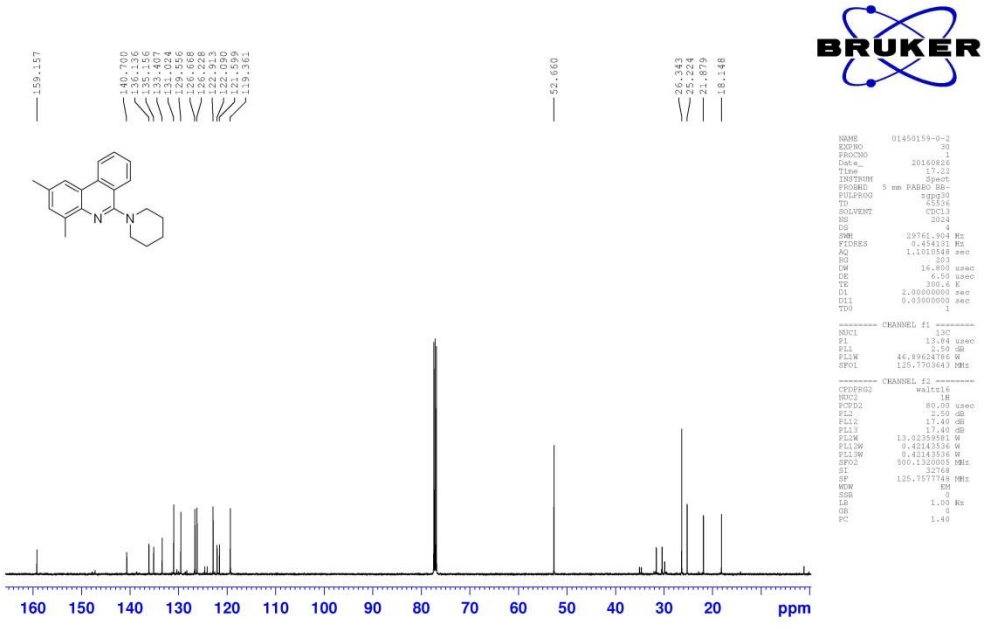
3k


7.908
7.848
7.829
7.732
7.713
7.513
7.495
7.474
7.471
7.468
7.449
7.431
7.390
4.059
4.038
4.017
3.648
3.636
3.626
2.745
2.536
BRUKER
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
1.02
1.07
2.15
0.99
4.13
4.10
3.03
3.06

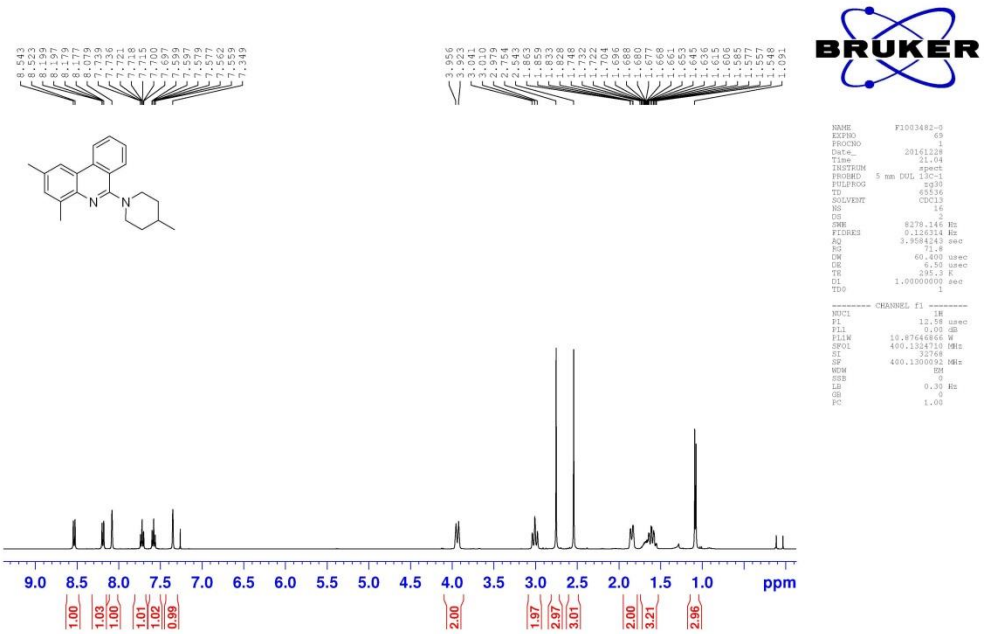

159.718
155.810
154.868
143.675
136.041
133.863
132.116
126.168
123.852
123.003
122.685
121.782
114.926
111.960
108.500
67.139
49.921
21.702
18.389
BRUKER
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

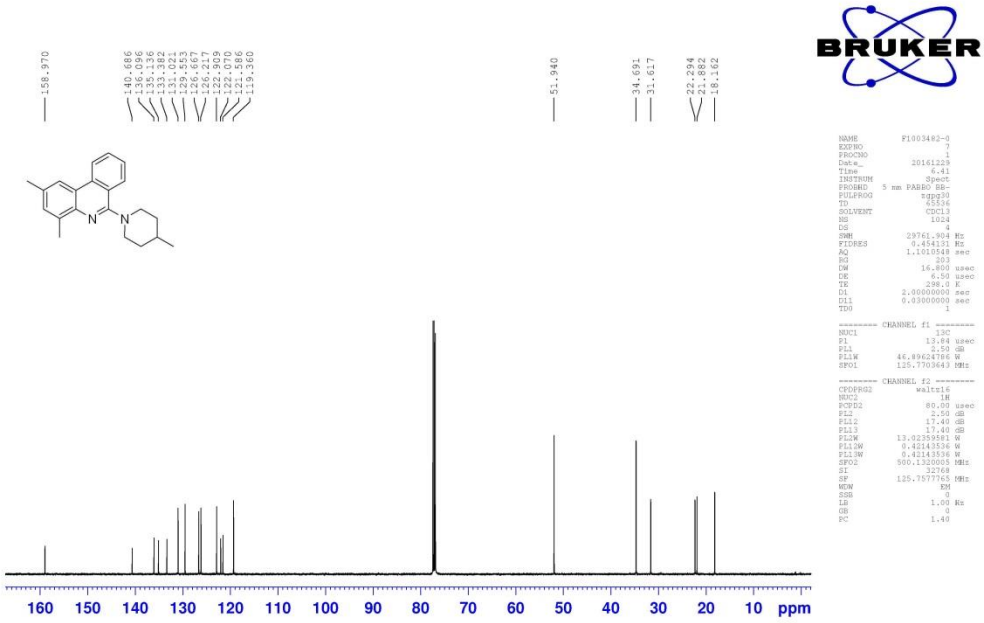
3l


8.542
8.521
8.204
8.184
8.183
8.076
7.736
7.734
7.718
7.716
7.698
7.695
7.598
7.596
7.579
7.561
7.345
3.468
3.455
3.442
2.749
2.540
1.909
1.885
1.881
1.869
1.854
1.764
1.738
1.735
1.721
BRUKER
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.01 1.01 1.00 0.99 1.28 0.92 4.07 2.87 3.00 4.12 2.18


159.157
140.700
136.136
135.156
133.407
131.454
130.576
126.668
126.228
122.913
122.599
120.090
119.361
52.660
26.343
25.224
21.879
18.148
BRUKER
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

