New Journal of Chemistry

Green Oxidant H_2O_2 as a Hydrogen Atom Transfer Reagent for Visible Light-Mediated Minisci Reaction

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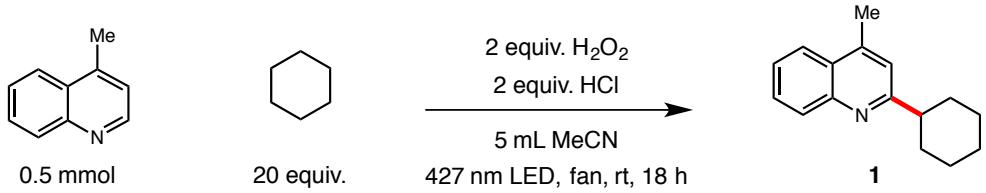
I. General Information

Commercial reagents, lepidine, cyclohexane, hydrogen peroxide (H_2O_2, 29-32% in water), hydrochloric acid (HCl, 36-38% in water), and MeCN were purchased from J&K, TCI, Alfa Aesar, and used directly without purification. All heteroarenes, alkanes, ethers and other reagents were utilized directly from commercial suppliers. Organic solutions were concentrated under reduced pressure on a Büchi rotary evaporator with a recirculating cooling system. Chromatographic purification of products was accomplished by flash chromatography on silica gel (Santai, 230-400 mesh). Thin layer chromatography (TLC) was performed on Huanghai 0.4-0.5 mm silica gel plates. Visualization of the developed chromatogram was performed by fluorescence quenching. ^1H and ^{13}C NMR spectra were recorded on a Bruker UltraShield Plus 400 MHz (101 MHz) instrument, and are internally referenced to residual protio solvent signals (note: $CDCl_3$ referenced at 7.26 and 77.0 ppm respectively). Data for ^1H NMR are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublets, dt = doublet of triplets, br = broad), coupling constant (Hz) and integration. Data for ^{13}C NMR are reported in terms of chemical shift and no special nomenclature is used for equivalent carbons. IR spectra were recorded on a Perkin Elmer Spectrum 100 FTIR spectrometer and are reported in wavenumbers (cm^{-1}). High resolution mass spectra were obtained at National Center for Organic Mass Spectrometry in Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences on a Thermo Fisher Scientific LTQ FTICR-MS instrument with electrospray ionization method.

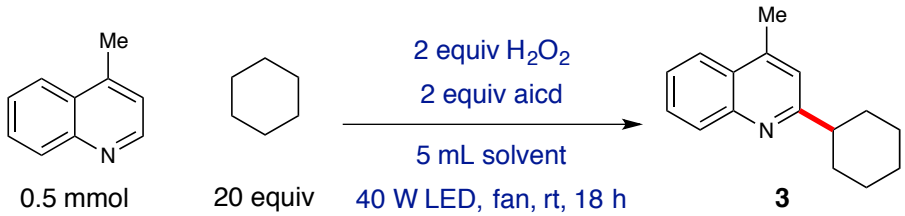
II. Preliminary Mechanistic Studies



Entry	Radical Scavenger	Product **1**
1	TEMPO, 1.0 equiv., [0.10 M]	9%
2	TEMPO, 1.5 equiv., [0.15 M]	0%

Yield determined by ^{1}H NMR using 1,3-benzodioxole as the internal standard.

Figure S1. Radical quenching experiments.



entry	solvent	acid	wavelength	Yield [%]
1	acetone	2 equiv TFA	427 nm	7
2	acetone	2 equiv HCl	427 nm	40
3	MeCN	2 equiv TFA	427 nm	29
4	MeCN	2 equiv HCl	427 nm	87
5	MeCN	none	427 nm	0
6	MeCN	2 equiv HCl	390 nm	60
7	MeCN	2 equiv HCl	440 nm	73
8	MeCN	2 equiv HCl	456 nm	32
9	MeCN	2 equiv HCl	dark	0
10	MeCN	2 equiv HCl	dark, 50 °C	0
11[b]	MeCN	2 equiv HCl	427 nm	87
12[c]	MeCN	2 equiv HCl	427 nm	0

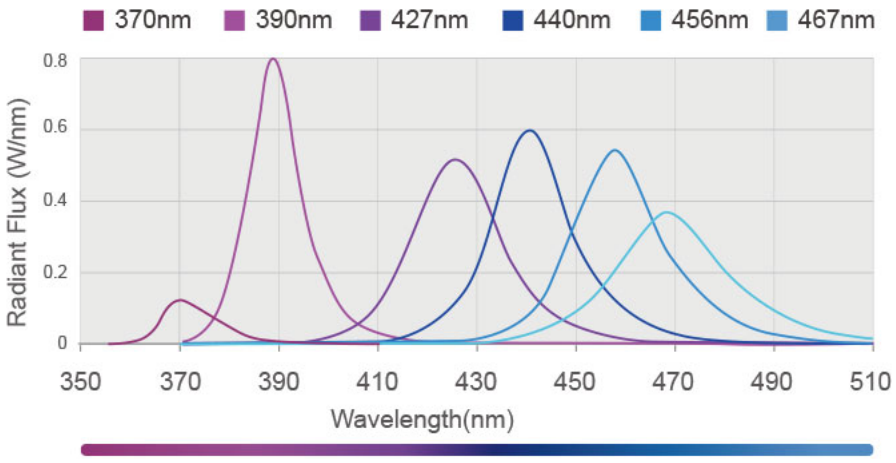
[a]Yields determined by ^{1}H NMR using 1,3-benzodioxole as the internal standard. [b]At 2 mmol scale, isolated yield. [c]1.5 equiv of TEMPO added. LED = light-emitting diode.

Figure S2. Control experiments.

III. Reaction Setup

Figure S3. Reaction setup with a magnetic stirrer, two Kessil 40 W 427 nm or 390 nm LED lamps, two mini fans and four reaction vials.

Kessil PR160_Spectrum

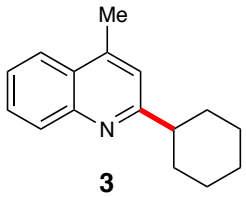


IV. Experimental Procedures and Product Characterization

General Procedure for the Oxidative Alkylation: To an 8 mL vial equipped with a Teflon septum and a magnetic stir bar was charged heteroarene (0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, and hydrogen peroxide (H_2O_2, 29-32% in water, 0.1 mL, 1.0 mmol, 2.0 equiv.). The reaction mixture was degassed by sparging with nitrogen for 10 min with an outlet needle, and added with hydrochloric acid (HCl, 36-38% in water, 86.0 μL, 1.0 mmol, 2.0 equiv.), alkane (10.0 mmol, 20.0 equiv.), then irradiated with Kessil 40 W 427 nm LEDs (approximately 8 cm away from the light source) under two mini fans at room temperature. Upon reaction completion as judged by TLC and LCMS (18 hours), the reaction mixture was diluted with 1 M NaOH aqueous solution (10 mL) and 1 M $Na_2S_2O_3$ aqueous solution (10 mL), and extracted with CH_2Cl_2 (3×20 mL), dried over Na_2SO_4, and concentrated *in vacuo*. Purification of the crude product by flash chromatography on silica gel using the indicated solvent system afforded the desired product.

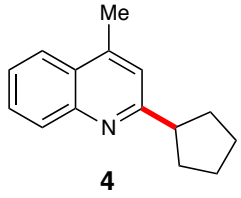
Representative example at 2 mmol scale: To a 40 mL vial equipped with a Teflon septum and a magnetic stir bar was charged lepidine (286.4 mg, 2.0 mmol, 1.0 equiv.), 20.0 mL of MeCN, and hydrogen peroxide (H_2O_2, 29-32% in water, 0.4 mL, 4.0 mmol, 2.0 equiv.). The reaction mixture was degassed by sparging with nitrogen for 10 min with an outlet needle, and added with hydrochloric acid (HCl, 36-38% in water, 344.0 μL, 4.0 mmol, 2.0 equiv.), alkane (4.4 mL, 40.0 mmol, 20.0 equiv.), then irradiated with Kessil 40 W 427 nm LEDs (approximately 8 cm away from the light source) under two mini fans at room temperature for 18 h. The reaction mixture was diluted with 1 M NaOH aqueous solution (40 mL) and 1 M $Na_2S_2O_3$ aqueous solution (40 mL), and extracted

with CH_2Cl_2 (3×80 mL), dried over Na_2SO_4, and concentrated *in vacuo*. Purification of the crude product by flash chromatography (10% ethyl acetate/hexanes) on silica gel provided the desired product 2-cyclohexyl-4-methylquinoline as a colorless oil (388.5 mg, 87% yield).



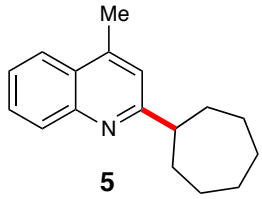
3

2-Cyclohexyl-4-methylquinoline (3): According to the general procedure, lepidine (71.6 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 μL, 1.0 mmol, 2.0 equiv.), cyclohexane (1.10 ml, 10.0 mmol, 20.0 equiv.) were used. After 18 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (10% ethyl acetate/hexanes) to provide the title compound as a colorless oil (98.0 mg, 87% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 8.4 Hz, 1H), 7.93 (d, *J* = 8.3 Hz, 1H), 7.66 (ddd, *J* = 8.4, 6.9, 1.5 Hz, 1H), 7.48 (ddd, *J* = 8.2, 6.8, 1.3 Hz, 1H), 7.16 (s, 1H), 2.88 (tt, *J* = 12.0, 3.4 Hz, 1H), 2.67 (s, 3H), 2.07 – 1.96 (m, 2H), 1.94 – 1.84 (m, 2H), 1.83 – 1.74 (m, 1H), 1.62 (qd, *J* = 12.4, 2.9 Hz, 2H), 1.47 (qt, *J* = 12.6, 3.1 Hz, 2H), 1.35 (tt, *J* = 12.6, 3.3 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.44, 147.52, 144.20, 129.39, 128.88, 126.97, 125.30, 123.50, 120.17, 47.54, 32.77, 26.50, 26.06, 18.79. Spectra data are consistent with those reported in the literature: *Angew. Chem. Int. Ed*. **2013**, *52*, 3267−3271.



4

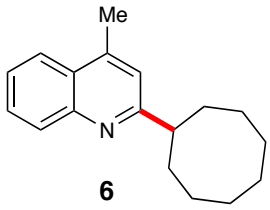
2-Cyclopentyl-4-methylquinoline (4): According to the general procedure, lepidine (71.6 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 µL, 1.0 mmol, 2.0 equiv.), cyclopentane (0.90 ml, 10.0 mmol, 20.0 equiv.) were used. After 18 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (10% ethyl acetate/hexanes) to provide the title compound as a colorless oil (85.6 mg, 81% yield). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.06 (d, *J* = 8.4 Hz, 1H), 7.89 (d, *J* = 8.3 Hz, 1H), 7.64 (ddd, *J* = 8.4, 6.9, 1.5 Hz, 1H), 7.46 (ddd, *J* = 8.2, 6.8, 1.3 Hz, 1H), 7.15 (s, 1H), 3.33 (p, *J* = 8.6 Hz, 1H), 2.64 (s, 3H), 2.22 – 2.11 (m, 2H), 1.95 – 1.81 (m, 4H), 1.81 – 1.69 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.73, 147.38, 143.94, 129.32, 128.76, 126.81, 125.20, 123.38, 120.51, 48.66, 33.43, 25.91, 18.65. Spectra data are consistent with those reported in the literature: *Org. Lett*. **2017**, *19*, 6594-6597.



5

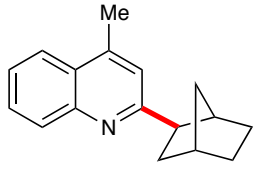
2-Cycloheptyl-4-methylquinoline (5): According to the general procedure, lepidine (71.6 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 µL, 1.0 mmol, 2.0 equiv.), cycloheptane (1.20 ml, 10.0 mmol, 20.0 equiv.) were used. After 18 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (10% ethyl acetate/hexanes) to provide the title compound as a colorless oil (91.0 mg, 76% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, *J* = 8.4 Hz, 1H), 7.92 – 7.86 (m, 1H), 7.63 (ddd, *J* = 8.4, 6.8, 1.5 Hz, 1H), 7.45 (ddd, *J* = 8.2, 6.8, 1.3 Hz, 1H), 7.11 (s, 1H), 3.03 (tt, *J* = 10.5,

3.5 Hz, 1H), 2.63 (s, 3H), 2.10 – 1.97 (m, 2H), 1.90 – 1.76 (m, 4H), 1.76 – 1.68 (m, 2H), 1.68 – 1.56 (m, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.88, 147.20, 144.15, 129.25, 128.73, 126.74, 125.12, 123.33, 120.10, 49.41, 34.89, 27.80, 27.29, 18.63. Spectra data are consistent with those reported in the literature: *Org. Lett.* **2019**, *21*, DOI: acs.orglett.9b01439.



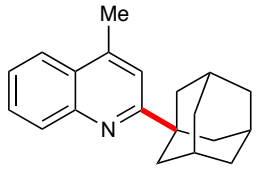
6

2-Cyclooctyl-4-methylquinoline (6): According to the general procedure, lepidine (71.6 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 µL, 1.0 mmol, 2.0 equiv.), cyclooctane (1.30 ml, 10.0 mmol, 20.0 equiv.) were used. After 18 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (10% ethyl acetate/hexanes) to provide the title compound as a colorless oil (86.2 mg, 68% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, *J* = 8.4 Hz, 1H), 7.89 (d, *J* = 8.3 Hz, 1H), 7.64 (ddd, *J* = 8.4, 6.8, 1.5 Hz, 1H), 7.45 (ddd, *J* = 8.2, 6.8, 1.3 Hz, 1H), 7.11 (s, 1H), 3.11 (tt, *J* = 9.8, 3.6 Hz, 1H), 2.64 (s, 3H), 2.05 – 1.93 (m, 2H), 1.93 – 1.77 (m, 4H), 1.77 – 1.54 (m, 8H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.66, 147.23, 144.17, 129.33, 128.78, 126.76, 125.18, 123.39, 120.53, 47.40, 33.39, 26.49, 26.30, 26.04, 18.71. Spectra data are consistent with those reported in the literature: *Org. Lett.* **2019**, *21*, DOI: acs.orglett.9b01635.



7

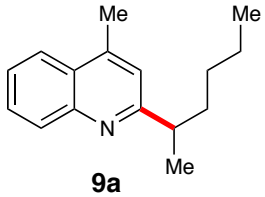
2-(Bicyclo[2.2.1]heptan-2-yl)-4-methylquinoline (7): According to the general procedure, lepidine (71.6 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 µL, 1.0 mmol, 2.0 equiv.), bicyclo[2.2.1]heptane (721.3 mg, 7.5 mmol, 15.0 equiv.) were used. After 18 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (20% ethyl acetate/hexanes) to provide the title compound as a colorless oil (87.8 mg, 74% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (dd, *J* = 8.4, 1.3 Hz, 1H), 7.92 (dd, *J* = 8.3, 1.4 Hz, 1H), 7.65 (ddd, *J* = 8.4, 6.9, 1.5 Hz, 1H), 7.48 (ddd, *J* = 8.2, 6.8, 1.3 Hz, 1H), 7.18 (s, 1H), 3.01 (dd, *J* = 8.8, 5.5 Hz, 1H), 2.66 (s, 3H), 2.59 – 2.54 (d, *J* = 1.9 Hz, 1H), 2.45 – 2.39 (s, 1H), 2.31 – 2.21 (m, 1H), 1.79 – 1.56 (m, 4H), 1.52 – 1.44 (m, 1H), 1.38 – 1.31 (m, 1H), 1.22 – 1.16 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.45, 147.35, 143.65, 129.60, 128.71, 126.66, 125.21, 123.39, 121.47, 49.96, 42.98, 36.68, 36.18, 36.00, 30.46, 29.09, 18.70. Spectra data are consistent with those reported in the literature: *Org. Lett*. **2019**, *21*, DOI: acs.orglett.9b01635.



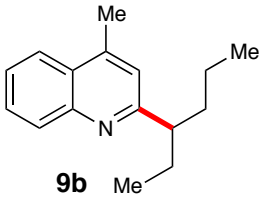
8

2-(Adamantan-1-yl)-4-methylquinoline (8): According to the general procedure, lepidine (71.6 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 µL, 1.0 mmol, 2.0 equiv.), Adamantane (1.02 g, 7.5 mmol, 15.0 equiv.) were used. After 18 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (20% ethyl acetate/hexanes) to provide the title compounds as a white solid (58.3 mg, 42%

yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (d, *J* = 8.4 Hz, 1H), 7.94 (d, *J* = 8.3 Hz, 1H), 7.66 (ddd, *J* = 8.4, 6.8, 1.5 Hz, 1H), 7.49 (ddd, *J* = 8.2, 6.8, 1.3 Hz, 1H), 7.33 (s, 1H), 2.69 (s, 3H), 2.16 (s, 3H), 2.13 (s, 6H), 1.84 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.64, 147.46, 143.54, 129.87, 128.59, 126.66, 125.28, 123.38, 118.48, 41.75, 39.50, 36.85, 28.80, 18.95; HRMS (ESI) m/z calculated for $C_{20}H_{24}N$ [$(M+H)^+$] 278.1903, found 278.1901. IR (film) 2902, 2846, 1595, 1552, 1505, 1444, 1412, 1340, 1309, 757 cm^{-1}. Spectra data are consistent with those reported in the literature: *Org. Lett*. **2019**, *21*, DOI: acs.orglett.9b01439.



9a



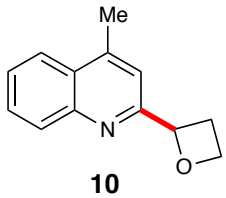
9b

2-(Hexan-2-yl)-4-methylquinoline (9a) and 2-(Hexan-3-yl)-4-methylquinoline (9b): According to the general procedure, lepidine (71.6 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 μL, 1.0 mmol, 2.0 equiv.), hexane (1.30 ml, 10.0 mmol, 20.0 equiv.) were used. After 18 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (10% ethyl acetate/hexanes) to provide the title compounds compounds as a colorless oil mixture of 50.1 mg, 44% total yield (28% yield for **9a**; 16% yield for **9b**).