3m


8.543
8.523
8.199
8.197
8.179
8.177
8.079
7.739
7.736
7.721
7.718
7.715
7.690
7.687
7.599
7.597
7.579
7.577
7.562
7.559
7.349
3.956
3.953
3.041
3.010
2.979
2.764
2.743
1.863
1.859
1.833
1.808
1.748
1.732
1.722
1.704
1.686
1.680
1.677
1.668
1.661
1.653
1.645
1.636
1.615
1.585
1.577
1.557
1.548
1.091
BRUKER
NAME F1003482-0
EXPNO 69
PROCNO 1
Date_ 20161228
Time 21.04
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 71.8
DW 60.400 usec
DE 6.50 usec
TE 295.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.00
1.03
1.00
1.01
1.02
0.99
2.00
1.97
2.97
3.01
2.00
3.21
2.96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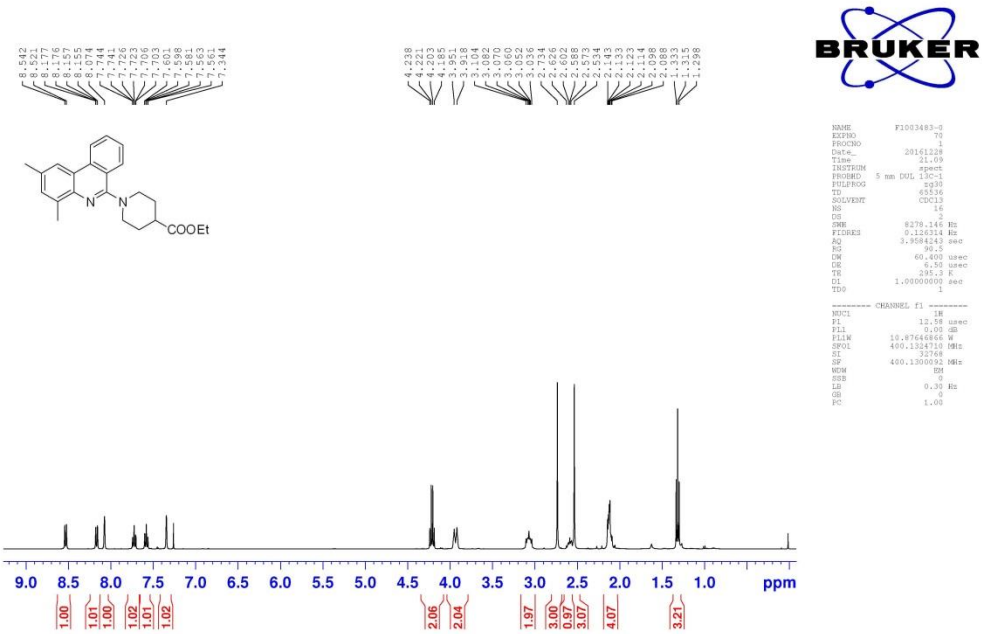
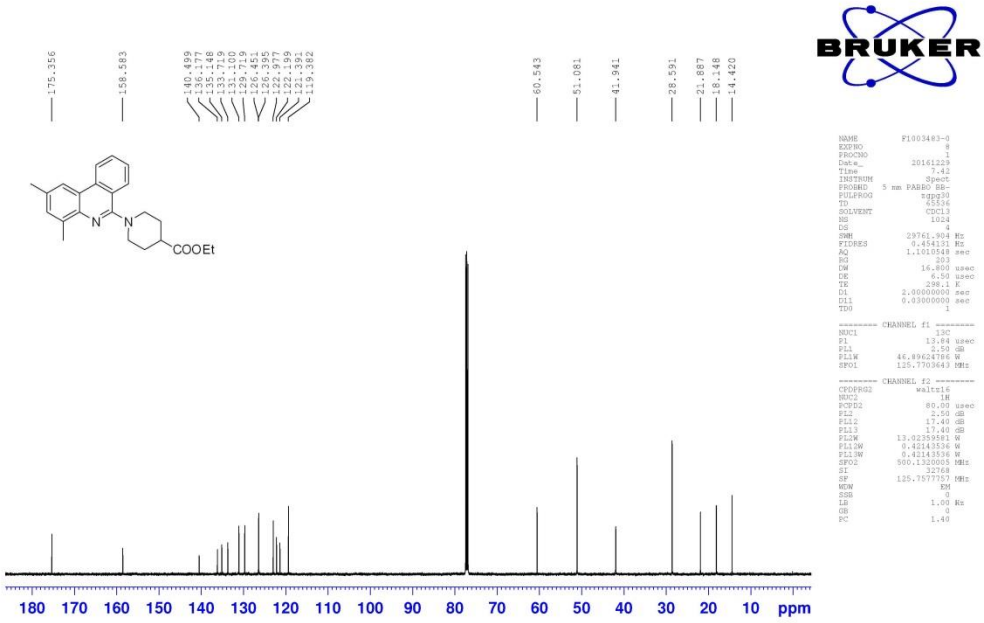
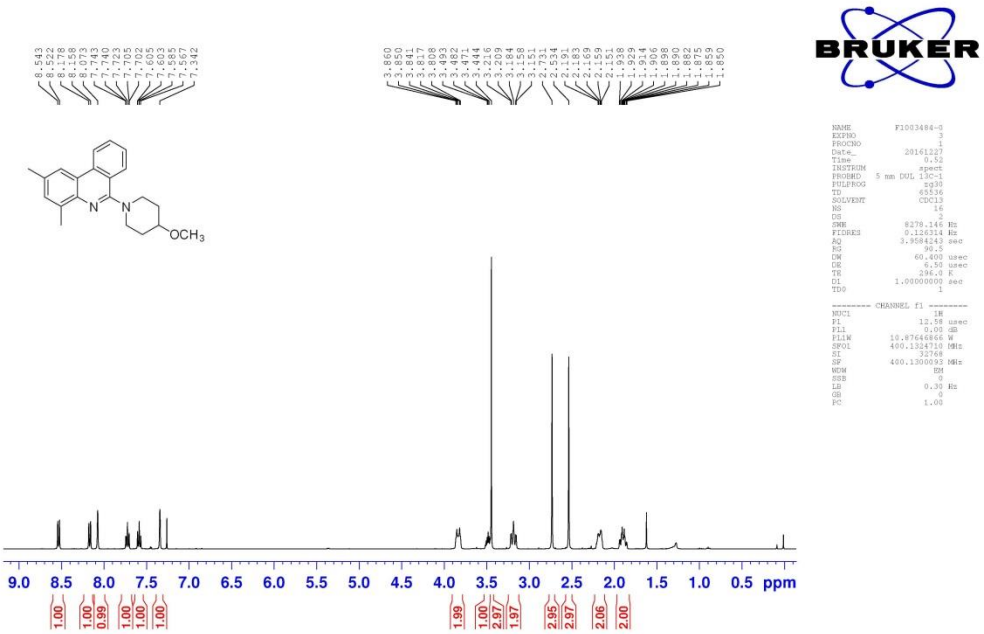
140.686
135.996
135.130
133.382
131.021
129.563
128.467
126.217
122.909
122.070
121.586
119.360
51.940
34.691
31.617
22.294
21.882
18.162
BRUKER
NAME F1003482-0
EXPNO 7
PROCNO 1
Date_ 20161229
Time 6.41
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 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 13.04 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7709643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577765 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

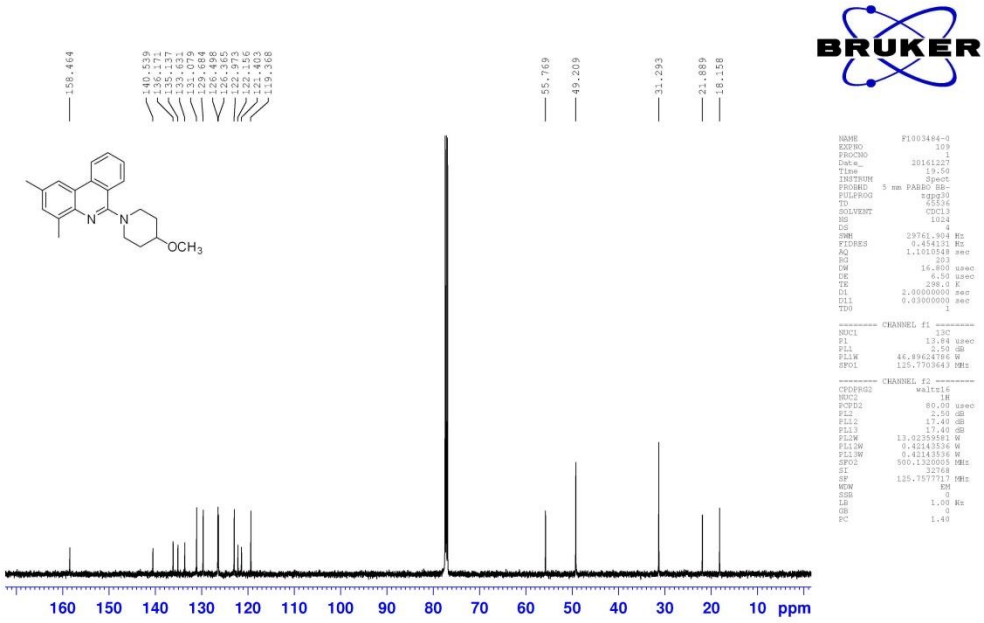
3n


8.542
8.521
8.177
8.176
8.167
8.155
8.074
7.744
7.741
7.726
7.723
7.706
7.703
7.701
7.698
7.581
7.563
7.561
7.344
4.238
4.221
4.203
4.185
3.961
3.918
3.084
3.082
3.070
3.060
3.052
3.036
2.734
2.636
2.620
2.602
2.588
2.573
2.554
2.143
2.133
2.123
2.114
2.098
2.088
1.333
1.315
1.298
COOEt
BRUKER
NAME F1003483-0
EXPNO 70
PROCNO 1
Date_ 20161228
Time 21.09
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 90.5
DW 60.400 usec
DE 6.50 usec
TE 295.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PLIW 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.00 1.01 1.00 1.02 1.01 1.02 2.06 2.04 1.97 3.00 0.97 3.07 4.07 3.21