Compound **9a**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, *J* = 8.3 Hz, 1H), 7.95 (d, *J* = 8.3 Hz, 1H), 7.66 (ddd, *J* = 8.4, 6.8, 1.4 Hz, 1H), 7.49 (ddd, *J* = 8.2, 6.9, 1.3 Hz, 1H), 7.14 (s, 1H), 3.03 (h, *J* = 7.0 Hz, 1H), 2.68 (s, 3H), 1.88 – 1.57 (m, 3H), 1.37 – 1.25 (m, 2H), 1.35 (d, *J* = 7.0 Hz, 3H), 1.24 – 1.12 (m, 1H), 0.85 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz,

$CDCl_3$) δ 166.91, 147.50, 144.23, 129.45, 128.87, 126.99, 125.34, 123.53, 120.10, 42.89, 36.77, 29.93, 22.80, 20.78, 18.84, 14.00. Spectra data are consistent with those reported in the literature: *Org. Lett*. **2019**, *21*, DOI: acs.orglett.9b01635.

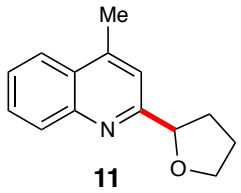
Compound **9b**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (d, *J* = 8.3 Hz, 1H), 7.95 (d, *J* = 8.3 Hz, 1H), 7.66 (ddd, *J* = 8.4, 6.8, 1.4 Hz, 1H), 7.49 (ddd, *J* = 8.2, 6.9, 1.3 Hz, 1H), 7.10 (s, 1H), 2.83 (p, *J* = 7.2 Hz, 1H), 2.68 (s, 3H), 1.88 – 1.57 (m, 2H), 1.37 – 1.25 (m, 3H), 1.24 – 1.12 (m, 1H), 0.86 (t, *J* = 7.0 Hz, 3H), 0.82 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.78, 147.54, 143.95, 129.50, 128.80, 127.00, 125.30, 123.53, 120.65, 50.27, 37.66, 28.51, 20.82, 18.84, 14.23, 12.21. Spectra data are consistent with those reported in the literature: *Org. Lett*. **2019**, *21*, DOI: acs.orglett.9b01635.



10

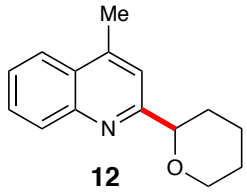
4-Methyl-2-(oxetan-2-yl)quinoline (10): According to the general procedure, lepidine (71.6 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 μL, 1.0 mmol, 2.0 equiv.), trimethylene oxide (0.65 ml, 10.0 mmol, 20.0 equiv.) were used. After 18 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (40% ethyl acetate/hexanes) to provide the title compound as a colorless oil (45.8 mg, 46% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 8.4 Hz, 1H), 7.98 (d, *J* = 8.3 Hz, 1H), 7.68 (ddd, *J* = 8.3, 6.9, 1.3 Hz, 1H), 7.67 (s, 1H), 7.53 (ddd, *J* = 8.3, 6.9, 1.3 Hz, 1H), 5.98 (dd, *J* = 8.4, 6.9 Hz, 1H), 4.92 (td, *J* = 8.0, 5.9 Hz, 1H), 4.77 (dt, *J* = 9.1, 6.0 Hz, 1H), 3.19 (dtd, *J* = 11.2, 8.4, 6.0 Hz, 1H), 2.80 (ddt, *J* = 11.3, 9.2, 7.3 Hz, 1H), 2.74 (s,

3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.52, 147.30, 145.48, 129.58, 129.33, 127.52, 126.09, 123.75, 118.21, 83.62, 69.20, 29.02, 18.93; HRMS (ESI) m/z calculated for $C_{13}H_{14}ON$ [$(M+H)^+$] 200.1070, found 200.1069. IR (film) 3062, 2885, 1601, 1562, 1508, 1448, 1412, 1348, 1306, 1226, 1038, 980 760 cm^{-1}. Spectra data are consistent with those reported in the literature: *Org. Lett*. **2019**, *21*, DOI: acs.orglett.9b01635.



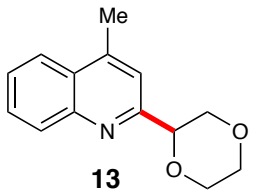
11

4-Methyl-2-(tetrahydrofuran-2-yl)quinoline (11): According to the general procedure, lepidine (71.6 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 μL, 1.0 mmol, 2.0 equiv.), tetrahydrofuran (0.91 ml, 10.0 mmol, 20.0 equiv.) were used. After 18 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (20% ethyl acetate/hexanes) to provide the title compound as a colorless oil (69.3 mg, 65% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, J = 8.4 Hz, 1H), 7.96 (d, J = 8.3 Hz, 1H), 7.68 (ddd, J = 8.4, 6.8, 1.4 Hz, 1H), 7.52 (ddd, J = 8.2, 6.8, 1.3 Hz, 1H), 7.44 (s, 1H), 5.14 (t, J = 6.9 Hz, 1H), 4.21 – 4.13 (m, 1H), 4.07 – 3.99 (m, 1H), 2.70 (s, 3H), 2.56 – 2.45 (m, 1H), 2.11 – 1.98 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.03, 147.21, 144.97, 129.44, 129.11, 127.40, 125.78, 123.65, 118.53, 82.00, 69.22, 33.30, 25.93, 18.87. Spectra data are consistent with those reported in the literature: *Synlett*, **2016**, *27*, 1282-1286.



12

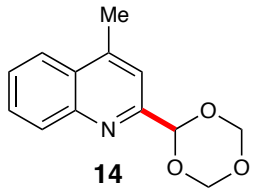
4-Methyl-2-(tetrahydro-2*H*-pyran-2-yl)quinoline (12): According to the general procedure, lepidine (71.6 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 µL, 1.0 mmol, 2.0 equiv.), tetrahydro- 2*H*-pyran (1.10 ml, 10.0 mmol, 20.0 equiv.) were used. After 18 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (20% ethyl acetate/hexanes) to provide the title compound as a colorless oil (76.1 mg, 67% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 8.4 Hz, 1H), 7.93 (d, *J* = 8.3 Hz, 1H), 7.65 (ddd, *J* = 8.4, 6.8, 1.4 Hz, 1H), 7.48 (ddd, *J* = 8.3, 6.8, 1.3 Hz, 1H), 7.45 (s, 1H), 4.59 (dd, *J* = 11.1, 2.3 Hz, 1H), 4.24 – 4.15 (m, 1H), 3.67 (td, *J* = 11.6, 2.5 Hz, 1H), 2.67 (s, 3H), 2.09 (dt, *J* = 12.5, 2.1 Hz, 1H), 2.00 – 1.91 (m, 1H), 1.80 – 1.68 (m, 2H), 1.67 – 1.52 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.99, 146.99, 144.86, 129.46, 128.93, 127.37, 125.70, 123.50, 118.67, 81.47, 68.75, 32.65, 25.73, 23.60, 18.70. Spectra data are consistent with those reported in the literature: *Angew. Chem. Int. Ed*. **2015**, *54*, 1565-1569.



13

2-(1,4-Dioxan-2-yl)-4-methylquinoline (13): According to the general procedure, lepidine (71.6 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 µL, 1.0 mmol, 2.0 equiv.), 1,4-dioxane (0.94 ml, 10.0 mmol, 20.0

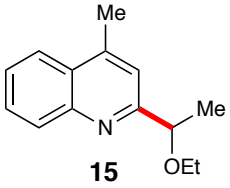
equiv.) were used. After 18 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (20% ethyl acetate/hexanes) to provide the title compound as a white solid (86.0 mg, 75% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 8.3 Hz, 1H), 7.93 (d, *J* = 8.3 Hz, 1H), 7.66 (ddd, *J* = 8.4, 6.8, 1.4 Hz, 1H), 7.50 (ddd, *J* = 8.3, 6.8, 1.3 Hz, 1H), 7.43 (s, 1H), 4.87 (dd, *J* = 10.1, 2.9 Hz, 1H), 4.22 (dd, *J* = 11.6, 3.0 Hz, 1H), 4.03 – 3.91 (m, 2H), 3.84 – 3.71 (m, 2H), 3.61 (dd, *J* = 11.6, 10.1 Hz, 1H), 2.67 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.67, 147.11, 145.00, 129.62, 129.11, 127.44, 126.05, 123.53, 118.95, 78.60, 70.94, 66.91, 66.26, 18.68. Spectra data are consistent with those reported in the literature: *Synlett*, **2016**, *27*, 1282-1286.



14

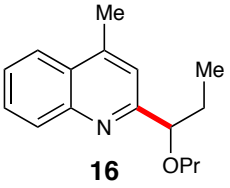
4-Methyl-2-(1,3,5-trioxan-2-yl)quinoline (14): According to the general procedure, lepidine (71.6 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 μL, 1.0 mmol, 2.0 equiv.), 1,3,5-trioxane (675 mg, 7.5 mmol, 15.0 equiv.) were used. After 18 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (20% ethyl acetate/hexanes) to provide the title compound as a white solid (41.6 mg, 36% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (d, *J* = 8.4 Hz, 1H), 8.01 (d, *J* = 8.3 Hz, 1H), 7.72 (ddd, *J* = 8.4, 6.8, 1.5 Hz, 1H), 7.66 (s, 1H), 7.59 (ddd, *J* = 8.3, 6.9, 1.3 Hz, 1H), 6.08 (s, 1H), 5.45 – 5.37 (m, 4H), 2.75 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 154.77, 146.99, 145.99, 130.10, 129.48, 128.36, 126.96, 123.72, 118.83, 102.12, 93.69, 18.90.

Spectra data are consistent with those reported in the literature: *Bioorg. Med. Chem. Lett.* **2012**, *22*, 3480-3484.



15

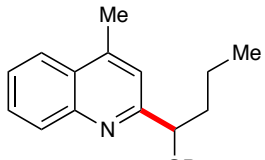
2-(1-Ethoxyethyl)-4-methylquinoline (15): According to the general procedure, lepidine (71.6 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 µL, 1.0 mmol, 2.0 equiv.), ethoxyethane (1.04 ml, 10.0 mmol, 20.0 equiv.) were used. After 18 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (10% ethyl acetate/hexanes) to provide the title compound as a colorless oil (92.9 mg, 77% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, J = 8.4 Hz, 1H), 7.98 (d, J = 8.3 Hz, 1H), 7.69 (ddd, J = 8.4, 6.8, 1.4 Hz, 1H), 7.54 (ddd, J = 8.3, 6.9, 1.3 Hz, 1H), 7.44 (s, 1H), 4.67 (q, J = 6.6 Hz, 1H), 3.51 (dq, J = 9.3, 7.0 Hz, 1H), 3.41 (dq, J = 9.3, 7.0 Hz, 1H), 2.73 (s, 3H), 1.53 (d, J = 6.6 Hz, 3H), 1.23 (t, J = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.95, 147.10, 145.26, 129.45, 129.16, 127.62, 125.96, 123.68, 118.26, 79.67, 64.61, 22.66, 18.98, 15.45. Spectra data are consistent with those reported in the literature: *Synlett*, **2016**, *27*, 1282-1286.



16

4-Methyl-2-(1-propoxypropyl)quinoline (16): According to the general procedure, lepidine (71.6 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol,

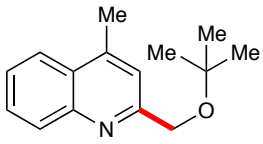
2.0 equiv.), HCl (86.0 µL, 1.0 mmol, 2.0 equiv.), 1-propoxypropane (1.39 ml, 10.0 mmol, 20.0 equiv.) were used. After 18 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (10% ethyl acetate/hexanes) to provide the title compound as a colorless oil (86.4 mg, 71% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, *J* = 8.4 Hz, 1H), 7.95 (d, *J* = 7.9 Hz, 1H), 7.66 (ddd, *J* = 8.4, 6.9, 1.5 Hz, 1H), 7.50 (ddd, *J* = 8.2, 6.9, 1.3 Hz, 1H), 7.40 (s, 1H), 4.40 (dd, *J* = 7.9, 5.5 Hz, 1H), 3.34 (td, *J* = 6.6, 1.6 Hz, 2H), 2.69 (s, 3H), 1.95 – 1.74 (m, 2H), 1.61 (h, *J* = 7.1 Hz, 2H), 0.99 (t, *J* = 7.4 Hz, 3H), 0.90 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.31, 147.09, 144.72, 129.44, 128.95, 127.53, 125.78, 123.57, 118.71, 85.16, 71.21, 29.92, 23.02, 18.84, 10.55, 10.19. Spectra data are consistent with those reported in the literature: *Eur. J. Org. Chem.* **2015**, *2015*, 4973-4981.



17

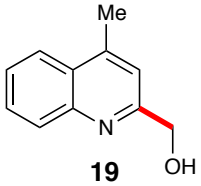
2-(1-Butoxybutyl)-4-methylquinoline (17): According to the general procedure, lepidine (71.6 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 µL, 1.0 mmol, 2.0 equiv.), 1-butoxybutane (1.70 ml, 10.0 mmol, 20.0 equiv.) were used. After 18 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (10% ethyl acetate/hexanes) to provide the title compound as a colorless oil (74.6 mg, 55% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (d, *J* = 8.4 Hz, 1H), 7.97 (d, *J* = 8.2 Hz, 1H), 7.68 (ddd, *J* = 8.4, 6.8, 1.4 Hz, 1H), 7.52 (ddd, *J* = 8.3, 6.9, 1.3 Hz, 1H), 7.41 (s, 1H), 4.48 (dd, *J* = 8.5, 4.9 Hz, 1H), 3.37 (t, *J* = 6.6 Hz, 2H), 2.71 (s, 3H), 1.89 – 1.77 (m, 1H),

1.77 – 1.66 (m, 1H), 1.63 – 1.50 (m, 3H), 1.46 – 1.33 (m, 3H), 0.93 (t, J = 7.4 Hz, 3H), 0.88 (t, J = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.63, 147.14, 144.84, 129.51, 129.02, 127.59, 125.84, 123.64, 118.73, 83.85, 69.30, 39.20, 31.98, 19.31, 19.13, 18.92, 13.97, 13.82. Spectra data are consistent with those reported in the literature: *Synlett*, **2016**, *27*, 1282-1286.



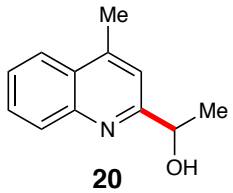
18

2-(*tert*-Butoxymethyl)-4-methylquinoline (18): According to the general procedure, lepidine (71.6 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 µL, 1.0 mmol, 2.0 equiv.), 2-methoxy-2-methylpropane (1.19 ml, 10.0 mmol, 20.0 equiv.) were used. After 18 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (10% ethyl acetate/hexanes) to provide the title compound as a colorless oil (55.0 mg, 48% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, J = 8.4 Hz, 1H), 7.94 (d, J = 7.8 Hz, 1H), 7.65 (ddd, J = 8.4, 6.8, 1.5 Hz, 1H), 7.52 – 7.45 (m, 2H), 4.71 (s, 2H), 2.68 (s, 3H), 1.32 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.18, 147.07, 144.69, 129.21, 129.02, 127.37, 125.68, 123.61, 119.99, 73.91, 65.93, 27.60, 18.76; HRMS (ESI) m/z calculated for $C_{15}H_{20}NO$ [$(M+H)^+$] 230.1539, found 230.1538. IR (film) 2974, 2931, 1603, 1565, 1509, 1448, 1390, 1363, 1250, 1194, 1096, 758 cm^{-1}.



19

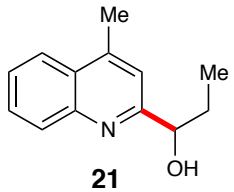
(4-Methylquinolin-2-yl)methanol (19): According to the general procedure, lepidine (71.6 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 µL, 1.0 mmol, 2.0 equiv.), methanol (0.30 ml, 7.5 mmol, 15.0 equiv.) were used. After 18 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (50% ethyl acetate/hexanes) to provide the title compound as a white solid (60.6 mg, 70% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, J = 8.4 Hz, 1H), 7.95 (d, J = 8.3 Hz, 1H), 7.69 (ddd, J = 8.4, 6.9, 1.5 Hz, 1H), 7.54 (ddd, J = 8.3, 6.9, 1.3 Hz, 1H), 7.11 (s, 1H), 4.86 (s, 2H), 4.55 (br s, 1H), 2.67 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 158.69, 146.45, 145.09, 129.41, 129.08, 127.57, 126.05, 123.79, 118.96, 64.03, 18.79. Spectra data are consistent with those reported in the literature: *Org. Lett.* **2018**, *20*, 3229-3232.



20

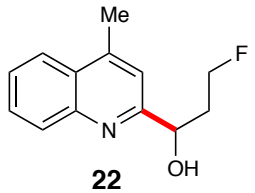
1-(4-Methylquinolin-2-yl)ethan-1-ol (20): According to the general procedure, lepidine (71.6 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 µL, 1.0 mmol, 2.0 equiv.), ethanol (0.44 ml, 7.50 mmol, 15.0 equiv.) were used. After 18 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (30% ethyl acetate/hexanes) to provide the title compound as a white solid (59.0 mg, 63% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (d, J = 8.4 Hz, 1H), 7.97 (d, J = 8.3 Hz, 1H), 7.71 (ddd, J = 8.4, 6.9, 1.4 Hz, 1H), 7.55 (ddd, J = 8.2, 6.9, 1.3 Hz, 1H), 7.18 (s, 1H), 5.14 (br s, 1H), 4.99 (q, J = 6.6 Hz, 1H), 2.71 (s, 3H), 1.57 (d, J = 6.6 Hz, 3H); ^{13}C NMR (101 MHz,

$CDCl_3$) δ 162.44, 146.02, 145.32, 129.42, 129.19, 127.41, 126.09, 123.69, 118.53, 68.58, 24.04, 18.91. Spectra data are consistent with those reported in the literature: *Org. Lett.* **2011**, *13*, 4581-4583.