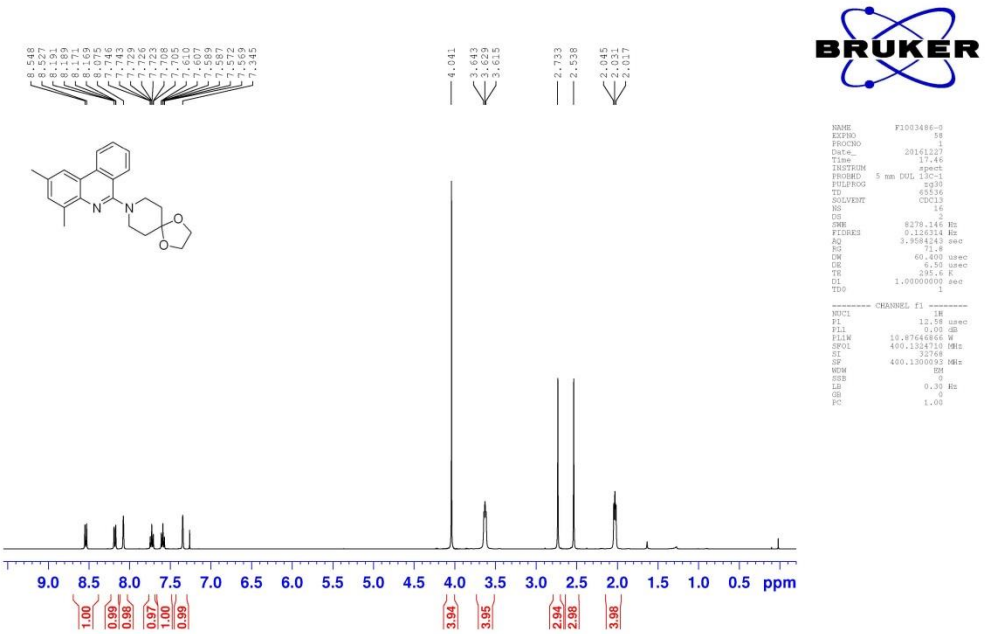

175.356
158.583
140.499
136.177
135.148
133.178
131.100
129.719
126.451
125.897
122.199
121.391
119.382
60.543
51.081
41.941
28.591
21.887
18.148
14.420
COOEt
BRUKER
NAME F1003483-0
EXPNO 8
PROCNO 1
Date_ 20161229
Time 7.42
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 13.04 usec
PL1 2.59 dB
PL1W 46.89624786 W
SFO1 125.7709643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577757 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

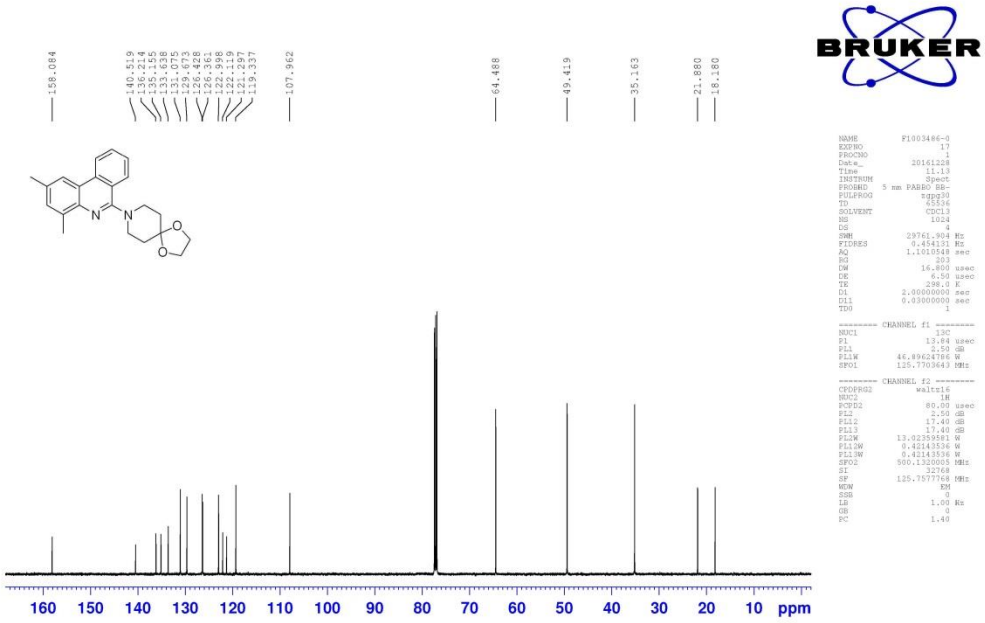
3o


8.543
8.522
8.178
8.158
8.153
8.143
7.740
7.723
7.705
7.702
7.605
7.603
7.585
7.567
7.342
3.860
3.850
3.841
3.817
3.808
3.493
3.482
3.471
3.444
3.415
3.209
3.184
3.158
3.151
3.131
2.534
2.191
2.183
2.168
2.159
2.151
1.938
1.929
1.914
1.906
1.898
1.892
1.885
1.859
1.850
OCH3
BRUKER
NAME F1003484-0
EXPNO 3
PROCNO 1
Date_ 20161227
Time 0.52
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 90.5
DW 60.400 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00 1.00 0.99 1.00 1.00 1.00 1.99 1.00 2.97 1.97 2.95 2.97 2.06 2.00

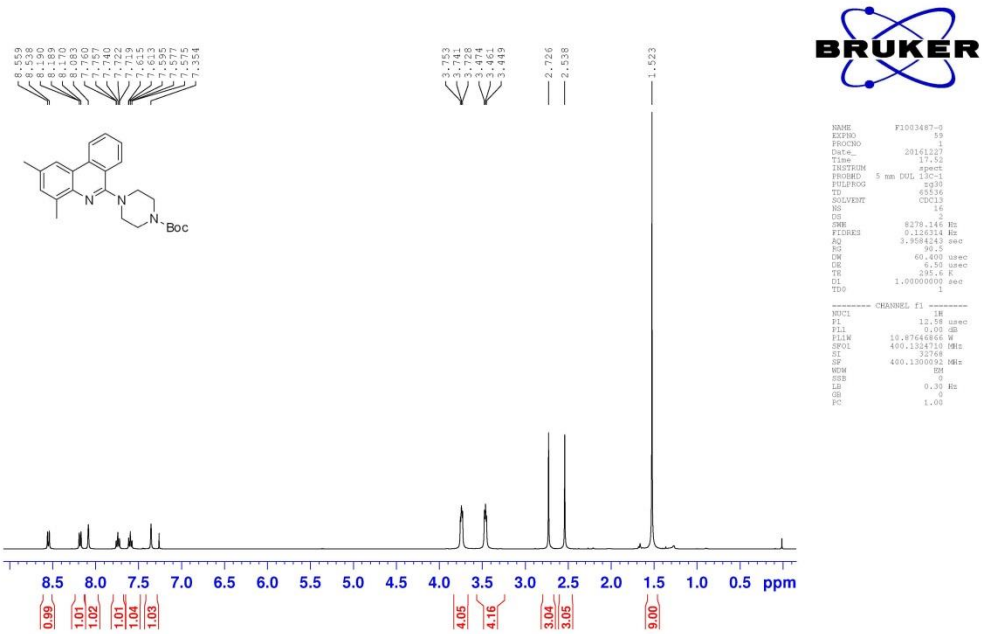

158.464
140.539
136.171
135.137
133.431
131.079
129.684
126.498
126.065
125.975
122.156
121.403
119.368
55.769
49.209
31.293
21.889
18.158
OCH3
BRUKER
NAME F1003484-0
EXPNO 109
PROCNO 1
Date_ 20161227
Time 19.50
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 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 13.04 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7709643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577717 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