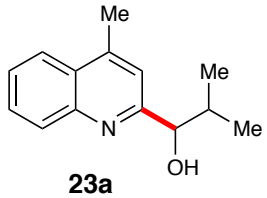
21

1-(4-Methylquinolin-2-yl)propan-1-ol (21): According to the general procedure, lepidine (71.6 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 μL, 1.0 mmol, 2.0 equiv.), 1-Propanol (0.75 ml, 10.0 mmol, 20.0 equiv.) were used. After 18 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (15% ethyl acetate/hexanes) to provide the title compound as a colorless oil (53.3 mg, 53% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (d, J = 8.4 Hz, 1H), 7.97 (d, J = 8.3 Hz, 1H), 7.70 (ddd, J = 8.4, 6.9, 1.4 Hz, 1H), 7.55 (ddd, J = 8.2, 6.9, 1.3 Hz, 1H), 7.17 (s, 1H), 5.03 (br s, 1H), 4.83 (dd, J = 7.1, 4.2 Hz, 1H), 2.70 (s, 3H), 2.03 – 1.94 (m, 1H), 1.76 (dp, J = 14.5, 7.3 Hz, 1H), 0.98 (t, J = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.50, 146.06, 145.09, 129.36, 129.21, 127.44, 126.05, 123.70, 118.88, 73.31, 30.83, 18.91, 9.28; HRMS (ESI) m/z calculated for $C_{13}H_{16}ON$ [$(M+H)^+$] 202.1226, found 202.1226. IR (film) 3370, 2965, 2931, 2875, 1603, 1567, 1509, 1449, 1411, 1095, 1050, 760 cm^{-1}. Spectra data are consistent with those reported in the literature: *Bull. Chem. Soc. Jpn*. **2004**, *77*, 553-559.

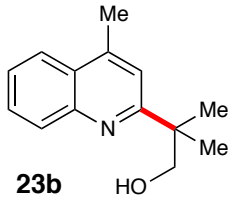


22

3-Fluoro-1-(4-methylquinolin-2-yl)propan-1-ol (22): According to the general procedure, lepidine (71.6 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 μL, 1.0 mmol, 2.0 equiv.), 3-fluoropropan-1-ol (0.75 ml, 10.0 mmol, 20.0 equiv.) were used. After 18 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (20% ethyl acetate/hexanes) to provide the title compound as a colorless oil (44.9 mg, 41% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (d, J = 7.9 Hz, 1H), 7.99 (d, J = 8.3 Hz, 1H), 7.72 (ddd, J = 8.4, 6.9, 1.5 Hz, 1H), 7.57 (ddd, J = 8.3, 6.8, 1.3 Hz, 1H), 7.20 (s, 1H), 5.03 (dd, J = 9.0, 3.3 Hz, 1H), 4.92 – 4.74 (m, 1H), 4.74 – 4.55 (m, 1H), 2.72 (s, 3H), 2.47 – 2.28 (m, 1H), 2.06 – 1.90 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.71, 146.12, 145.57, 129.58, 129.19, 127.59, 126.30, 123.80, 118.73, 81.12 (d), 68.73 (d), 39.02 (d), 18.95; HRMS (ESI) m/z calculated for $C_{18}H_{26}N_3$ [$(M+H)^+$] 284.2121, found 284.2119. IR (film) 3344, 2918, 2850, 1603, 1508, 1452, 1412, 1384, 1336, 1229, 1104, 1034, 760 cm^{-1}.



23a



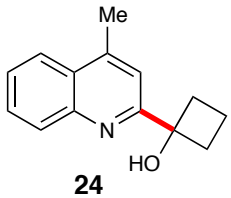
23b

2-Methyl-1-(4-methylquinolin-2-yl)propan-1-ol (23a) and 2-Methyl-2-(4-methylquinolin-2-yl)propan-1-ol (23b): According to the general procedure, lepidine (71.6 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0

equiv.), HCl (86.0 μL, 1.0 mmol, 2.0 equiv.), 2-methylpropan-1-ol (0.92 ml, 10.0 mmol, 20.0 equiv.) were used. After 18 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (20% ethyl acetate/hexanes) to provide the title compounds as an oil mixture of 62.4 mg, 58% total yield (33% yield for **23a**; 25% yield for **23b**). HRMS (ESI) m/z calculated for $C_{14}H_{18}NO$ [$(M+H)^+$] 216.1383, found 216.1381. IR (film) 3385, 2961, 2871, 1603, 1560, 1448, 1411, 1051, 1018, 759 cm^{-1}.

Compound **23a**: ^1H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, J = 8.3 Hz, 1H), 7.92 (d, J = 8.4 Hz, 1H), 7.68 – 7.60 (m, 1H), 7.54 – 7.45 (m, 1H), 7.13 (s, 1H), 5.02 (br s, 1H), 4.68 (d, J = 3.6 Hz, 1H), 2.65 (s, 3H), 2.21 – 2.08 (m, 1H), 1.11 (d, J = 6.9 Hz, 3H), 0.73 (d, J = 6.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.08, 145.91, 144.74, 129.18, 129.14, 127.34, 125.92, 123.60, 119.21, 76.74, 34.53, 19.83, 18.79, 15.47.

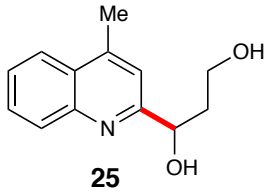
Compound **23b**: ^1H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, J = 8.4 Hz, 1H), 7.90 (d, J = 8.4 Hz, 1H), 7.68 – 7.60 (m, 1H), 7.54 – 7.45 (m, 1H), 7.24 (s, 1H), 3.86 (s, 2H), 2.65 (s, 3H), 1.37 (s, 6H); ^{13}C NMR (101 MHz, $cdcl_3$) δ 168.20, 145.96, 145.02, 129.24, 129.16, 126.54, 125.83, 123.41, 119.38, 71.71, 41.50, 25.54, 18.87.



24

1-(4-Methylquinolin-2-yl)cyclobutan-1-ol (24): According to the general procedure, lepidine (71.6 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 μL, 1.0 mmol, 2.0 equiv.), cyclobutanol (0.78 ml, 10.0 mmol, 20.0 equiv.) were used. After 18 hours, the reaction mixture was subjected to the workup

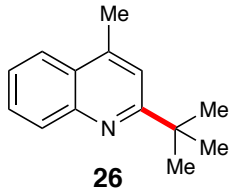
procedure outlined in the general procedure and purified by flash chromatography (10% ethyl acetate/hexanes) to provide the title compound as a light yellow solid (25.6 mg, 24% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, J = 8.4 Hz, 1H), 7.98 (d, J = 7.8 Hz, 1H), 7.70 (ddd, J = 8.4, 6.9, 1.5 Hz, 1H), 7.60 – 7.50 (m, 2H), 6.13 (br s, 1H), 2.77 (s, 3H), 2.72 – 2.54 (m, 4H), 2.25 – 2.10 (m, 1H), 2.08 – 1.95 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.65, 146.00, 145.24, 129.52, 129.24, 127.20, 126.10, 123.54, 117.28, 75.46, 37.44, 19.16, 13.30; HRMS (ESI) m/z calculated for $C_{18}H_{26}N_3$ [$(M+H)^+$] 284.2121, found 284.2119. IR (film) 3374, 2985, 2936, 1603, 1562, 1508, 1447, 1408, 1249, 1151, 1124, 1088, 759 cm^{-1}.



25

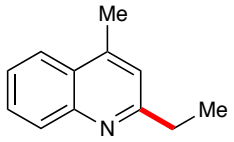
1-(4-Methylquinolin-2-yl)propane-1,3-diol (25): According to the general procedure, lepidine (71.6 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 μL, 1.0 mmol, 2.0 equiv.), propane-1,3-diol (0.72 ml, 10.0 mmol, 20.0 equiv.) were used. After 18 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (50% ethyl acetate/hexanes) to provide the title compound as a white solid (69.5 mg, 64% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, J = 8.4 Hz, 1H), 7.95 (d, J = 8.2 Hz, 1H), 7.68 (t, J = 7.6 Hz, 1H), 7.53 (t, J = 7.4 Hz, 1H), 7.25 (s, 1H), 5.11 (dd, J = 8.9, 3.6 Hz, 1H), 3.91 (t, J = 5.6 Hz, 2H), 2.67 (s, 3H), 2.24– 2.12 (m, 1H), 1.98 – 1.85 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.36, 146.03, 145.61, 129.48, 129.01, 127.45, 126.18, 123.71, 118.65, 72.17, 60.54, 39.70, 18.87; HRMS (ESI) m/z calculated for $C_{13}H_{16}NO_2$

[(M+H)$^+$] 218.1176, found 218.1174. IR (film) 3385, 2925, 1691, 1603, 1567, 1510, 1447, 1413, 1054, 909, 760 cm^{-1}.



26

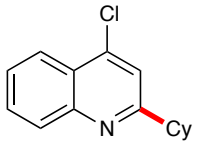
2-(*tert*-Butyl)-4-methylquinoline (26): According to the general procedure, lepidine (71.6 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 µL, 1.0 mmol, 2.0 equiv.), pivalaldehyde (1.09 ml, 10.0 mmol, 20.0 equiv.) were used. After 18 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (10% ethyl acetate/hexanes) to provide the title compound as a colorless oil (95.7 mg, 96% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (d, J = 8.4 Hz, 1H), 7.95 (d, J = 8.3 Hz, 1H), 7.67 (ddd, J = 8.4, 6.9, 1.5 Hz, 1H), 7.50 (ddd, J = 8.3, 6.8, 1.3 Hz, 1H), 7.37 (s, 1H), 2.70 (s, 3H), 1.49 (s, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.86, 147.22, 143.59, 129.86, 128.65, 126.49, 125.34, 123.33, 118.84, 37.86, 30.08, 18.91. Spectra data are consistent with those reported in the literature: *Org. Lett*. **2018**, *20*, 3229-3232.



27

2-Ethyl-4-methylquinoline (27): According to the general procedure, lepidine (71.6 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 µL, 1.0 mmol, 2.0 equiv.), propionaldehyde (0.72 ml, 10.0 mmol, 20.0 equiv.) were used. After 18 hours, the reaction mixture was subjected to the workup procedure

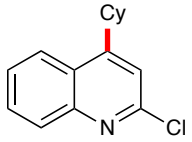
outlined in the general procedure and purified by flash chromatography (10% ethyl acetate/hexanes) to provide the title compound as a colorless oil (47.9 mg, 59% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 8.3 Hz, 1H), 7.94 (d, *J* = 8.3 Hz, 1H), 7.66 (ddd, *J* = 8.4, 6.9, 1.5 Hz, 1H), 7.49 (ddd, *J* = 8.2, 6.8, 1.3 Hz, 1H), 7.15 (s, 1H), 2.95 (q, *J* = 7.7 Hz, 2H), 2.67 (s, 3H), 1.38 (t, *J* = 7.7 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.64, 147.56, 144.39, 129.20, 129.02, 126.75, 125.39, 123.54, 121.50, 32.13, 18.66, 14.02. Spectra data are consistent with those reported in the literature: *Org. Lett*. **2018**, *20*, 3229-3232.



28

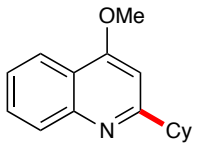
4-Chloro-2-cyclohexylquinoline (28): According to the general procedure, 4-chloroquinoline (81.8 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 µL, 1.0 mmol, 2.0 equiv.), cyclohexane (1.10 ml, 10.0 mmol, 20.0 equiv.) were used. After 24 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (10% ethyl acetate/hexanes) to provide the title compound as a colorless oil (103.3 mg, 84% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (d, *J* = 8.4 Hz, 1H), 8.04 (d, *J* = 8.2 Hz, 1H), 7.69 (ddd, *J* = 8.5, 6.9, 1.5 Hz, 1H), 7.52 (ddd, *J* = 8.2, 6.9, 1.2 Hz, 1H), 7.39 (s, 1H), 2.86 (tt, *J* = 12.0, 3.5 Hz, 1H), 2.05 – 1.95 (m, 2H), 1.92 – 1.82 (m, 2H), 1.81 – 1.70 (m, 1H), 1.65 – 1.51 (m, 2H), 1.50 – 1.37 (m, 2H), 1.36 – 1.23 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 166.64, 148.53, 142.46, 130.01, 129.18, 126.44, 124.98,

123.73, 119.66, 47.25, 32.55, 26.31, 25.88. Spectra data are consistent with those reported in the literature: *Org. Lett*. **2018**, *20*, 4686-4690.



29

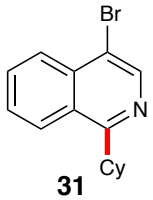
2-Chloro-4-cyclohexylquinoline (29): According to the general procedure, 2-chloroquinoline (81.8 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 µL, 1.0 mmol, 2.0 equiv.), cyclohexane (1.10 ml, 10.0 mmol, 20.0 equiv.) were used. After 24 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (10% ethyl acetate/hexanes) to provide the title compound as a white solid (116.7 mg, 95% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (dd, J = 8.4, 1.6 Hz, 2H), 7.66 (ddd, J = 8.4, 6.8, 1.4 Hz, 1H), 7.51 (ddd, J = 8.3, 6.8, 1.3 Hz, 1H), 7.22 (s, 1H), 3.24 (tt, J = 11.4, 3.0 Hz, 1H), 2.00 – 1.92 (m, 2H), 1.92 – 1.86 (m, 2H), 1.86 – 1.78 (m, 1H), 1.56 – 1.41 (m, 4H), 1.36 – 1.24 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 156.78, 150.93, 148.02, 129.79, 129.33, 126.39, 125.45, 123.02, 118.54, 38.93, 33.24, 26.62, 25.99. Spectra data are consistent with those reported in the literature: *Angew. Chem. Int. Ed.* **2013**, *52*, 3267-3271.



30

2-Cyclohexyl-4-methoxyquinoline (30): According to the general procedure, 4-methoxyquinoline (79.6 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL,

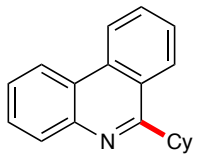
1.0 mmol, 2.0 equiv.), HCl (86.0 μL, 1.0 mmol, 2.0 equiv.), cyclohexane (1.10 ml, 10.0 mmol, 20.0 equiv.) were used. After 24 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (10% ethyl acetate/hexanes) to provide the title compound as a colorless oil (51.9 mg, 43% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 (d, J = 8.3 Hz, 1H), 7.98 (d, J = 8.3 Hz, 1H), 7.63 (ddd, J = 8.4, 6.8, 1.5 Hz, 1H), 7.41 (ddd, J = 8.2, 6.9, 1.2 Hz, 1H), 6.63 (s, 1H), 4.01 (s, 3H), 2.87 (tt, J = 12.0, 3.4 Hz, 1H), 2.11 – 1.95 (m, 2H), 1.94 – 1.82 (m, 2H), 1.82 – 1.74 (m, 1H), 1.62 (qd, J = 12.4, 3.1 Hz, 2H), 1.53 – 1.40 (m, 2H), 1.40 – 1.28 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.05, 162.36, 148.55, 129.47, 128.28, 124.62, 121.46, 120.16, 97.84, 55.34, 48.13, 32.83, 26.46, 26.01. Spectra data are consistent with those reported in the literature: *Org. Lett.* **2019**, *21*, DOI: acs.orglett.9b01635.



31

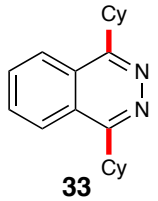
4-Bromo-1-cyclohexylisoquinoline (31): According to the general procedure, 4-bromoisoquinoline (104.0 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 μL, 1.0 mmol, 2.0 equiv.), cyclohexane (1.10 ml, 10.0 mmol, 20.0 equiv.) were used. After 18 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (10% ethyl acetate/hexanes) to provide the title compound as a colorless oil (105.9 mg, 73% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.65 (s, 1H), 8.22 (d, J = 8.5 Hz, 1H), 8.18 (d, J = 8.4 Hz, 1H), 7.76 (ddd, J = 8.3, 6.8, 1.2 Hz, 1H), 7.64 (ddd, J = 8.3,

6.9, 1.3 Hz, 1H), 3.52 (tt, *J* = 11.7, 3.2 Hz, 1H), 2.02 – 1.88 (m, 4H), 1.86 – 1.75 (m, 3H), 1.60 – 1.45 (m, 2H), 1.44 – 1.33 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.31, 143.52, 134.87, 130.79, 127.71, 127.57, 126.83, 125.05, 117.50, 41.44, 32.50, 26.75, 26.12. Spectra data are consistent with those reported in the literature: *Adv. Synth. Catal*. **2015**, *357*, 2055-2060.



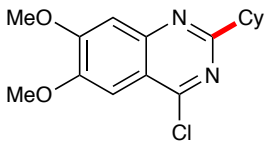
32

6-Cyclohexylphenanthridine (32): According to the general procedure, phenanthridine (89.6 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 μL, 1.0 mmol, 2.0 equiv.), cyclohexane (1.10 ml, 10.0 mmol, 20.0 equiv.) were used. After 24 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (5% ethyl acetate/hexanes) to provide the title compound as a colorless oil (112.4 mg, 86% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.65 (d, *J* = 8.2 Hz, 1H), 8.54 (d, *J* = 8.1 Hz, 1H), 8.32 (d, *J* = 8.2 Hz, 1H), 8.14 (d, *J* = 8.1 Hz, 1H), 7.81 (t, *J* = 7.6 Hz, 1H), 7.70 (q, *J* = 6.7 Hz, 2H), 7.61 (t, *J* = 7.6 Hz, 1H), 3.62 (tt, *J* = 11.3, 3.4 Hz, 1H), 2.14 – 2.03 (m, 2H), 2.03 – 1.89 (m, 4H), 1.89 – 1.81 (m, 1H), 1.63 – 1.51 (m, 2H), 1.50 – 1.41 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.27, 143.83, 132.98, 129.89, 129.88, 128.35, 127.03, 126.10, 125.59, 124.69, 123.31, 122.54, 121.78, 41.95, 32.26, 26.85, 26.28. Spectra data are consistent with those reported in the literature: *J. Org. Chem*. **2018**, *83*, 10015-10024.



33

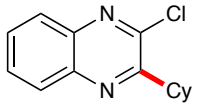
1,4-Dicyclohexylphthalazine (33): According to the general procedure, phthalazine (65.1 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 μL, 1.0 mmol, 2.0 equiv.), cyclohexane (1.10 ml, 10.0 mmol, 20.0 equiv.) were used. After 18 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (20% ethyl acetate/hexanes) to provide the title compound as a yellow solid (136.7 mg, 43% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (dd, J = 6.3, 3.4 Hz, 2H), 7.78 (dd, J = 6.3, 3.3 Hz, 2H), 3.41 (tt, J = 11.2, 3.6 Hz, 2H), 2.06 – 1.96 (m, 4H), 1.96 – 1.83 (m, 8H), 1.96 – 1.83 (m, 2H), 1.80 – 1.71 (m, 4H), 1.39 – 1.26 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.57, 130.94, 124.70, 123.96, 40.22, 32.14, 26.69, 26.06. Spectra data are consistent with those reported in the literature: *Angew. Chem. Int. Ed.* **2013**, *52*, 3267-3271.