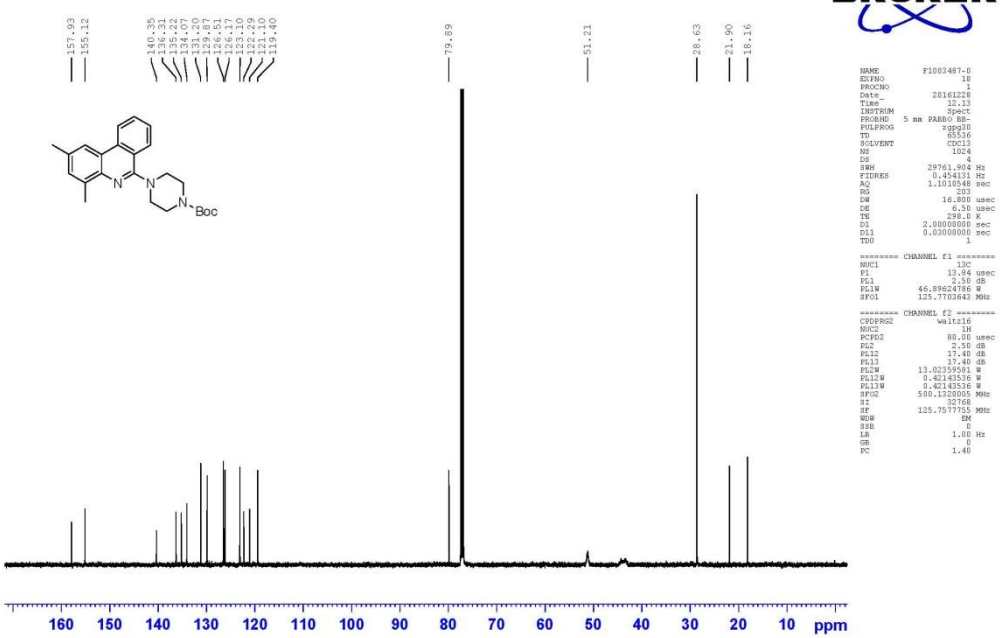
3p


8.548
8.527
8.211
8.189
8.171
8.169
8.045
7.743
7.729
7.726
7.723
7.708
7.705
7.610
7.607
7.589
7.587
7.572
7.569
7.345
4.041
3.643
3.629
3.615
2.733
2.538
2.045
2.031
2.017
BRUKER
NAME F1003486-0
EXPNO 58
PROCNO 1
Date_ 20161227
Time 17.46
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 71.8
DW 60.400 usec
DE 6.50 usec
TE 295.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
0.99
0.98
0.97
1.00
0.99
3.94
3.95
2.94
2.98
3.98

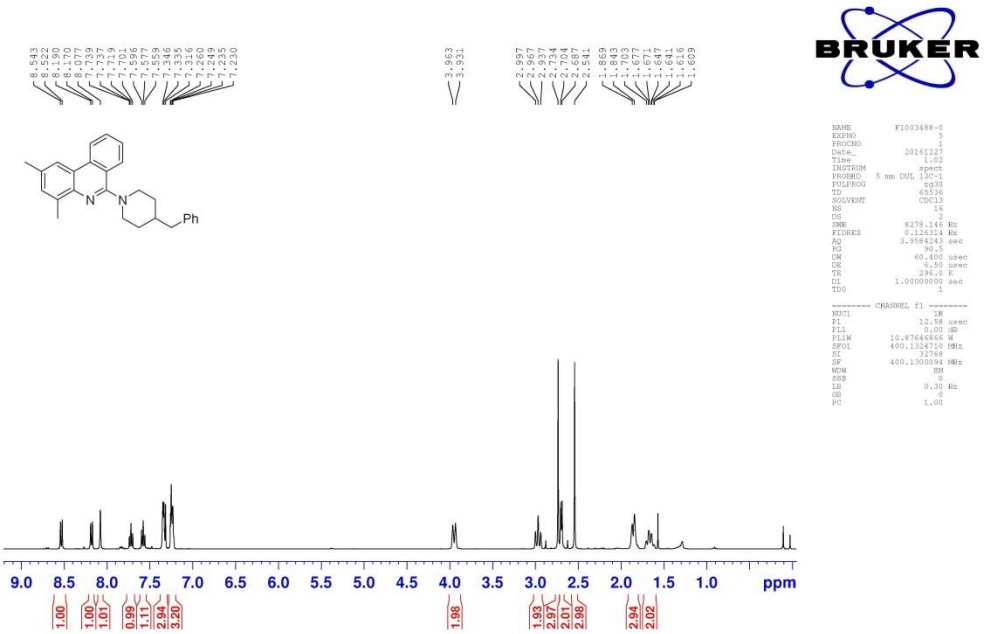

158.084
140.519
136.214
135.455
133.638
131.075
129.673
126.428
126.061
122.998
122.119
121.297
119.337
107.962
64.488
49.419
35.163
21.880
18.280
BRUKER
NAME F1003486-0
EXPNO 17
PROCNO 1
Date_ 20161228
Time 11.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 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 13.04 usec
PL1 2.59 dB
PL1W 46.89624786 W
SFO1 125.7709643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577768 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

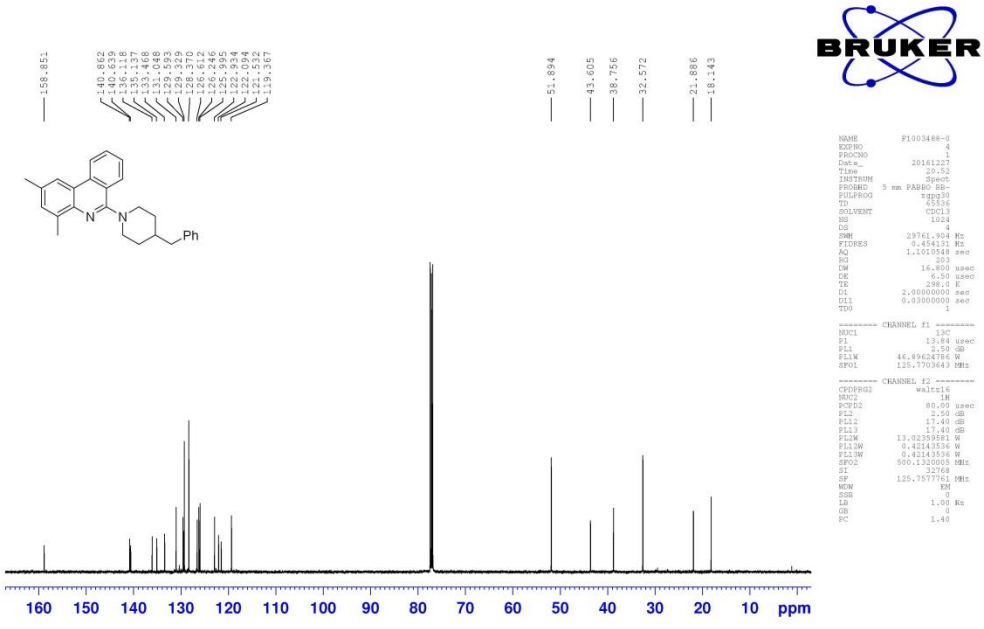
3q


8.559
8.558
8.180
8.169
8.170
8.083
7.760
7.747
7.740
7.722
7.719
7.615
7.613
7.595
7.577
7.575
7.354
3.753
3.741
3.728
3.474
3.461
3.449
2.726
2.538
1.523
Boc
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.99
1.01
1.02
1.01
1.04
1.03
4.05
4.16
3.04
3.05
9.00


157.93
155.12
140.35
136.31
135.22
134.07
131.20
129.87
126.51
126.17
125.10
122.29
121.10
119.40
79.89
51.21
28.63
21.90
18.16
Boc
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