34

4-Chloro-2-cyclohexyl-6,7-dimethoxyquinazoline (34): According to the general procedure, 4-chloro-6,7-dimethoxyquinazoline (112.3 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 μL, 1.0 mmol, 2.0 equiv.), cyclohexane (1.10 ml, 10.0 mmol, 20.0 equiv.) were used. After 24 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (50% ethyl acetate/hexanes) to provide the title

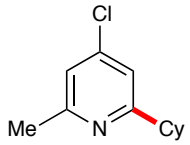
compound as a colorless oil (104.4 mg, 68% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 (s, 1H), 7.24 (s, 1H), 4.01 (s, 6H), 2.89 (tt, *J* = 11.8, 3.5 Hz, 1H), 2.08 – 1.95 (m, 2H), 1.90 – 1.81 (m, 2H), 1.75 – 1.61 (m, 3H), 1.45 – 1.24 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.87, 159.09, 156.44, 150.52, 149.34, 117.31, 106.65, 102.70, 56.50, 56.28, 47.30, 31.80, 26.19, 25.86; HRMS (ESI) m/z calculated for $C_{16}H_{20}O_2N_2Cl$ [$(M+H)^+$] 307.1208, found 307.1206; IR (film) 2924, 2848, 1619, 1570, 1501, 1416, 1293, 1239, 1220, 1162, 848 cm^{-1}. Spectra data are consistent with those reported in the literature: *Org. Lett*. **2019**, *21*, DOI: acs.orglett.9b01635.



35

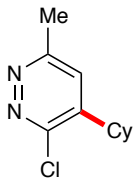
2-Chloro-3-cyclohexylquinoxaline (35): According to the general procedure, 2-chloro-quinoxaline (82.3 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 μL, 1.0 mmol, 2.0 equiv.), cyclohexane (1.10 ml, 10.0 mmol, 20.0 equiv.) were used. After 24 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (10% ethyl acetate/hexanes) to provide the title compound as a white solid (40.7 mg, 33% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 – 8.00 (m, 1H), 8.00 – 7.92 (m, 1H), 7.76 – 7.64 (m, 2H), 3.34 (tt, *J* = 11.6, 3.3 Hz, 1H), 2.07 – 1.97 (m, 2H), 1.97 – 1.86 (m, 2H), 1.85 – 1.76 (m, 1H), 1.70 (qd, *J* = 12.6, 3.2 Hz, 2H), 1.48 (qt, *J* = 12.7, 3.3 Hz, 2H), 1.35 (qt, *J* = 12.8, 3.3 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.09, 147.40, 141.11, 140.55, 129.77 (2C), 128.74, 128.00, 42.49, 31.20, 26.34, 25.90. Spectra

data are consistent with those reported in the literature: *Angew. Chem. Int. Ed.* **2013**, *52*, 3267-3271.



36

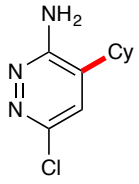
4-Chloro-2-cyclohexyl-6-methylpyridine (36): According to the general procedure, 4-chloro-2-methylpyridine (63.8 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 μL, 1.0 mmol, 2.0 equiv.), cyclohexane (1.10 ml, 10.0 mmol, 20.0 equiv.) were used. The reaction mixture was irradiated under 390 nm blue LEDs, after 24 hours, subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (10% ethyl acetate/hexanes) to provide the title compound as a colorless oil (67.1 mg, 64% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.96 (s, 2H), 2.64 (ddt, *J* = 11.5, 6.7, 3.4 Hz, 1H), 2.49 (s, 3H), 1.99 – 1.90 (m, 2H), 1.86 – 1.78 (m, 2H), 1.77 – 1.68 (m, 1H), 1.47 – 1.31 (m, 4H), 1.31 – 1.22 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.64, 158.94, 144.21, 120.71, 117.91, 46.50, 32.85, 26.42, 25.95, 24.35. Spectra data are consistent with those reported in the literature: *Org. Lett.* **2019**, *21*, DOI: acs.orglett.9b01635.



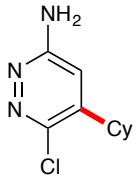
37

3-Chloro-4-cyclohexyl-6-methylpyridazine (37): According to the general procedure, 3-chloro-6-methylpyridazine (64.3 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2

(0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 µL, 1.0 mmol, 2.0 equiv.), cyclohexane (1.10 ml, 10.0 mmol, 20.0 equiv.) were used. The reaction mixture was irradiated under 390 nm blue LEDs, after 18 hours, subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (20% ethyl acetate/hexanes) to provide the title compound as a light yellow solid (86.4 mg, 82% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.14 (s, 1H), 2.82 (tt, J = 11.8, 2.9 Hz, 1H), 2.63 (s, 3H), 1.95 – 1.81 (m, 4H), 1.81 – 1.72 (m, 1H), 1.42 (qt, J = 12.7, 3.5 Hz, 2H), 1.35 – 1.19 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.63, 155.42, 145.46, 125.98, 39.53, 31.72, 26.15, 25.65, 21.50. Spectra data are consistent with those reported in the literature: *J. Heterocyclic Chem*. **1991**, *28*, 583-587.



38a



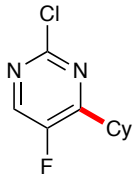
38b

6-Chloro-4-cyclohexylpyridazin-3-amine (38a) and **6-Chloro-5-cyclohexylpyridazin-3-amine (38b):** According to the general procedure, 6-chloropyridazin-3-amine (64.8 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 µL, 1.0 mmol, 2.0 equiv.), cyclohexane (1.10 ml, 10.0 mmol, 20.0 equiv.) were used. After 18 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (50% ethyl acetate/hexanes) to provide the title compounds as a white solid (70.9 mg, 67% yield for **38a**) and a white solid (14.8 mg, 14% yield for **38b**).

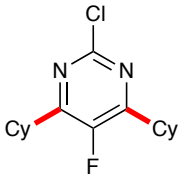
Compound **38a**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.03 (s, 1H), 5.58 (br s, 2H), 2.30 (tt, J = 11.4, 3.0 Hz, 1H), 1.97 – 1.82 (m, 4H), 1.82 – 1.73 (m, 1H), 1.45 – 1.20 (m, 5H); ^{13}C

NMR (101 MHz, $CDCl_3$) δ 158.18, 147.90, 135.88, 125.07, 37.89, 31.09, 26.22, 25.77; HRMS (ESI) m/z calculated for $C_{10}H_{15}N_3Cl$ [$(M+H)^+$] 212.0949, found 212.0949. IR (film) 3377, 3312, 3176, 2933, 2853, 1658, 1625, 1599, 1456, 1209, 1150, 962 cm^{-1}. Spectra data are consistent with those reported in the literature: *Tetrahedron Lett.* **2015**, *56*, 6791-6794.

Compound **38b**: ^1H NMR (400 MHz, $CDCl_3$) δ 6.64 (s, 1H), 5.06 (br s, 2H), 2.75 (tt, J = 11.9, 3.0 Hz, 1H), 1.96 – 1.82 (m, 4H), 1.82 – 1.75 (m, 1H), 1.49 – 1.35 (m, 2H), 1.30 – 1.20 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 159.71, 148.73, 147.27, 113.86, 39.73, 31.86, 26.28, 25.83; HRMS (ESI) m/z calculated for $C_{10}H_{15}N_3Cl$ [$(M+H)^+$] 212.0949, found 212.0948. IR (film) 3318, 3160, 2930, 2855, 1658, 1635, 1581, 1533, 1438, 1344, 1107, 935 cm^{-1}.



39a

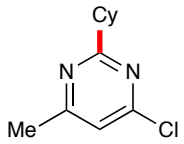


39b

2-Chloro-4-cyclohexyl-5-fluoro-pyrimidine (39a) and 2-Chloro-4,6-dicyclohexyl-5-fluoropyrimidine (39b): According to the general procedure, 2-chloro-5-fluoropyrimidine (68.3 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 μL, 1.0 mmol, 2.0 equiv.), cyclohexane (1.10 ml, 10.0 mmol, 20.0 equiv.) were used. The reaction mixture was irradiated under 390 nm blue LEDs, after 18 hours, subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (10% ethyl acetate/hexanes) to provide the title compounds as a colorless oil (54.7 mg, 51% yield for **39a**) and a white solid (26.7 mg, 18% yield for **39b**).

Compound **39a**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.31 (d, *J* = 1.4 Hz, 1H), 3.05 – 2.92 (m, 1H), 1.91 – 1.76 (m, 4H), 1.76 – 1.69 (m, 1H), 1.69 – 1.57 (m, 2H), 1.44 – 1.22 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.60 (d), 155.25 (d), 155.07 (d), 146.02 (d), 39.32 (d), 30.24 (d), 25.87, 25.45; HRMS (ESI) m/z calculated for $C_{16}H_{23}N_2ClF$ [$(M+H)^+$] 215.0746, found 215.0745. IR (film) 2932, 2855, 1578, 1450, 1413, 1344, 1202, 923, 780, 658 cm^{-1}.

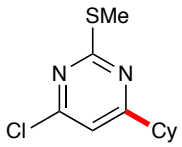
Compound **39b**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.02 – 2.91 (m, 2H), 1.92 – 1.82 (m, 4H), 1.82 – 1.70 (m, 6H), 1.70 – 1.58 (m, 4H), 1.44 – 1.25 (m, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.09 (d), 154.53 (d), 154.00, 151.43, 39.12 (d), 30.43 (d), 26.01, 25.55. HRMS (ESI) m/z calculated for $C_{16}H_{23}N_2ClF$ [$(M+H)^+$] 297.1528, found 297.1527. IR (film) 2931, 2852, 1589, 1453, 1405, 1362, 1308, 1254, 1008, 924 cm^{-1}.



40

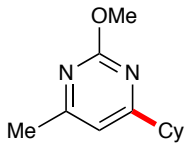
4-Chloro-2-cyclohexyl-6-methylpyrimidine (40): According to the general procedure, 4-chloro-6-methylpyrimidine (64.3 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 μL, 1.0 mmol, 2.0 equiv.), cyclohexane (1.10 ml, 10.0 mmol, 20.0 equiv.) were used. The reaction mixture was irradiated under 390 nm blue LEDs, after 24 hours, subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (20% ethyl acetate/hexanes) to provide the title compound as a white solid (80.0 mg, 76% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.98 (s, 1H), 2.77 (tt, *J* = 11.9, 3.5 Hz, 1H), 2.44 (s, 3H), 1.98 – 1.86 (m, 2H), 1.84 – 1.75 (m, 2H), 1.72 – 1.64 (m, 1H), 1.58 (qd, *J* = 12.4, 2.9 Hz, 2H), 1.39 – 1.21 (m, 3H); ^{13}C

NMR (101 MHz, $CDCl_3$) δ 175.22, 168.50, 160.87, 117.74, 47.23, 31.56, 26.00, 25.71, 23.86. Spectra data are consistent with those reported in the literature: *Org. Lett.* **2019**, *21*, DOI: acs.orglett.9b01635.



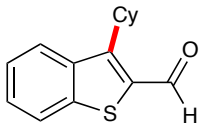
41

4-Chloro-6-cyclohexyl-2-(methylthio)pyrimidine (41): According to the general procedure, 4-chloro-2-(methylthio)pyrimidine (121.4 mg, 0.5 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 μL, 1.0 mmol, 2.0 equiv.), cyclohexane (1.10 ml, 10.0 mmol, 20.0 equiv.) were used. The reaction mixture was irradiated under 390 nm blue LEDs, after 18 hours, subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (10% ethyl acetate/hexanes) to provide the title compound as a colorless oil (104.4 mg, 86% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.81 (s, 1H), 2.58 (tt, J = 11.6, 3.3 Hz, 1H), 2.54 (s, 3H), 1.96 – 1.87 (m, 2H), 1.87 – 1.78 (m, 2H), 1.78 – 1.70 (m, 1H), 1.53 – 1.41 (m, 2H), 1.41 – 1.30 (m, 2H), 1.29 – 1.19 (m, 1H); ^{13}C NMR (101 MHz, $cdcl_3$) δ 176.50, 172.82, 160.82, 113.48, 45.65, 31.67, 26.00, 25.72, 14.20; HRMS (ESI) m/z calculated for $C_{11}H_{16}N_2ClS$ [$(M+H)^+$] 243.0717, found 243.0716. IR (film) 2928, 2853, 1552, 1449, 1371, 1323, 1275, 1230, 1131, 930, 826 cm^{-1}.



42

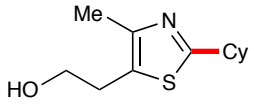
4-Cyclohexyl-2-methoxy-6-methylpyrimidine (42): According to the general procedure, 2-methoxy-4-methylpyrimidine (62.1 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 μL, 1.0 mmol, 2.0 equiv.), cyclohexane (1.10 ml, 10.0 mmol, 20.0 equiv.) were used. The reaction mixture was irradiated under 390 nm blue LEDs, after 18 hours, subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (20% ethyl acetate/hexanes) to provide the title compound as a colorless oil (59.8 mg, 58% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.59 (s, 1H), 3.93 (s, 3H), 2.49 (tt, *J* = 11.8, 3.4 Hz, 1H), 2.36 (s, 3H), 1.90 – 1.82 (m, 2H), 1.82 – 1.74 (m, 2H), 1.72 – 1.64 (m, 1H), 1.46 (qd, *J* = 12.2, 2.5 Hz, 2H), 1.38 – 1.26 (m, 2H), 1.26 – 1.15 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.83, 169.10, 165.25, 111.42, 54.25, 45.56, 31.74, 26.10, 25.81, 23.87. Spectra data are consistent with those reported in the literature: *Org. Lett.* **2019**, *21*, DOI: acs.orglett.9b01635.



43

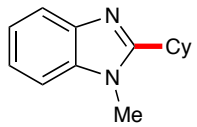
3-Cyclohexylbenzo[*b*]thiophene-2-carbaldehyde (43): According to the general procedure, benzo[*b*]thiophene-2-carbaldehyde (81.1 mg, 0.5mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 μL, 1.0 mmol, 2.0 equiv.), cyclohexane (1.10 ml, 10.0 mmol, 20.0 equiv.) were used. The reaction mixture was irradiated under 390 nm blue LEDs, after 18 hours, subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (10% ethyl acetate/hexanes) to provide the title compound as a light yellow oil (55.0 mg, 45% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.49 (s, 1H), 8.08 (d, *J* = 8.2 Hz, 1H), 7.86 (d, *J* = 8.0 Hz, 1H), 7.52 – 7.43 (m, 1H), 7.40 (ddd, *J* = 8.2, 7.0, 1.2 Hz, 1H), 3.54 (p, *J* = 8.1 Hz, 1H), 2.07 – 1.92 (m, 6H), 1.92 – 1.84 (m, 1H), 1.58 – 1.35 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 184.26, 152.08, 142.79, 138.92, 138.01, 127.77, 125.58, 124.31, 123.45, 39.43, 33.58, 27.03, 25.97; HRMS (EI) m/z calculated for $C_{15}H_{16}OS$ [M+] 244.0922, found 244.0929; IR (film) 2922, 2852, 1684, 1655, 1613, 1518, 1201, 759, 726 cm^{-1}.



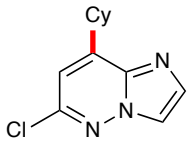
44

2-(2-Cyclohexyl-4-methylthiazol-5-yl)ethanol (44): According to the general procedure, 2-(4-methylthiazol-5-yl)ethanol (71.6 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 μL, 1.0 mmol, 2.0 equiv.), cyclohexane (1.10 ml, 10.0 mmol, 20.0 equiv.) were used. The reaction mixture was irradiated under 390 nm blue LEDs, after 18 hours, subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (50% ethyl acetate/hexanes) to provide the title compound as a light yellow oil (46.2 mg, 41% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.77 (t, *J* = 6.5 Hz, 2H), 2.92 (t, *J* = 6.5 Hz, 2H), 2.86 (tt, *J* = 11.5, 3.6 Hz, 1H), 2.55 (br s, 1H), 2.29 (s, 3H), 2.09 – 2.03 (m, 2H), 1.84 – 1.76 (m, 2H), 1.72 – 1.67 (m, 1H), 1.49 – 1.29 (m, 4H), 1.28 – 1.19 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.82, 147.64, 125.80, 62.82, 42.55, 33.75, 29.79, 26.06, 25.71, 14.80. Spectra data are consistent with those reported in the literature: *Org. Lett.* **2019**, *21*, DOI: acs.orglett.9b01635.



45

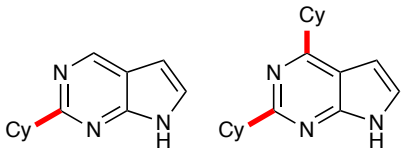
2-Cyclohexyl-1-methyl-1*H*-benzo[*d*]imidazole (45): According to the general procedure, 1-methyl-1*H*-benzo[*d*]imidazole (66.1 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 µL, 1.0 mmol, 2.0 equiv.), cyclohexane (1.10 ml, 10.0 mmol, 20.0 equiv.) were used. The reaction mixture was irradiated under 390 nm blue LEDs, after 18 hours, subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (50% ethyl acetate/hexanes) to provide the title compound as a white solid (44.9 mg, 41% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 – 7.65 (m, 1H), 7.24 – 7.20 (m, 1H), 7.20 – 7.13 (m, 2H), 3.66 (s, 3H), 2.78 (tt, *J* = 11.8, 3.5 Hz, 1H), 1.98 – 1.89 (m, 2H), 1.89 – 1.81 (m, 2H), 1.80 – 1.68 (m, 3H), 1.43 – 1.26 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 158.80, 141.72, 135.25, 122.06, 121.90, 118.92, 108.87, 36.31, 31.23, 29.53, 26.24, 25.68. Spectra data are consistent with those reported in the literature: *Org. Lett.* **2017**, *19*, 6594-6597.



46

6-Chloro-8-cyclohexylimidazo[1,2-*b*]pyridazine (46) : According to the general procedure, 6-chloroimidazo[1,2-*b*]pyridazine (76.8 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 µL, 1.0 mmol, 2.0 equiv.), cyclohexane (1.10 ml, 10.0 mmol, 20.0 equiv.) were used. After 18 hours, the reaction

mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (20% ethyl acetate/hexanes) to provide the title compounds as a white solid (69.5 mg, 59% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 1.2 Hz, 1H), 7.70 (d, *J* = 1.3 Hz, 1H), 6.84 (d, *J* = 0.8 Hz, 1H), 3.34 (tt, *J* = 11.6, 3.9 Hz, 1H), 2.12 – 2.01 (m, 2H), 1.93 – 1.83 (m, 2H), 1.83 – 1.76 (m, 1H), 1.58 – 1.43 (m, 4H), 1.36 – 1.25 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 148.04, 147.31, 137.87, 133.09, 117.16, 114.55, 38.70, 31.99, 26.20, 25.91. Spectra data are consistent with those reported in the literature: *Angew. Chem. Int. Ed*. **2017**, *56*, 12336-12339.