3r


8.543
8.522
8.190
8.170
8.077
7.739
7.737
7.719
7.701
7.596
7.577
7.559
7.546
7.335
7.316
7.260
7.249
7.235
7.230
3.963
3.931
2.957
2.937
2.937
2.734
2.704
2.687
2.541
1.869
1.853
1.703
1.677
1.671
1.647
1.641
1.616
1.609
Ph
BRUKER
NAME F1003488-0
EXPNO 5
PROCNO 1
Date_ 20161227
Time 1.02
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 90.5
DW 60.400 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.00 1.00 1.01 0.99 1.11 2.94 3.20 1.98 1.93 2.97 2.01 2.98 2.94 2.02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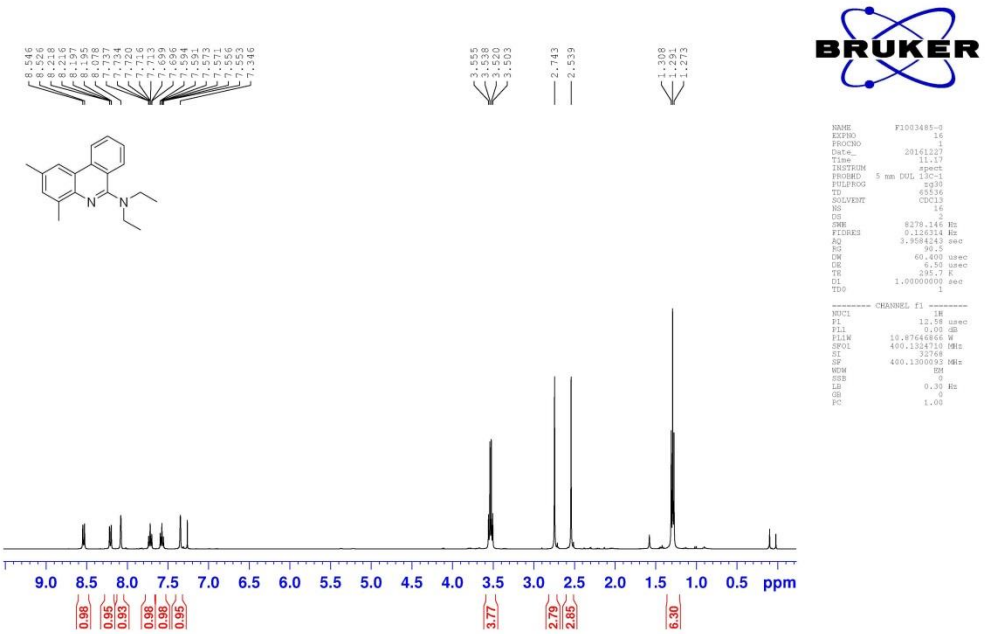
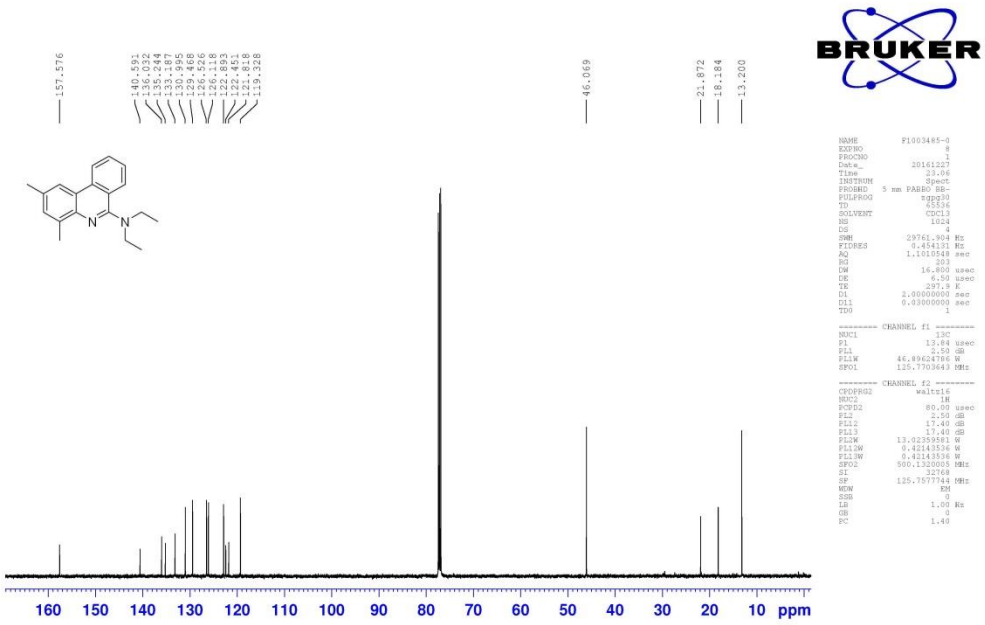
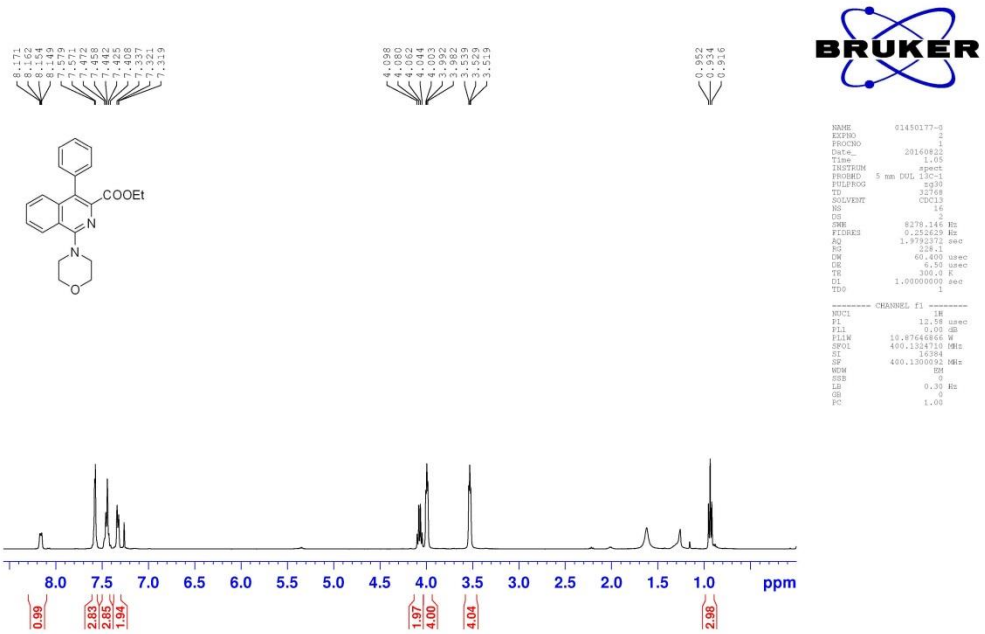
140.662
140.139
136.136
135.137
133.468
131.048
129.323
128.370
126.612
126.046
125.885
122.934
122.094
121.532
119.367
51.894
43.605
38.756
32.572
21.886
18.143
Ph
BRUKER
NAME F1003488-0
EXPNO 4
PROCNO 1
Date_ 20161227
Time 20.52
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 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 13.64 usec
PL1 2.59 dB
PL1W 46.89624786 W
SFO1 125.7709643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577761 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

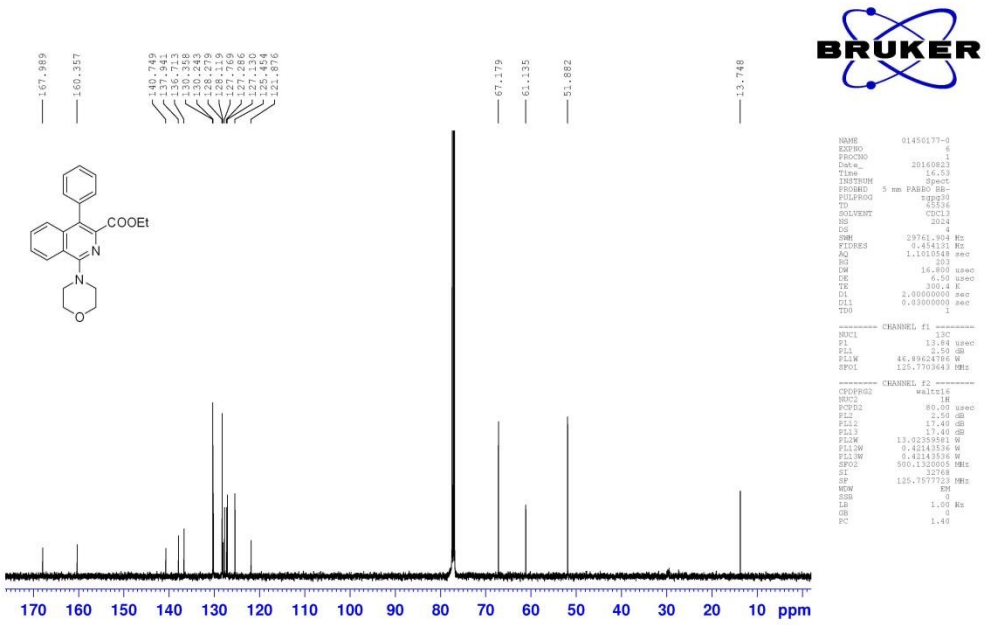
3s


8.546
8.536
8.218
8.197
8.179
8.155
8.130
7.737
7.734
7.720
7.716
7.713
7.699
7.696
7.584
7.581
7.573
7.571
7.556
7.553
7.546
3.555
3.538
3.520
3.503
2.743
2.539
1.308
1.291
1.273
BRUKER
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.98
0.95
0.93
0.98
0.98
0.95
3.77
2.79
2.85
6.30

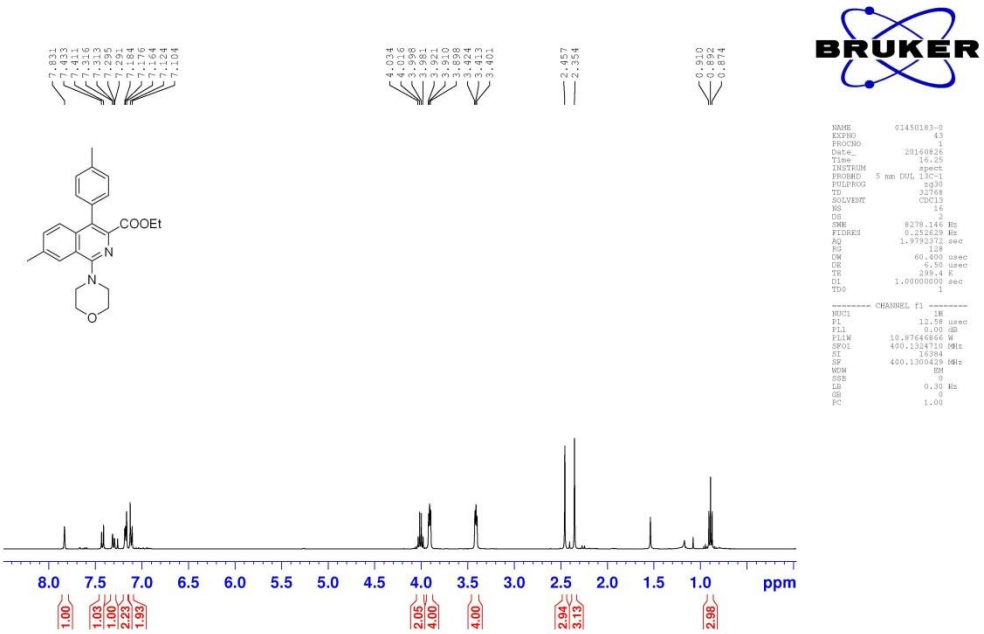

157.576
140.591
136.032
135.244
133.687
130.195
129.468
126.526
126.118
122.493
122.451
121.818
119.328
46.069
21.872
18.184
13.200
BRUKER
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