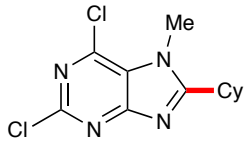
47a **47b**

4-cyclohexyl-7*H*-pyrrolo[2,3-*d*]pyrimidine (47a)and 2,4-dicyclohexyl-7*H*-pyrrolo[2,3-*d*]pyrimidine (47b): According to the general procedure, 7*H*-pyrrolo[2,3-*d*]pyrimidine (59.5 mg, 0.5 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 µL, 1.0 mmol, 2.0 equiv.), cyclohexane (1.10 ml, 10.0 mmol, 20.0 equiv.) were used. The reaction mixture was irradiated under 390 nm blue LEDs, after 18 hours, subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (50% ethyl acetate/hexanes) to provide the title compounds as a white solid (44.3 mg, 44% yield for **47a**) and a white solid (24.1 mg, 17% yield for **47b**).

Compound **47a**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.07 (s, 1H), 8.86 (s, 1H), 7.35 (d, *J* = 3.6 Hz, 1H), 6.64 (d, *J* = 3.6 Hz, 1H), 3.11 (tt, *J* = 11.8, 3.5 Hz, 1H), 2.06 – 1.94 (m, 2H), 1.94 – 1.87 (m, 2H), 1.87 – 1.70 (m, 3H), 1.54 – 1.30 (m, 3H); ^{13}C NMR (101 MHz,

$CDCl_3$) δ 167.23, 151.62, 150.77, 124.79, 116.48, 99.73, 44.30, 31.57, 26.42, 25.98; HRMS (ESI) m/z calculated for $C_{12}H_{16}N_3$ [$(M+H)^+$] 202.1339, found 202.1337. IR (film) 3132, 2925, 2852, 1588, 1505, 1425, 1348, 1254, 1110, 899, 733 cm^{-1}.

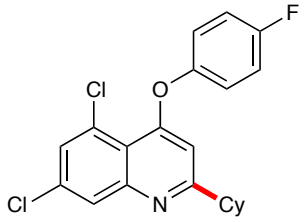
Compound **47b**: ^1H NMR (400 MHz, $CDCl_3$) δ 11.41 (s, 1H), 7.24 (dd, *J* = 3.6, 2.0 Hz, 1H), 6.61 (dd, *J* = 3.6, 1.6 Hz, 1H), 3.15 – 2.87 (m, 2H), 2.16 – 2.05 (m, 2H), 1.98 – 1.68 (m, 12H), 1.56 – 1.33 (m, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.12, 167.05, 152.59, 123.60, 113.88, 99.85, 47.50, 45.20, 32.58, 31.59, 26.45 (2C), 26.23, 26.09; HRMS (ESI) m/z calculated for $C_{18}H_{26}N_3$ [$(M+H)^+$] 284.2121, found 284.2119. IR (film) 3116, 2928, 2850, 1604, 1574, 1501, 1450, 1396, 1295, 1254, 1114, 896 cm^{-1}.



48

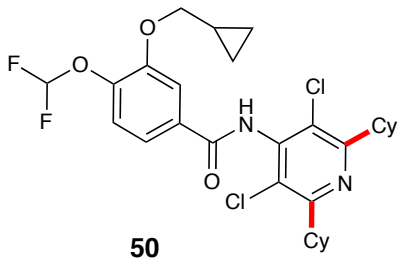
2,6-Dichloro-8-cyclohexyl-7-methyl-7*H*-purine (48): According to the general procedure, 2,6-dichloro-7-methyl-7*H*-purine (101.5 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 μL, 1.0 mmol, 2.0 equiv.), cyclohexane (1.10 ml, 10.0 mmol, 20.0 equiv.) were used. The reaction mixture was irradiated under 390 nm blue LEDs, after 18 hours, subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (50% ethyl acetate/hexanes) to provide the title compound as a white solid (122.6 mg, 86% yield). ^1H NMR (400 MHz, $CDCl_3$) δ 4.05 (s, 3H), 2.88 (tt, *J* = 11.6, 3.4 Hz, 1H), 1.96 – 1.86 (m, 4H), 1.83 – 1.72 (m, 3H), 1.46 – 1.30 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.54, 162.81, 152.30, 142.01, 122.68, 36.45, 32.06, 30.76, 25.74, 25.39. Spectra data are

consistent with those reported in the literature: *Org. Lett*. **2019**, *21*, DOI: acs.orglett.9b01635.



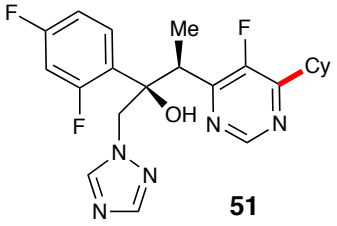
49

5,7-Dichloro-2-cyclohexyl-4-(4-fluorophenoxy)quinoline (49): According to the general procedure, 5,7-dichloro-4-(4-fluorophenoxy)quinoline (154.1 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 μL, 1.0 mmol, 2.0 equiv.), cyclohexane (1.10 ml, 10.0 mmol, 20.0 equiv.) were used. After 24 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (10% ethyl acetate/hexanes) to provide the title compound as a colorless oil (136.7 mg, 70% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, J = 2.1 Hz, 1H), 7.45 (d, J = 2.1 Hz, 1H), 7.18 – 7.04 (m, 4H), 6.53 (s, 1H), 2.68 (tt, J = 11.7, 3.4 Hz, 1H), 1.94 – 1.84 (m, 2H), 1.84 – 1.76 (m, 2H), 1.75 – 1.67 (m, 1H), 1.50 – 1.30 (m, 4H), 1.29 – 1.18 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.42, 162.18, 159.73 (d), 151.29, 150.25 (d), 134.59, 129.77, 128.44, 127.41, 121.84 (d), 116.90 (d), 116.88, 105.66, 47.12, 32.24, 26.21, 25.77. Spectra data are consistent with those reported in the literature: *Asian J. Org. Chem*. **2018**, *7*, 1307-1310.



50

3-(Cyclopropylmethoxy)-*N*-(3,5-dichloro-2,6-dicyclohexylpyridin-4-yl)-4-(difluoromethoxy)benzamide (50): According to the general procedure, 3-(cyclopropylmethoxy)-*N*-(3,5-dichloropyridin-4-yl)-4-(difluoromethoxy)benzamide (201.6 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl (86.0 μL, 1.0 mmol, 2.0 equiv.), cyclohexane (1.10 ml, 10.0 mmol, 20.0 equiv.) were used. After 18 hours, the reaction mixture was subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (20% ethyl acetate/hexanes) to provide the title compound as a white solid (136.1 mg, 48% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (s, 1H), 7.57 (d, *J* = 2.1 Hz, 1H), 7.45 (dd, *J* = 8.2, 2.0 Hz, 1H), 7.21 (d, *J* = 8.2 Hz, 1H), 6.72 (t, *J* = 75.0 Hz, 1H), 3.91 (d, *J* = 6.9 Hz, 2H), 3.12 (tt, *J* = 11.3, 3.3 Hz, 2H), 1.89 – 1.71 (m, 10H), 1.70 – 1.57 (m, 4H), 1.47 – 1.25 (m 7H), 0.72 – 0.59 (m, 2H), 0.40 – 0.29 (m, 2H); ^{13}C NMR (101 MHz, cdcl$_3$) δ 164.17, 160.57, 150.67, 143.45, 138.65, 131.35, 124.57, 122.11, 119.81, 115.67 (t), 114.07, 74.01, 42.31, 31.20, 26.34, 25.97, 9.93, 3.19; HRMS (ESI) m/z calculated for $C_{29}H_{35}O_3N_2Cl_2F_2$ [$(M+H)^+$] 567.1987, found 567.1987. IR (film) 3245, 2929, 2853, 1660, 1605, 1591, 1548, 1493, 1291, 1200, 1139, 1061 cm^{-1}.

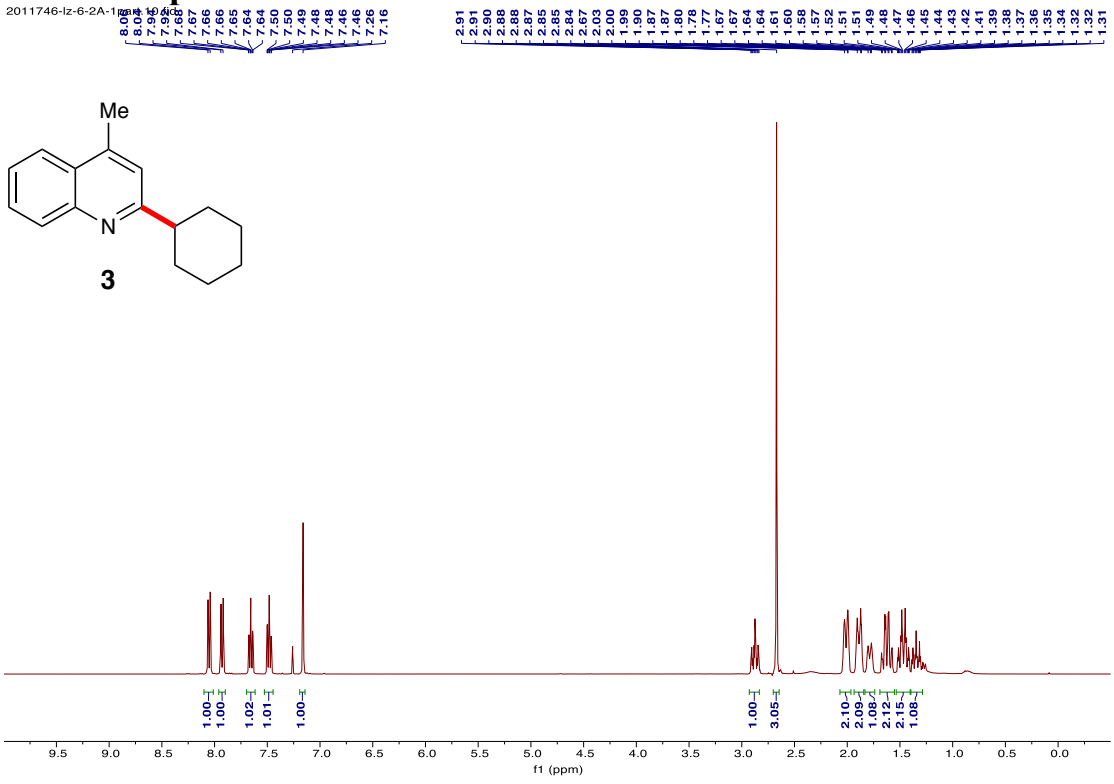


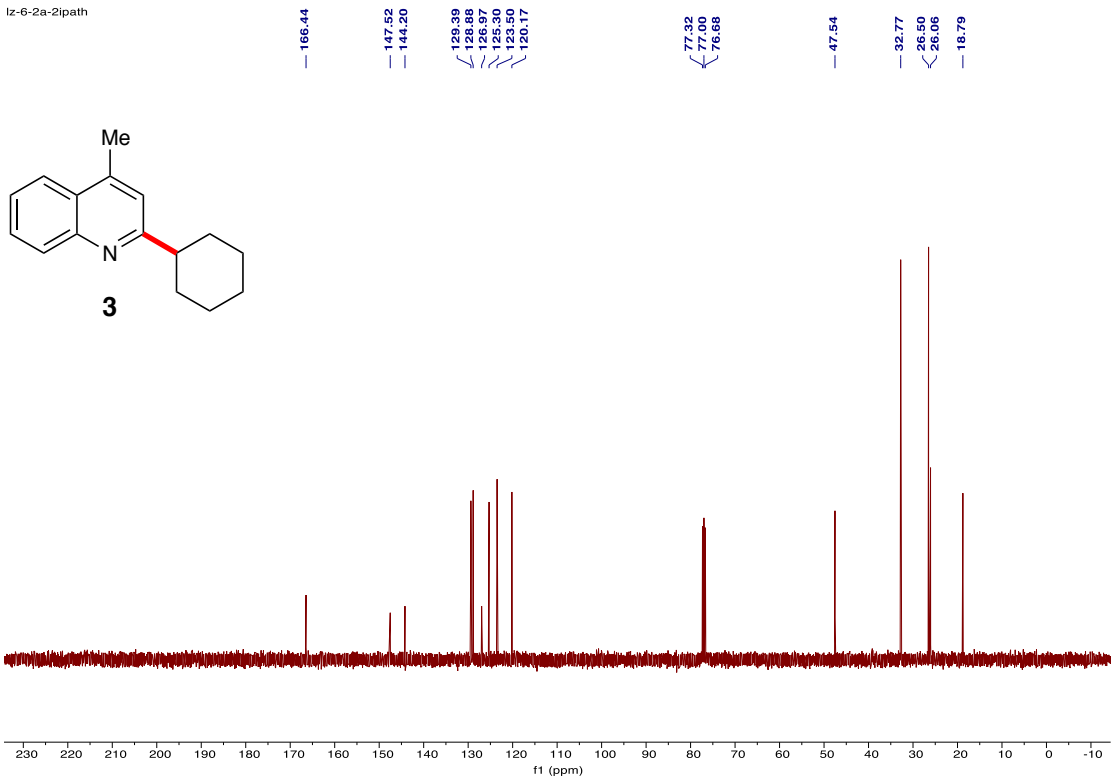
51

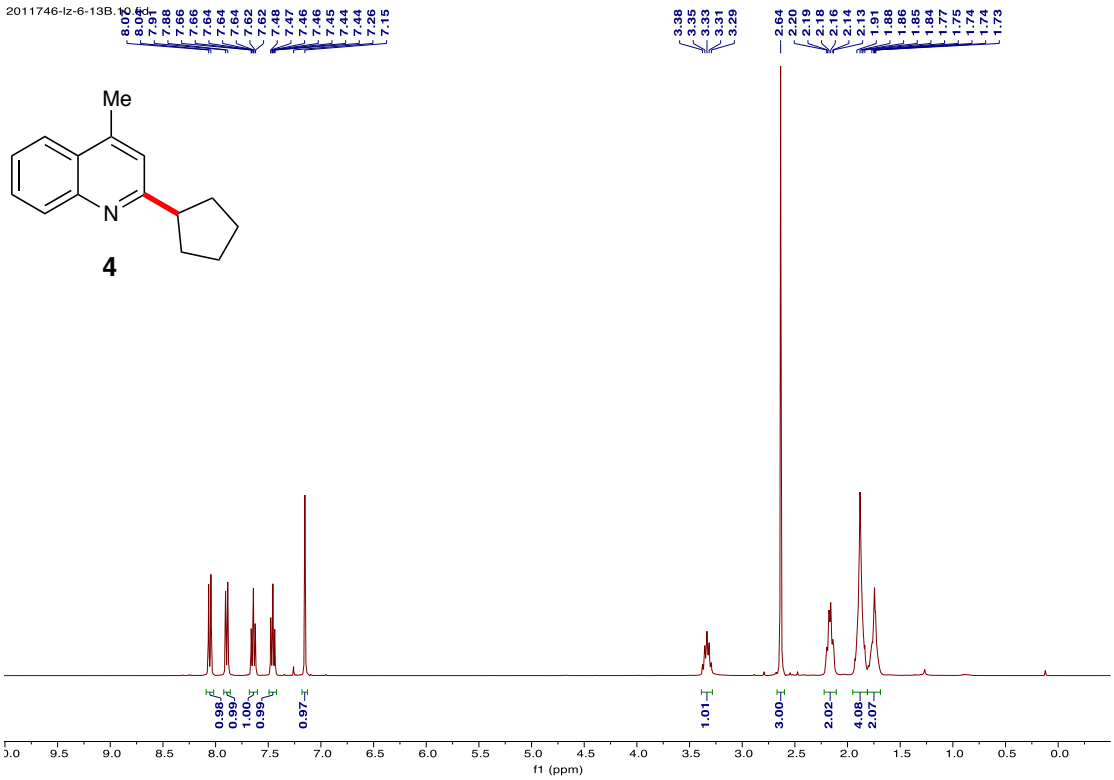
(2R,3S)-3-(6-cyclohexyl-5-fluoropyrimidin-4-yl)-2-(2,4-difluorophenyl)-1-(1*H*-1,2,4-triazol-1-yl)butan-2-ol (51): According to the general procedure, (2R,3S)-2-(2,4-difluorophenyl)-3-(5-fluoropyrimidin-4-yl)-1-(1*H*-1,2,4-triazol-1-yl)butan-2-ol (174.7 mg, 0.50 mmol, 1.0 equiv.), 5.0 mL of MeCN, H_2O_2 (0.1 mL, 1.0 mmol, 2.0 equiv.), HCl

(86.0 μL, 1.0 mmol, 2.0 equiv.), cyclohexane (1.10 ml, 10.0 mmol, 20.0 equiv.) were used. The reaction mixture was irradiated under 390 nm blue LEDs, after 18 hours, subjected to the workup procedure outlined in the general procedure and purified by flash chromatography (50% ethyl acetate/hexanes) to provide the title compound as a white solid (146.3 mg, 57% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.80 (d, *J* = 1.8 Hz, 1H), 7.99 (s, 1H), 7.65 – 7.54 (m, 1H), 7.52 (s, 1H), 6.86 – 6.78 (m, 2H), 6.76 (br s, 1H), 4.70 (d, *J* = 14.2 Hz, 1H), 4.28 (d, *J* = 14.1 Hz, 1H), 4.10 (q, *J* = 6.9 Hz, 1H), 3.15 – 3.02 (m, 1H), 1.93 – 1.77 (m, 5H), 1.72 – 1.60 (m, 2H), 1.49 – 1.29 (m, 3H), 1.07 (d, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.00, 163.88, 163.03, 162.89, 161.51, 161.39, 159.77, 159.66, 157.80, 157.65, 157.32, 157.20, 154.62, 152.75, 152.65, 152.00, 150.68, 143.92, 130.68, 130.63, 130.59, 130.53, 123.64, 123.61, 123.53, 123.49, 111.62, 111.59, 111.42, 111.39, 104.29, 104.03, 104.01, 103.76, 77.32, 77.00, 76.68, 57.54, 57.50, 39.07, 36.31, 36.25, 30.69, 30.47, 26.04, 26.01, 25.64, 16.20; HRMS (ESI) m/z calculated for $C_{22}H_{25}ON_5F_3$ [$(M+H)^+$] 432.2006, found 432.1997. IR (film) 3312, 2934, 2855, 1618, 1593, 1501, 1451, 1410, 1272, 1136, 966, 850, 738 cm^{-1}.

V. NMR Spectra



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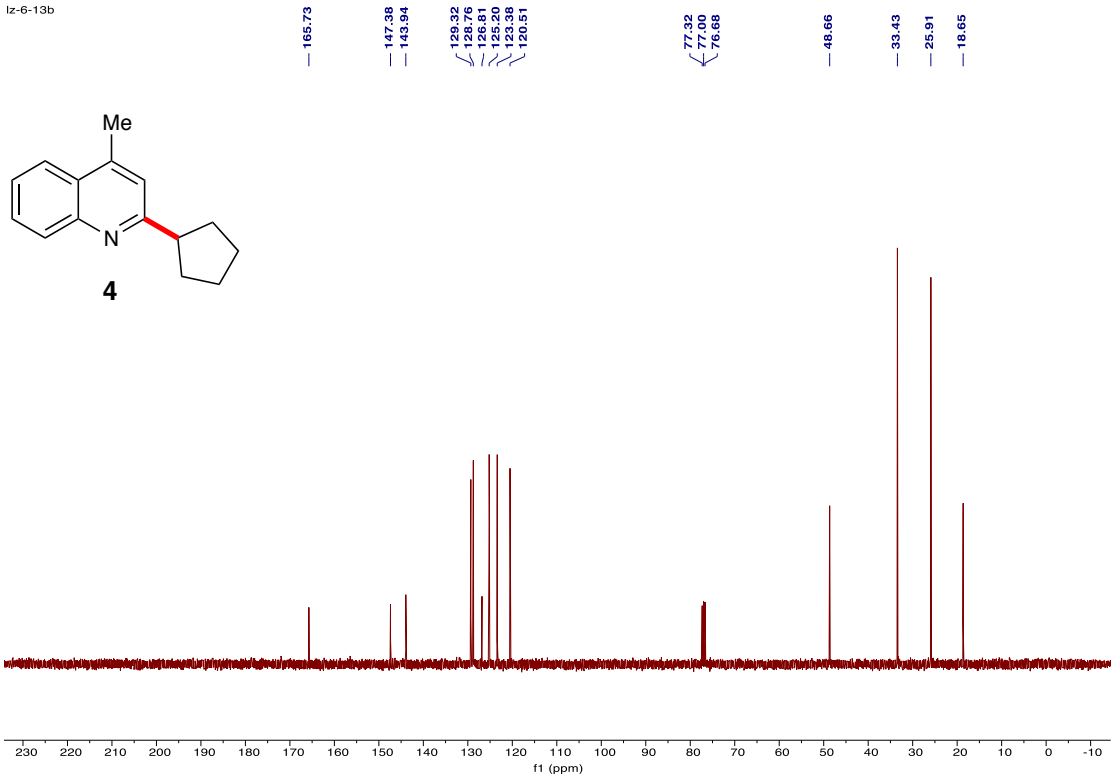
8.07
8.05
7.97
7.88
7.66
7.66
7.64
7.64
7.64
7.62
7.62
7.48
7.47
7.46
7.46
7.45
7.44
7.44
7.26
7.15
3.38
3.35
3.33
3.31
3.29
2.64
2.20
2.19
2.18
2.16
2.14
2.13
1.91
1.88
1.86
1.85
1.84
1.77
1.75
1.74
1.74
1.73
Me
N
4
0.98
0.99
1.00
0.99
0.97
1.01
3.00
2.02
4.08
2.07
f1 (ppm)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143.94
129.32
128.76
126.81
125.20
123.38
120.51
77.32
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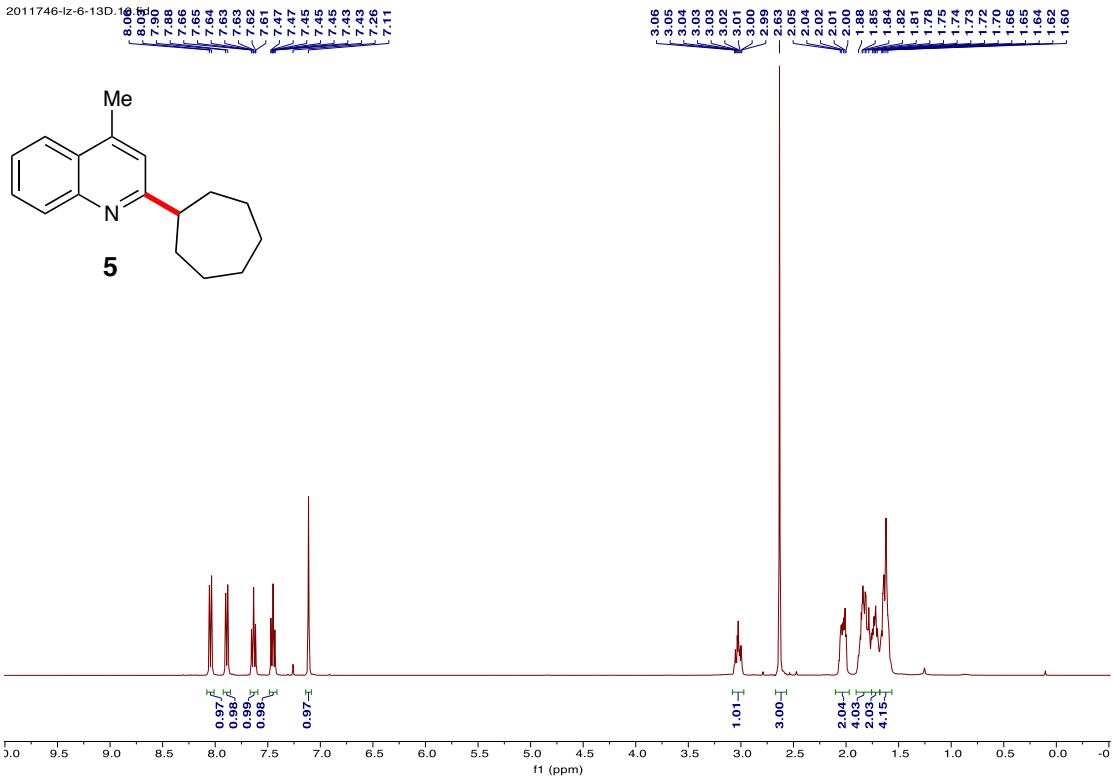
25.91
18.65
Me
N
4
f1 (ppm)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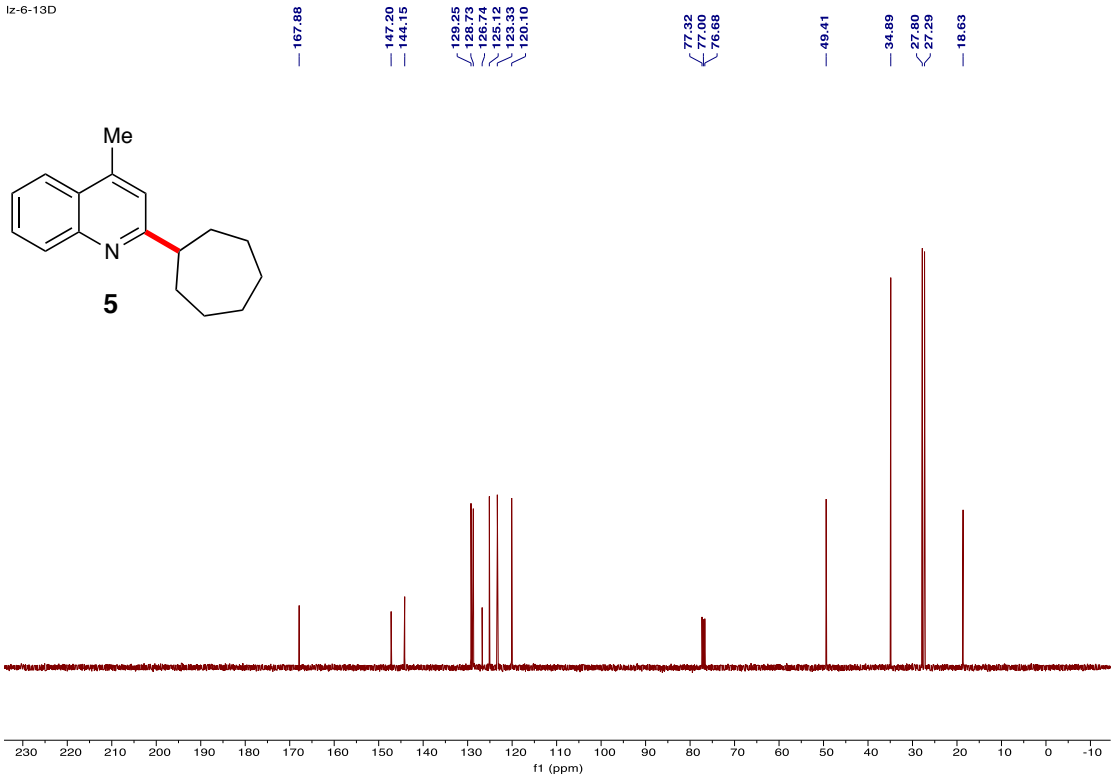
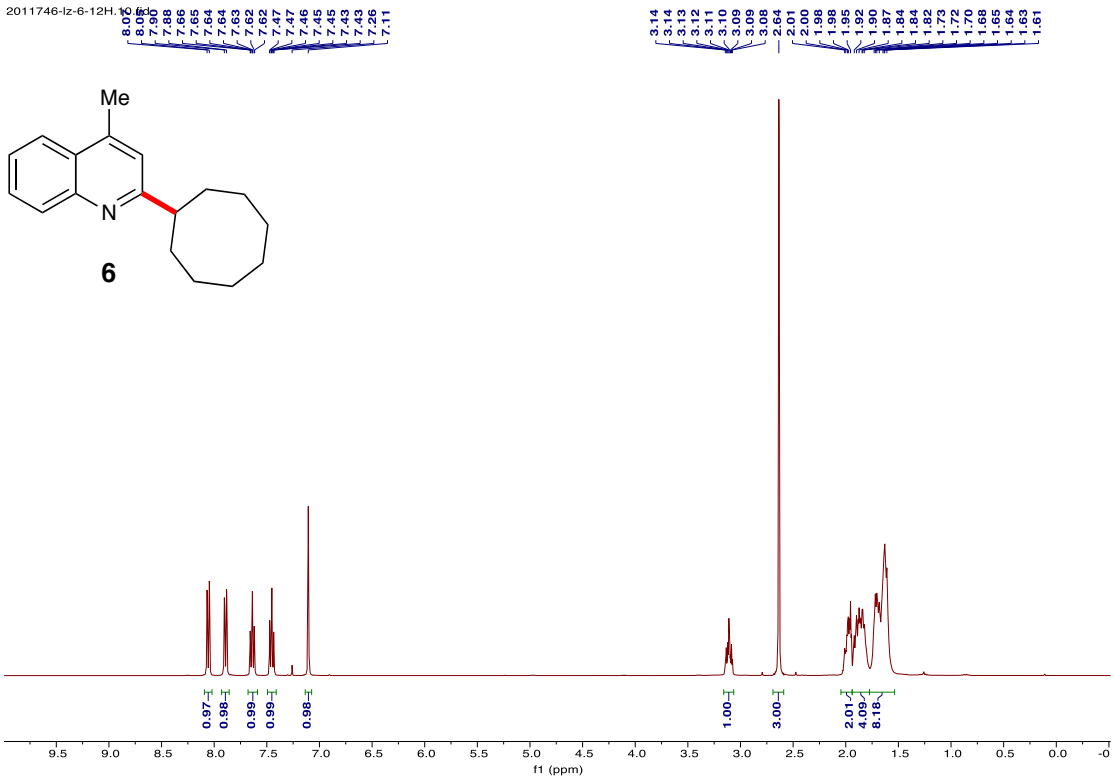
Me
N
5
f1 (ppm)


lz-6-13D
Me
N
5
f1 (ppm)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8.05
7.90
7.88
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7.64
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7.46
7.45
7.45
7.43
7.43
7.26
7.11
3.14
3.14
3.13
3.12
3.11
3.10
3.09
3.09
3.08
2.64
2.01
2.00
1.98
1.98
1.95
1.92
1.90
1.87
1.84
1.84
1.82
1.73
1.72
1.70
1.68
1.65
1.64
1.63
1.61
Me
N
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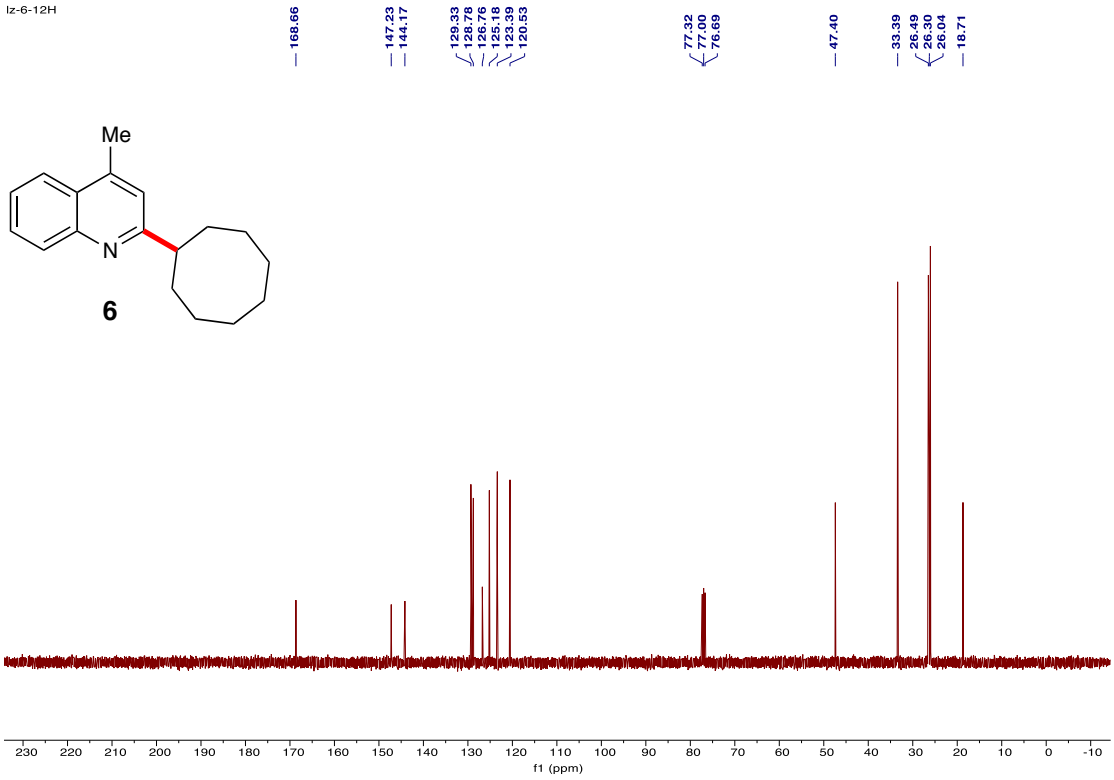
0.98
0.99
0.99
0.98
1.00
3.00
2.01
4.09
8.18
f1 (ppm)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128.78
126.76
125.18
123.39
120.53
77.32
77.00
76.69
47.40
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26.49
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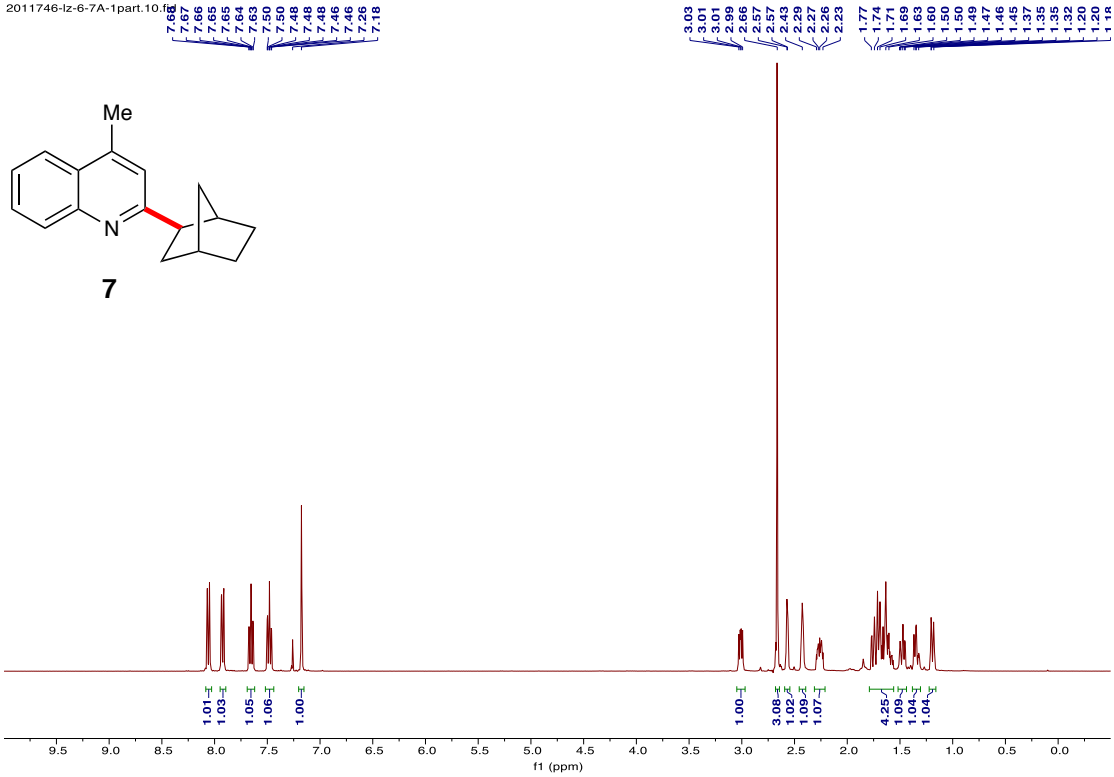
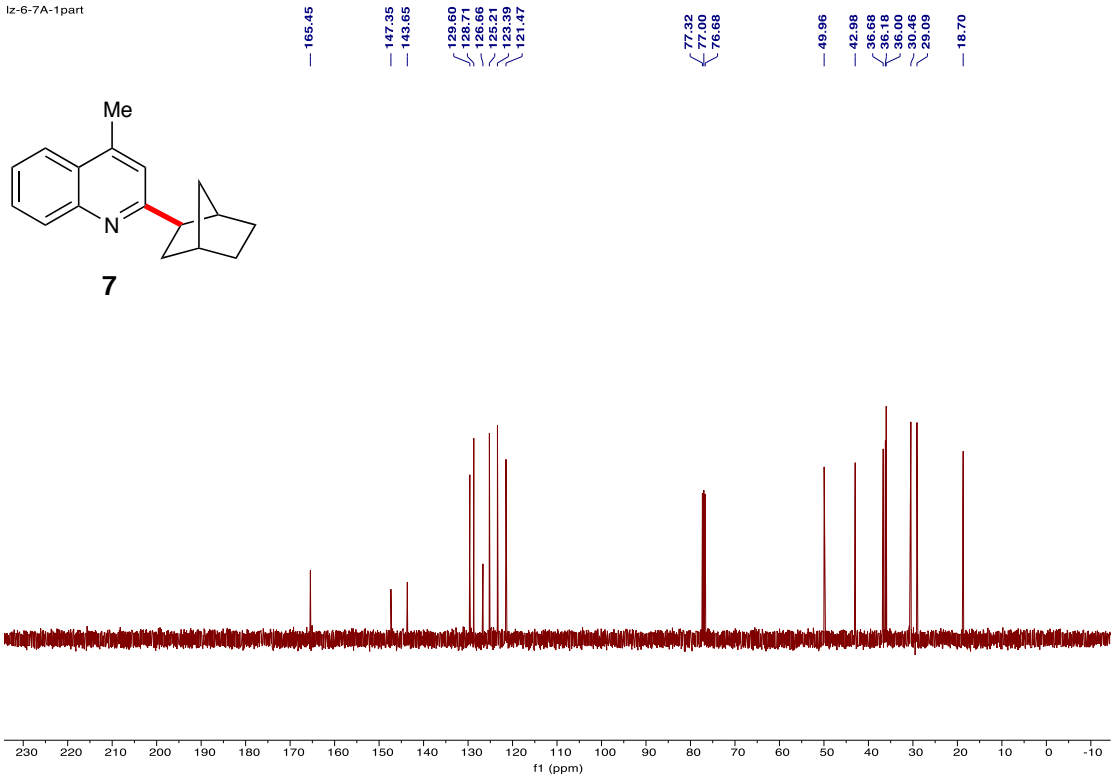
26.04
18.71
Me
N
6
f1 (ppm)