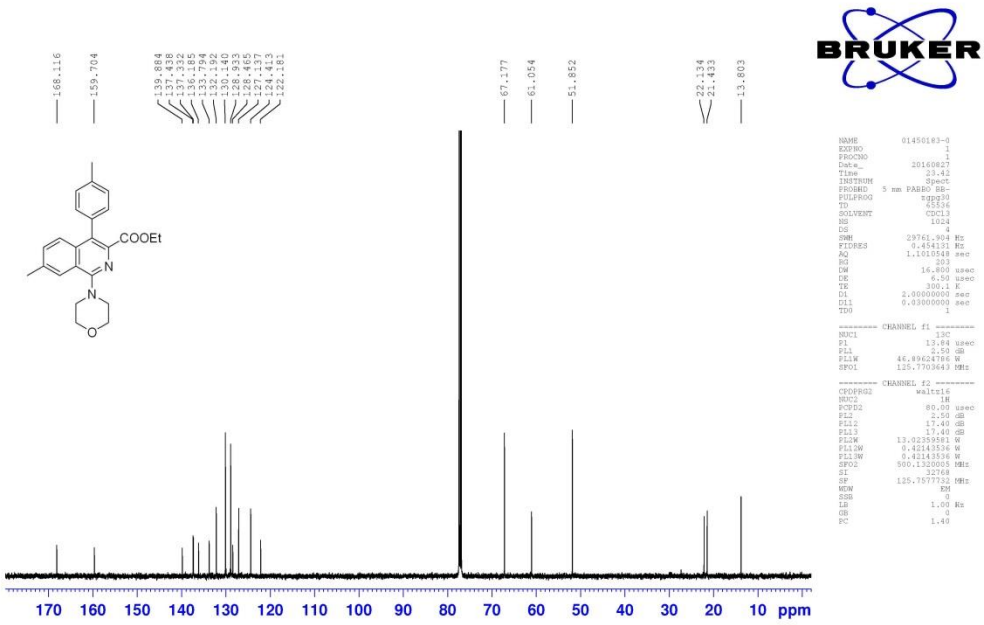
5a


8.171
8.162
8.154
8.149
7.579
7.571
7.472
7.458
7.442
7.425
7.408
7.337
7.321
7.319
4.098
4.080
4.062
4.044
4.003
3.992
3.982
3.539
3.526
3.509
0.952
0.934
0.916
COOEt
N
N
O
BRUKER
NAME 01450177-0
EXPNO 2
PROCNO 1
Date_ 20160822
Time 1.05
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 228.1
DW 60.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PLIW 10.87646866 W
SFO1 400.1324710 MHz
SI 16384
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
0.99
2.83
2.85
1.94
1.97
4.00
4.04
2.98

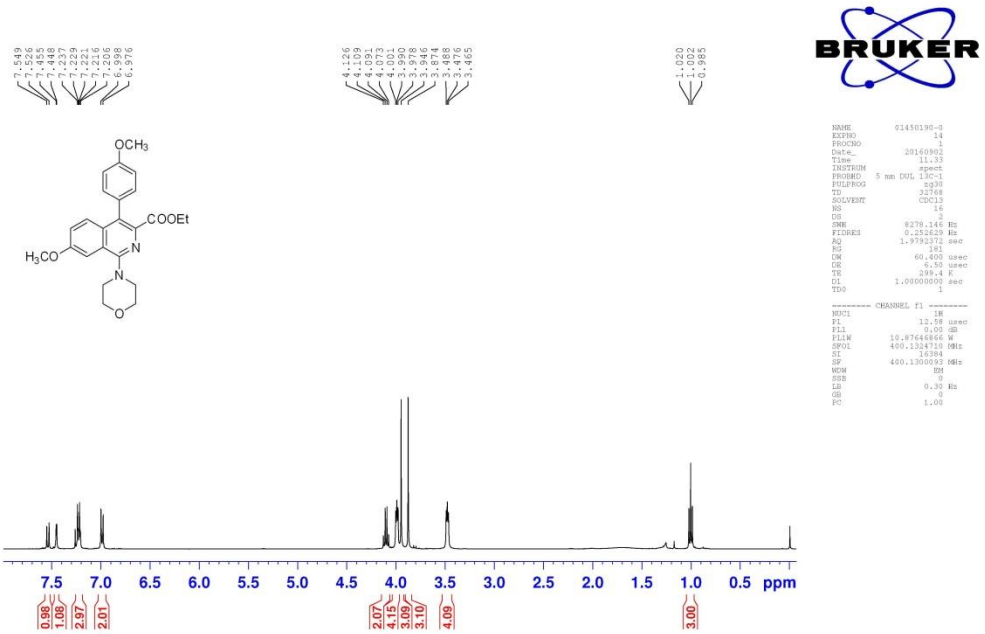

167.989
160.357
140.749
137.941
136.713
130.358
130.043
129.879
128.119
127.769
127.286
125.150
123.454
121.876
67.179
61.135
51.882
13.748
COOEt
N
N
O
BRUKER
NAME 01450177-0
EXPNO 6
PROCNO 1
Date_ 20160823
Time 16.53
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 300.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 13.04 usec
PL1 2.59 dB
PL1W 46.89624786 W
SFO1 125.7709643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577723 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

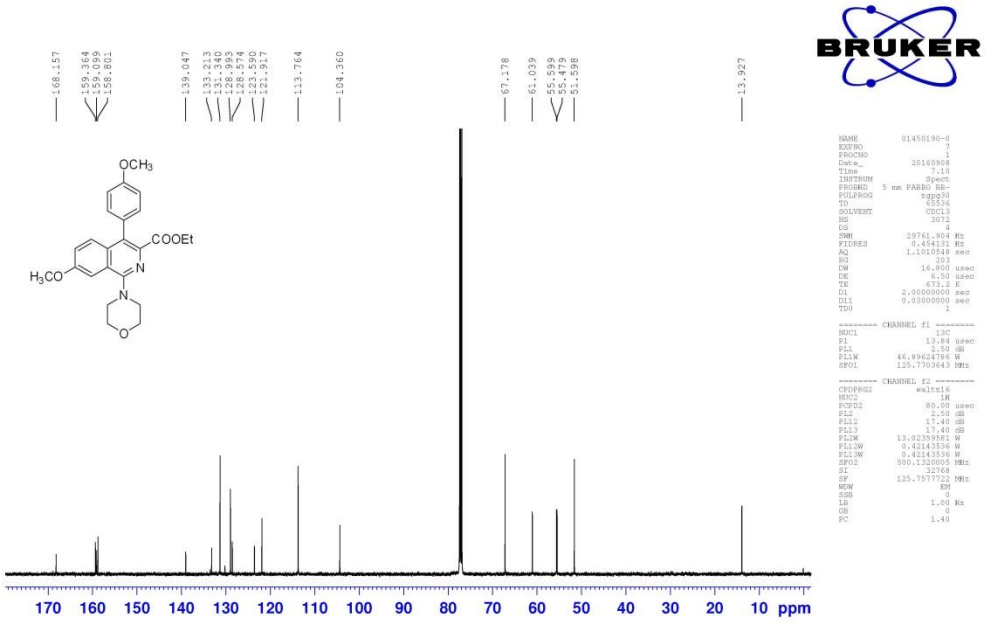
5b


7.831
7.433
7.411
7.316
7.313
7.295
7.291
7.184
7.176
7.164
7.124
7.104
4.034
4.026
3.998
3.961
3.921
3.910
3.898
3.424
3.413
3.401
2.457
2.354
0.910
0.892
0.874
COOEt
N
O
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.00 1.03 1.00 2.23 1.93 2.05 4.00 4.00 2.94 3.13 2.98
BRUKER
NAME 01450183-0
EXPNO 43
PROCNO 1
Date_ 20160826
Time 16.25
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 128
DW 60.400 usec
DE 6.50 usec
TE 299.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 16384
SF 400.1300429 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

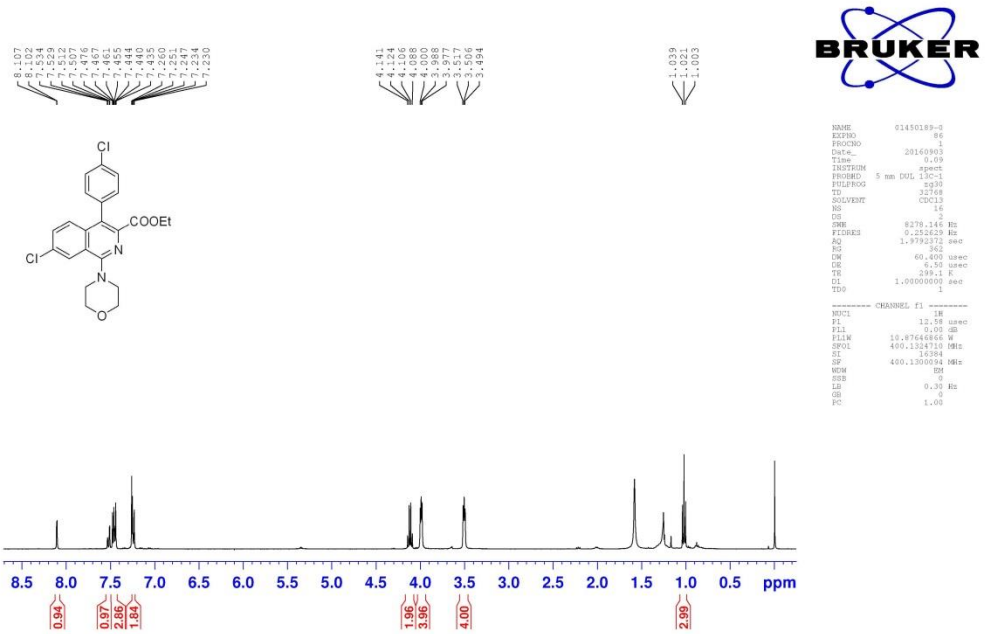

168.116
159.704
139.684
137.438
137.332
136.185
133.794
132.192
130.340
129.033
128.465
127.137
124.413
122.181
67.177
61.054
51.852
22.134
21.433
13.803
COOEt
N
O
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
BRUKER
NAME 01450183-0
EXPNO 1
PROCNO 1
Date_ 20160827
Time 23.42
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 13.04 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7709643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577731 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

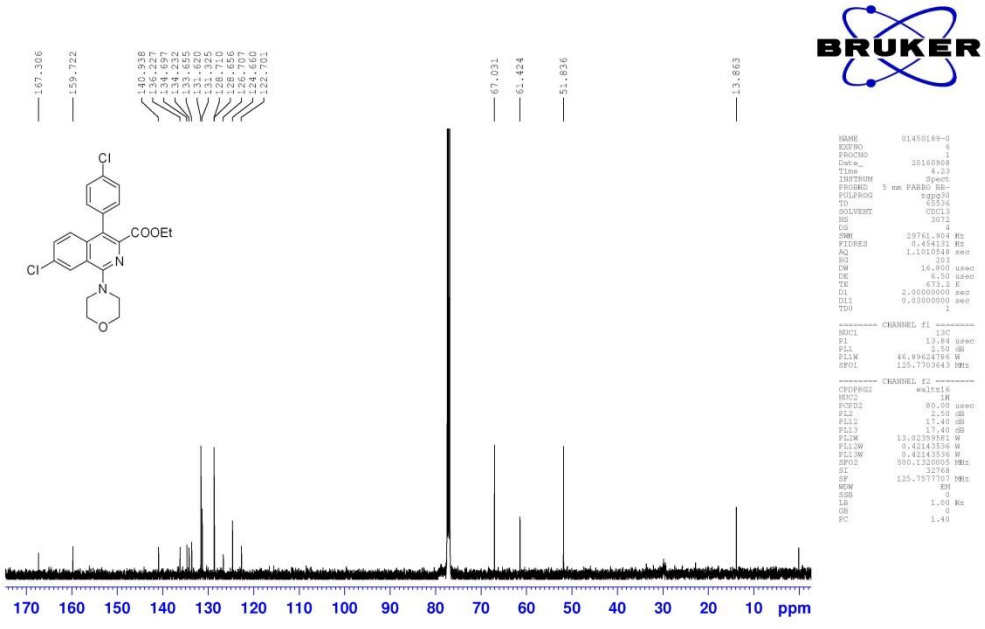
5c


7.549
7.526
7.455
7.448
7.237
7.229
7.221
7.216
7.206
6.998
6.976
4.126
4.109
4.091
4.073
4.031
3.990
3.978
3.946
3.874
3.488
3.476
3.465
1.020
1.002
0.985
OCH3
COOEt
H3CO
N
N
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.98
1.08
2.97
2.01
2.07
4.15
3.09
3.10
4.09
3.00

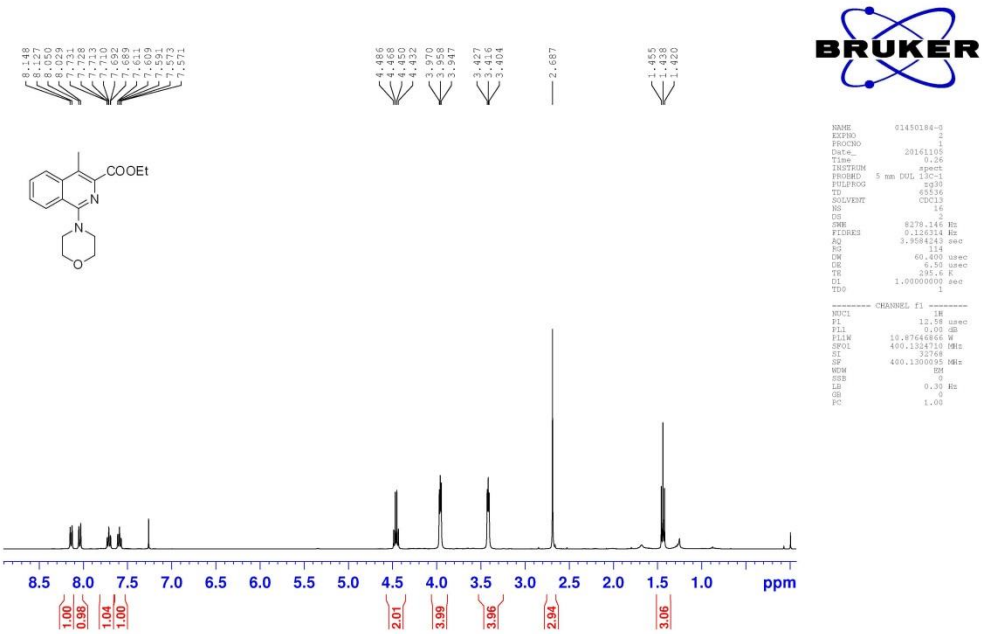

168.157
159.364
158.969
158.801
139.047
133.213
131.340
128.993
128.574
123.590
121.917
113.764
104.360
67.178
61.039
55.599
55.479
51.598
13.927
OCH3
COOEt
H3CO
N
N
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

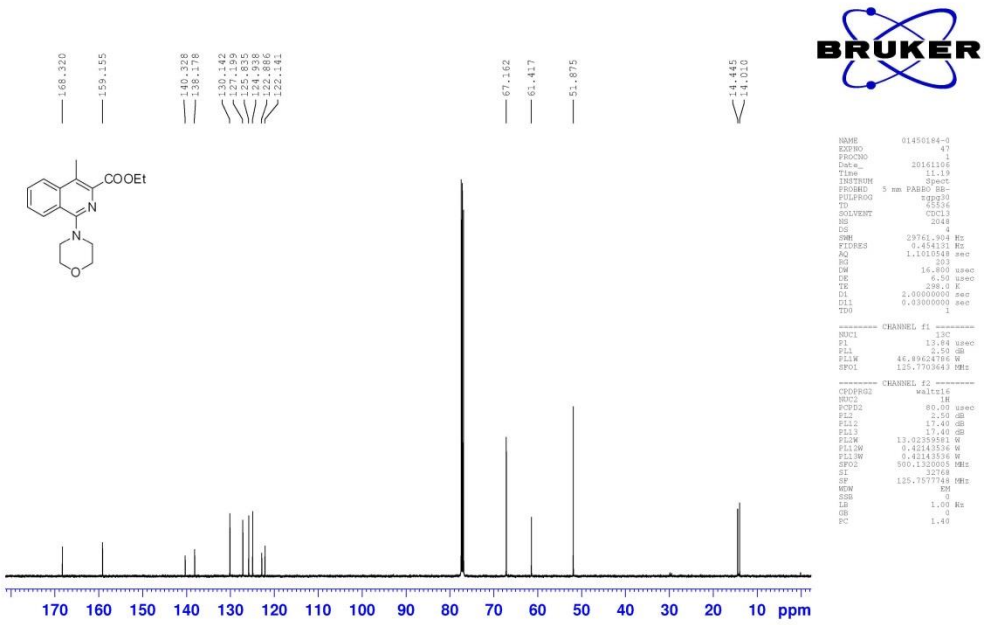
5d


8.107
8.102
8.094
7.529
7.512
7.507
7.476
7.467
7.461
7.455
7.444
7.440
7.435
7.260
7.251
7.247
7.244
7.230
4.141
4.134
4.126
4.088
4.000
3.988
3.977
3.517
3.506
3.494
1.039
1.021
1.003
Cl
COOEt
Cl
N
N
O
BRUKER
NAME 01450189-0
EXPNO 86
PROCNO 1
Date_ 20160903
Time 0.09
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 362
DW 60.400 usec
DE 6.50 usec
TE 299.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PLIW 10.87646866 W
SFO1 400.1324710 MHz
SI 16384
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.94
0.97
2.86
1.64
1.96
3.96
4.00
2.99