2011746-lz-6-7A-1part.10.fid

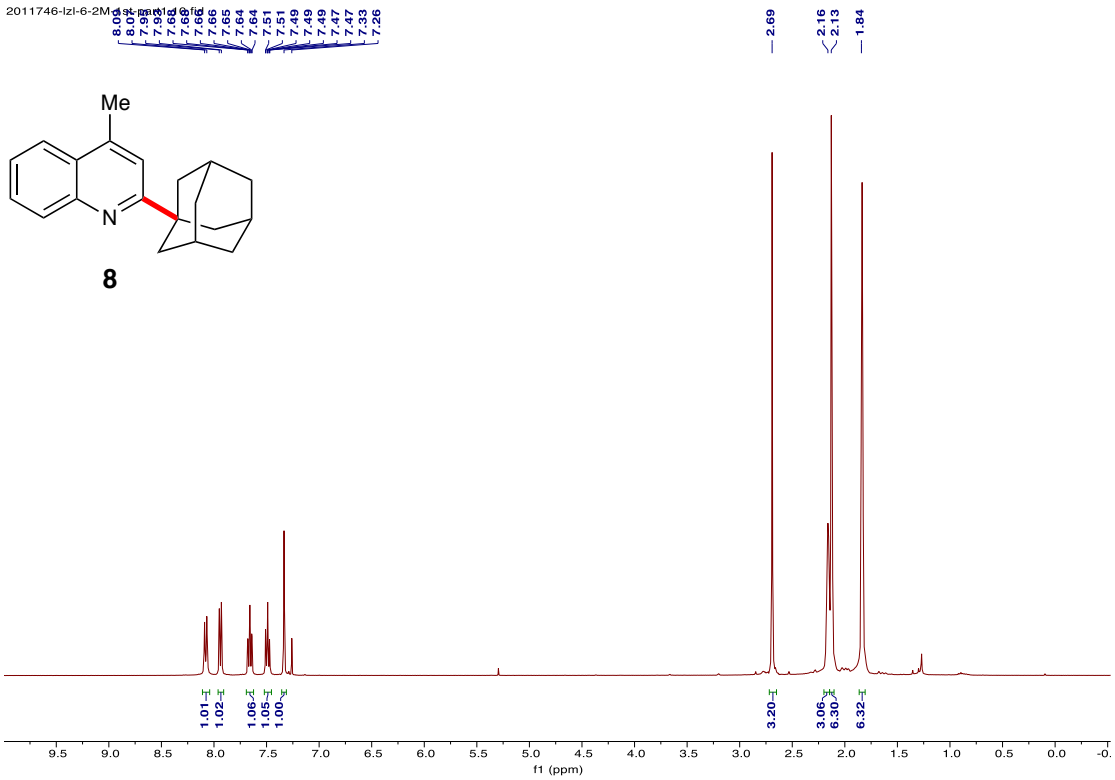


7

lz-6-7A-1part



7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7.66
7.66
7.65
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7.51
7.49
7.49
7.49
7.47
7.47
7.33
7.26
2.69
2.16
2.13
1.84
Me
N
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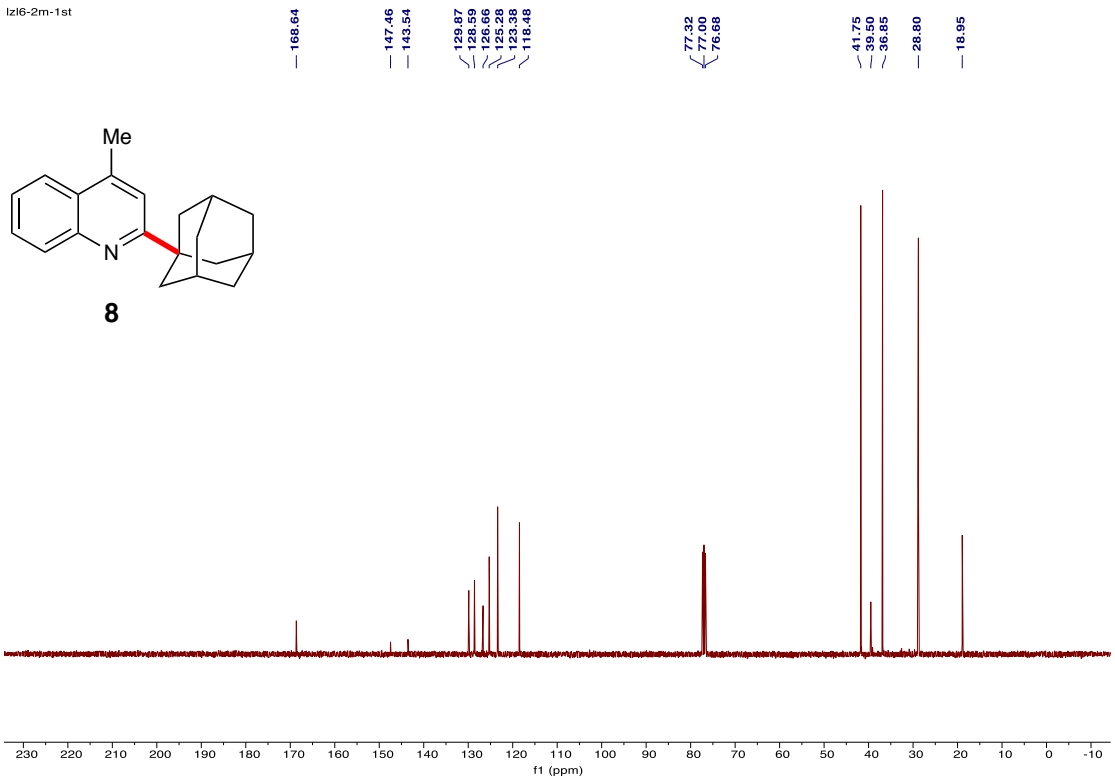
1.02
1.06
1.05
1.00
3.20
3.06
6.30
6.32
f1 (ppm)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126.66
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39.50
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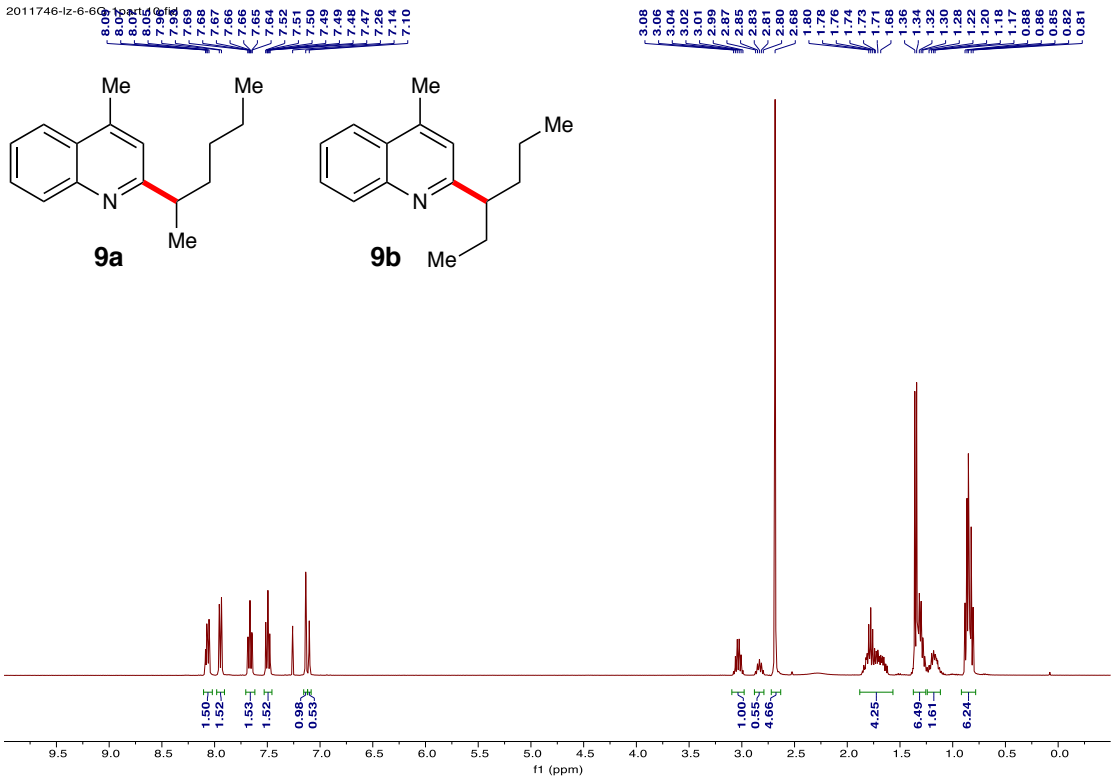
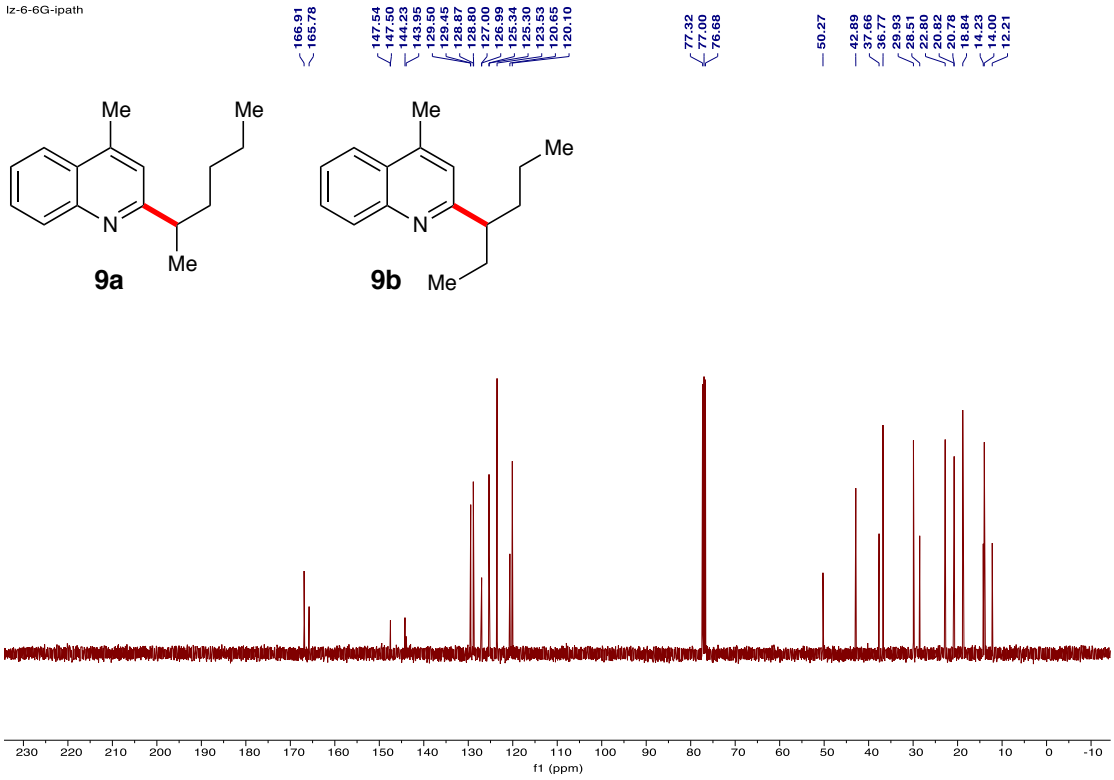
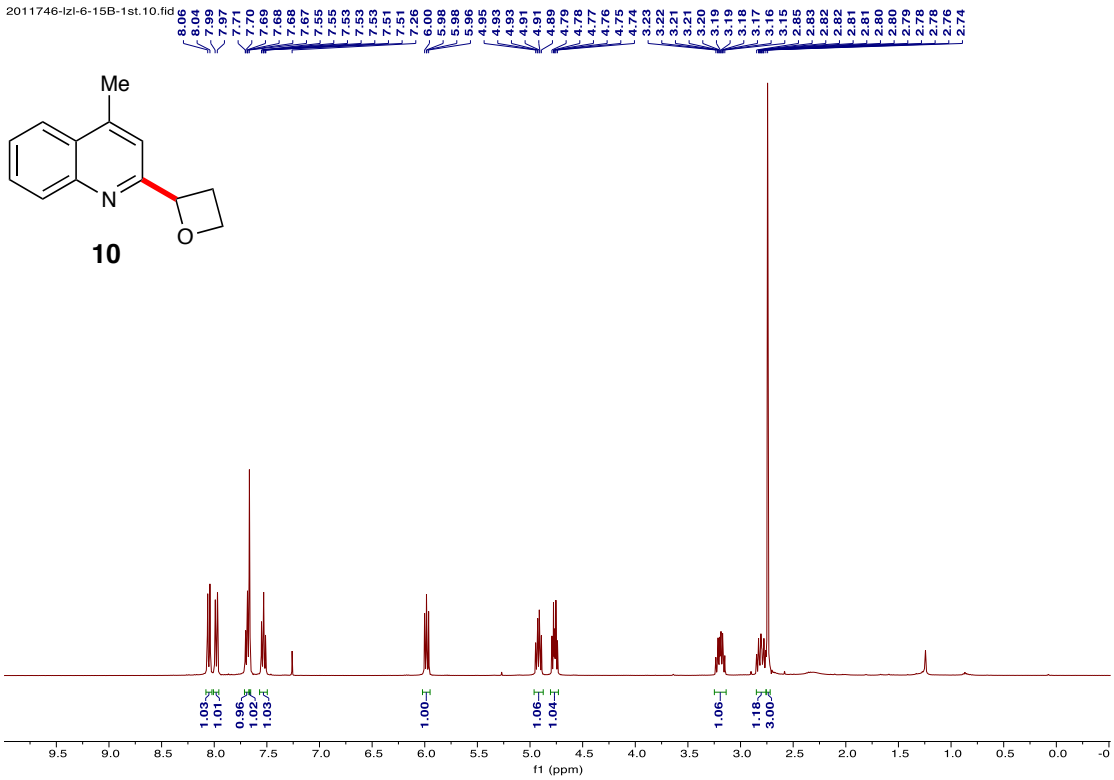
28.80
18.95
Me
N
8
f1 (ppm)