167.306
159.722
140.938
136.627
134.697
134.232
133.655
131.620
131.235
128.710
128.656
126.707
124.660
122.701
67.031
61.424
51.836
13.863
Cl
COOEt
Cl
N
N
O
BRUKER
NAME 01450189-0
EXPNO 6
PROCNO 1
Date_ 20160908
Time 4.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3072
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 13.04 usec
PL1 2.50 dB
PL1W 46.89624786 W
SFO1 125.7709643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.50 dB
PL12 17.40 dB
PL13 17.40 dB
PL2W 13.02359581 W
PL12W 0.42143536 W
PL13W 0.42143536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577707 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

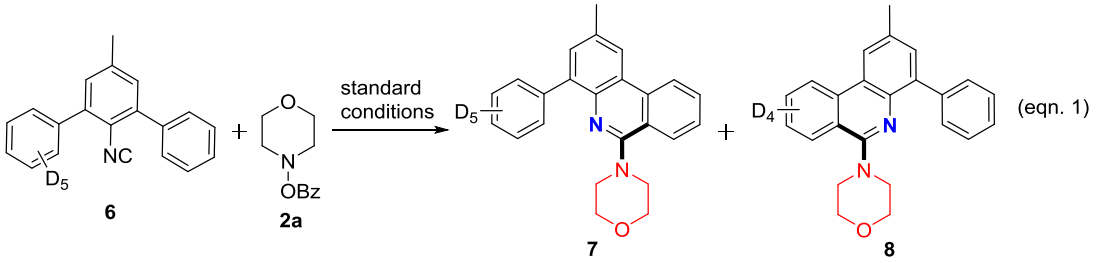
5e


8.148
8.127
8.050
8.029
7.731
7.728
7.713
7.710
7.692
7.689
7.611
7.609
7.591
7.573
7.571
4.466
4.448
4.450
4.432
3.970
3.958
3.947
3.427
3.416
3.404
2.687
1.455
1.438
1.420
COOEt
N
N
O
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.00
0.98
1.04
1.00
2.01
3.99
3.96
2.94
3.06

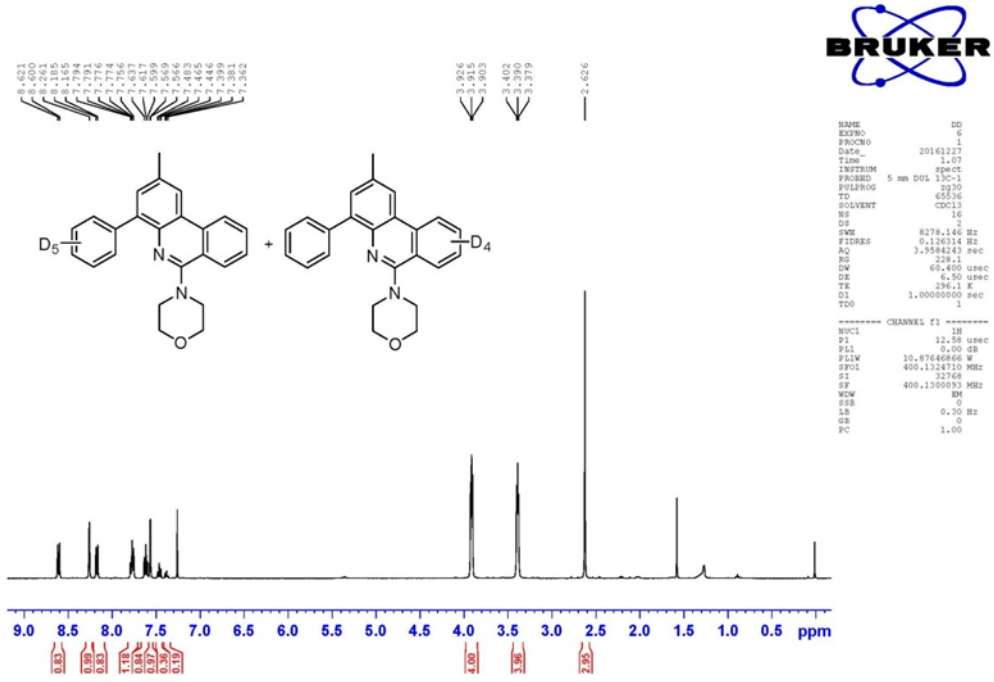

168.320
159.155
140.328
138.178
130.142
127.199
125.835
124.938
122.886
122.481
67.162
61.417
51.875
14.445
14.010
COOEt
N
N
O
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

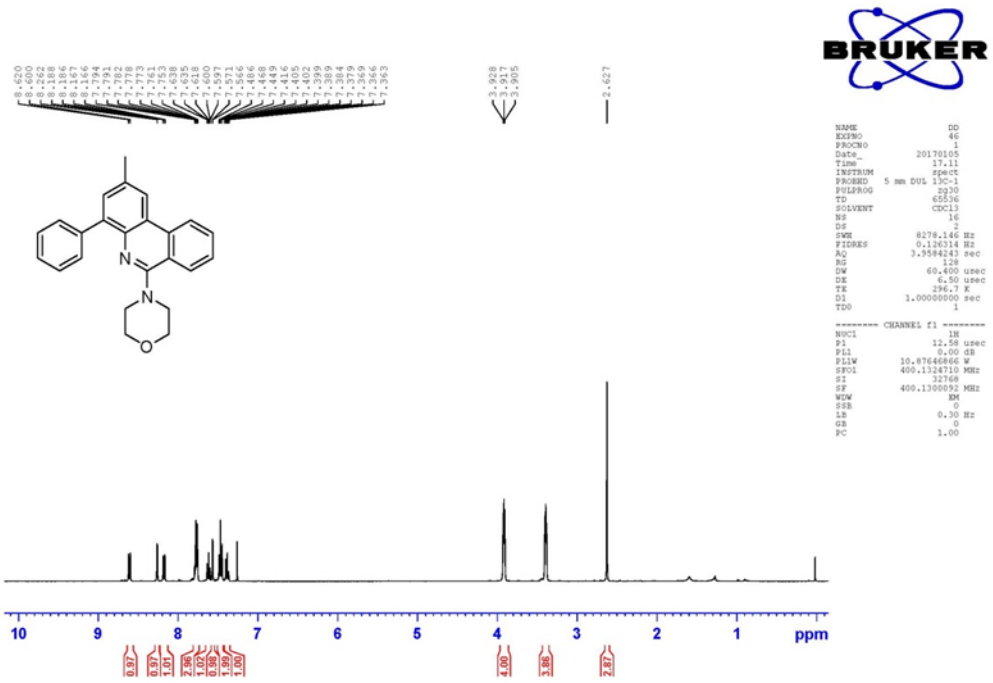
V. Deuteration Studies

To provide preliminary insight into the reaction mechanism, 2,6-diphenyl aryl isocyanide **6** with one of the phenyl rings fully deuterated was used in an intramolecular competition study. An obvious kinetic isotope effect (KIE) k_H/k_D = 4.9 was observed (eqn. 1).



k_H/k_D = 4.9
determined by the ^{1}HNMR ratio of **7** to **8**





BRUKER

NAME	DD
EXPNO	46
PROCNO	1
Date_	20170105
Time	17.11
INSTRUM	spect
PROBHD	5 mm DUL 13C-1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8278.146 Hz
FIDRES	0.126314 Hz
AQ	3.9584243 sec
RG	128
DW	60.400 usec
DE	6.50 usec
TE	296.7 K
D1	1.00000000 sec
TD0	1
-------- CHANNEL f1 --------	
NUC1	1H
P1	12.58 usec
PL1	0.00 dB
PL1W	10.87646866 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300092 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00