Me
Me
N
Me
9a
Me
Me
N
Me
9b
Me
f1 (ppm)


lz-6-6G-ipath
Me
Me
N
Me
9a
Me
Me
N
Me
9b
Me
f1 (ppm)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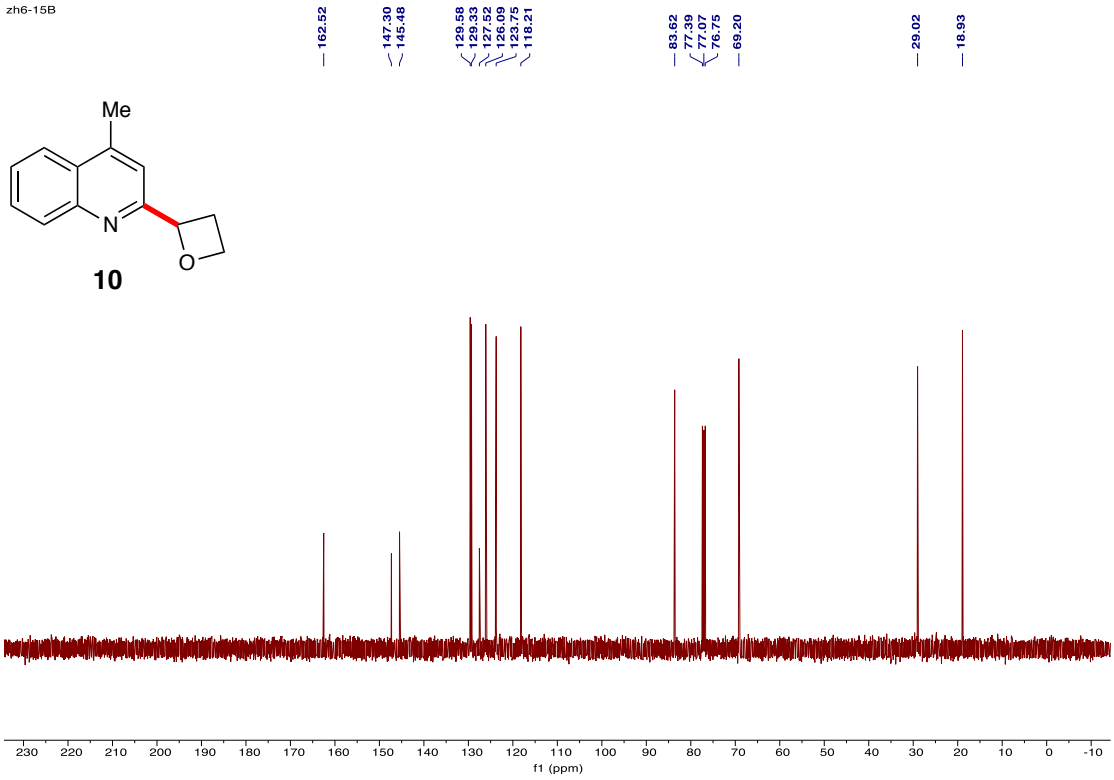
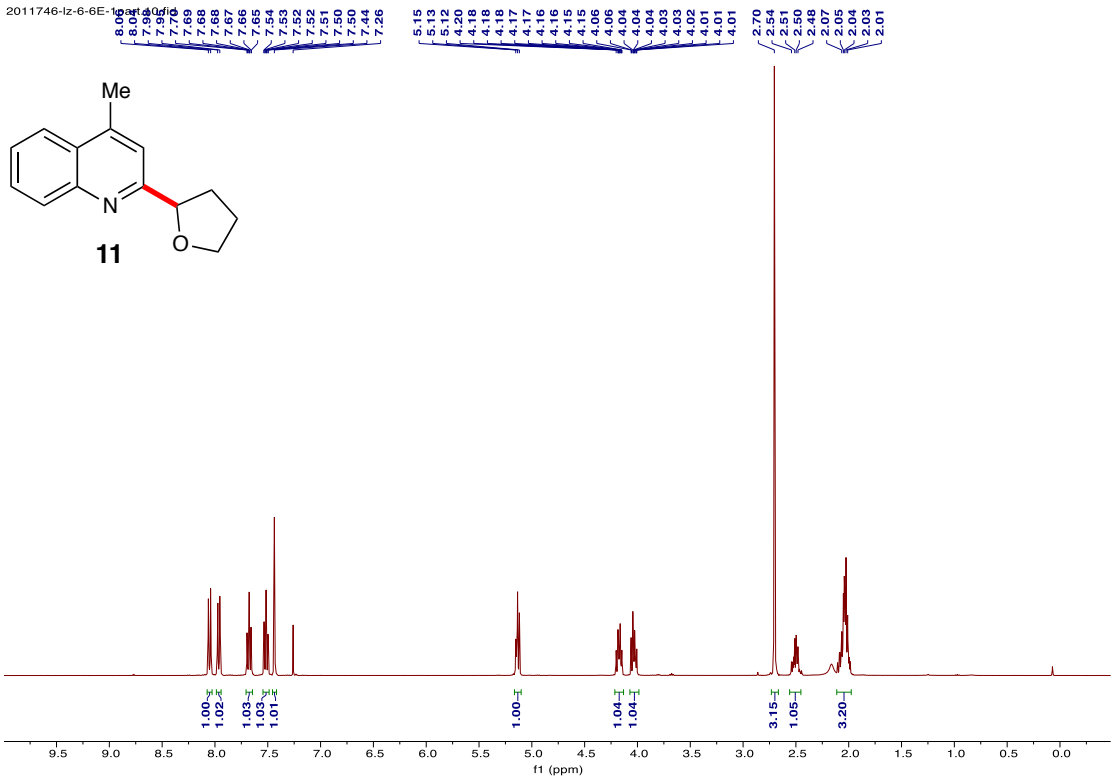
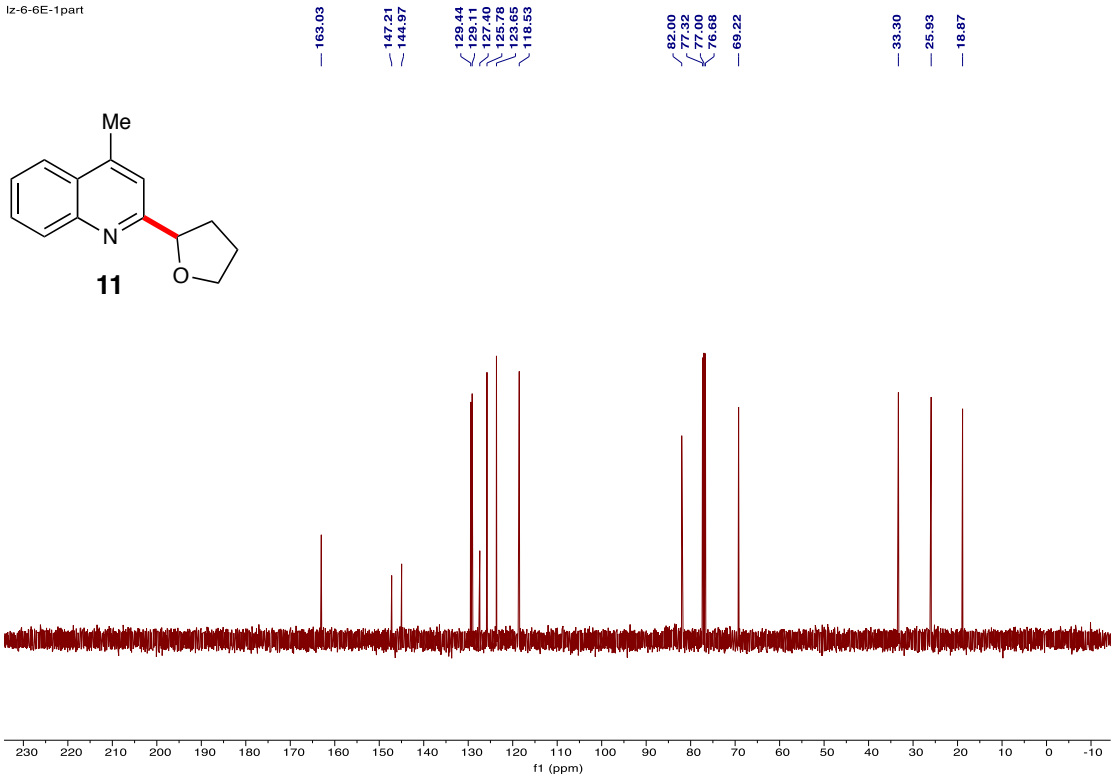
Me
N
O
10
f1 (ppm)


zh6-15B
Me
N
O
10
f1 (ppm)


2011746-lz-6-6E-1-part-10.jd
Me
N
O
11
f1 (ppm)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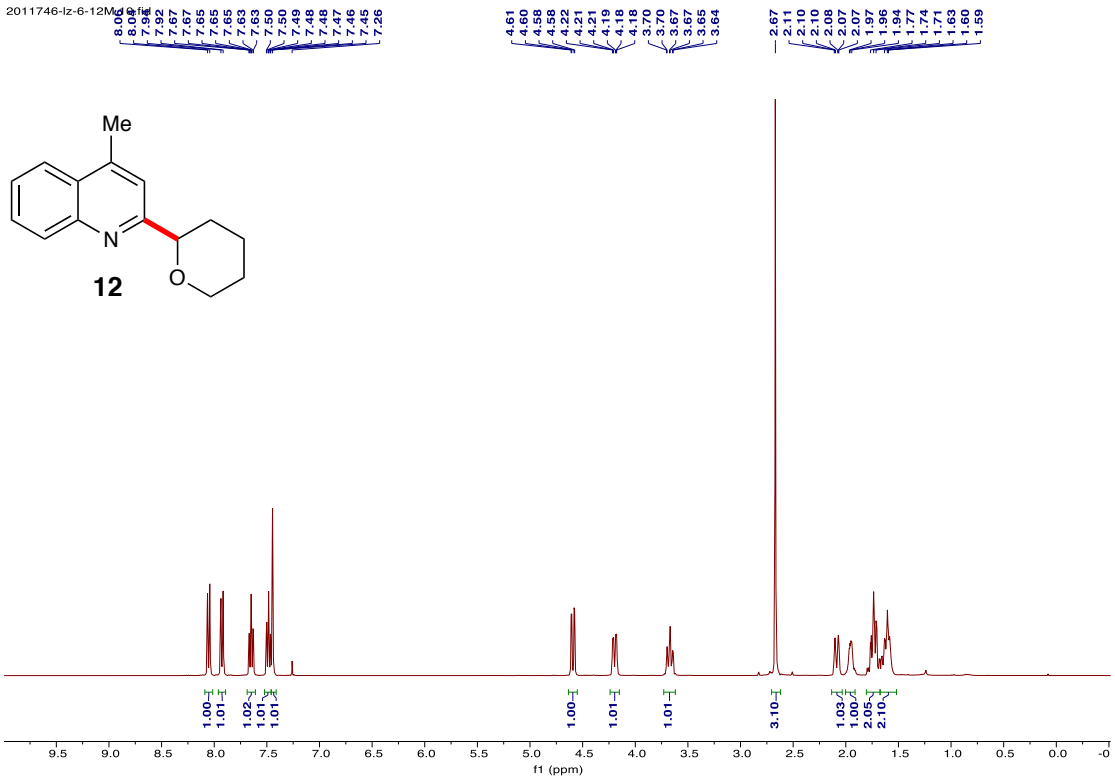
Me
N
O
11
f1 (ppm)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N
O
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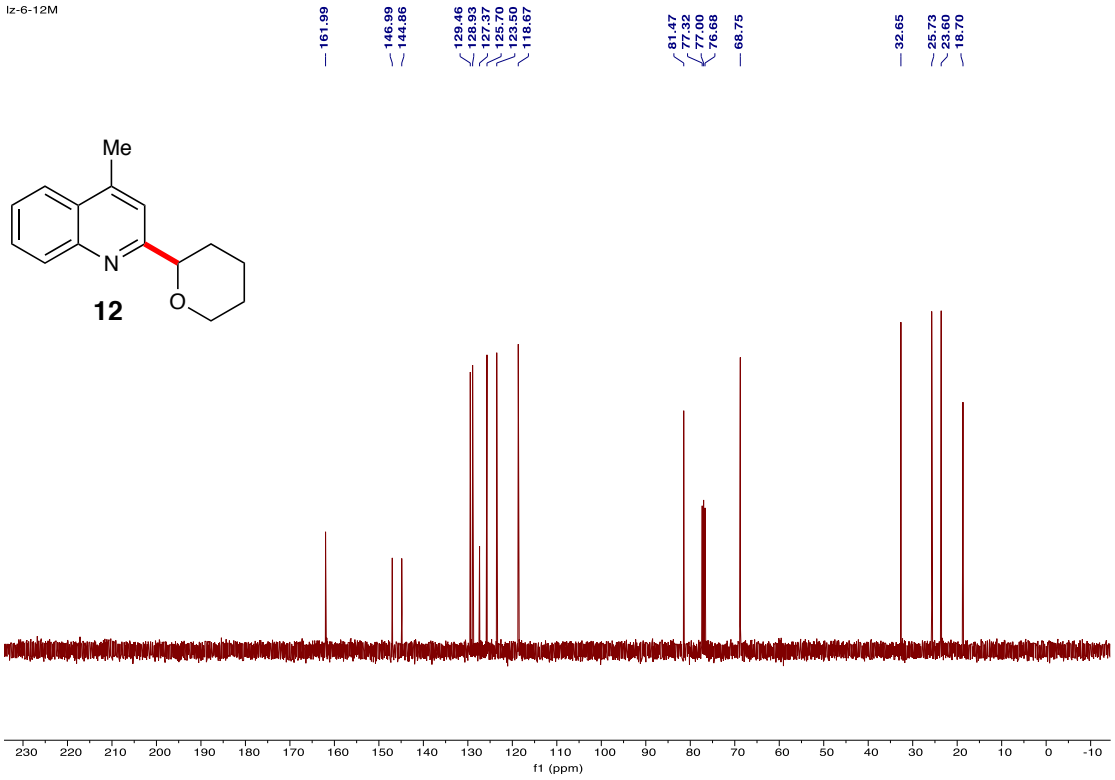
f1 (ppm)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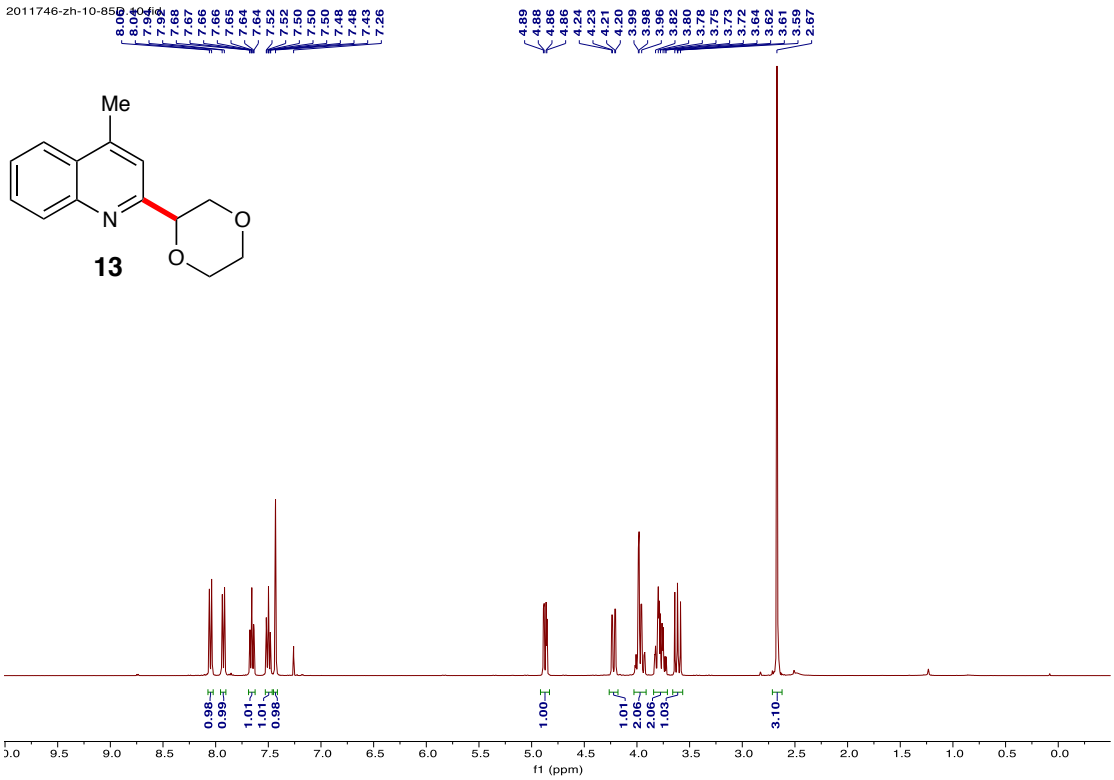
Me
N
O
12
f1 (ppm)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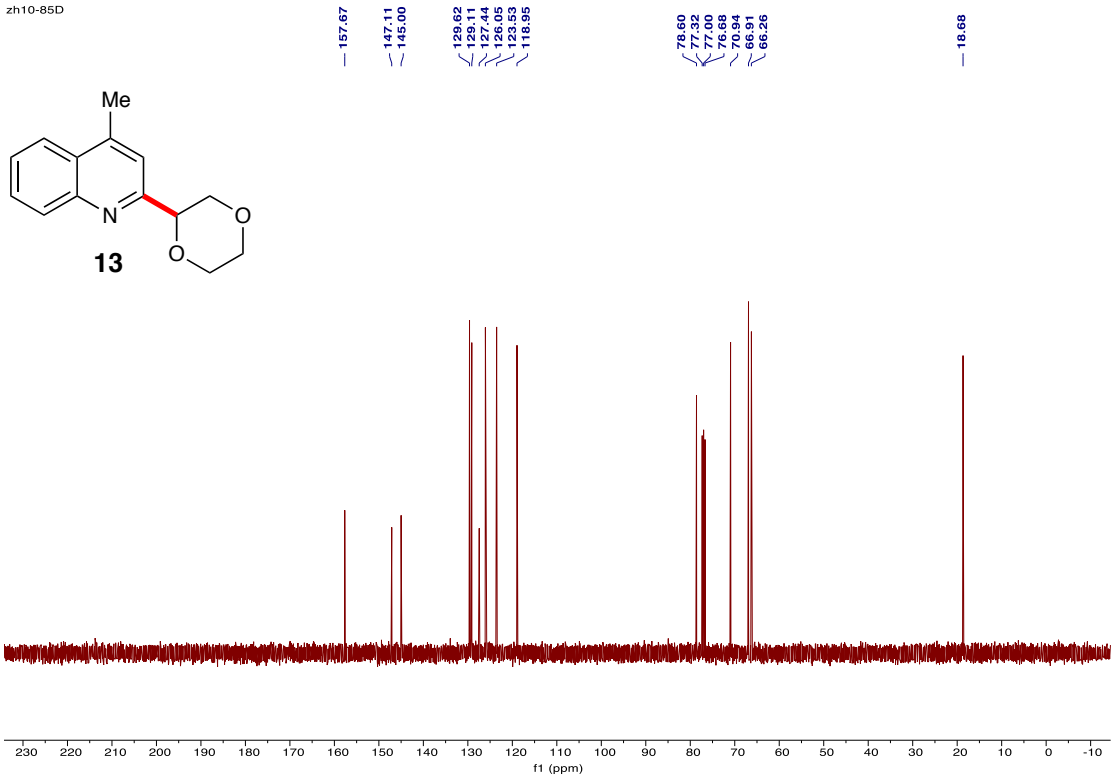
7.50
7.50
7.50
7.48
7.48
7.43
7.26
4.89
4.88
4.86
4.86
4.24
4.23
4.21
4.20
3.99
3.98
3.96
3.92
3.80
3.78
3.75
3.73
3.72
3.64
3.62
3.61
3.59
2.67
Me
N
O
O
13
0.98
0.99
1.01
1.01
0.98
1.00
1.01
2.06
2.06
1.03
3.10
f1 (ppm)


zh10-85D
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129.11
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123.53
118.95
78.60
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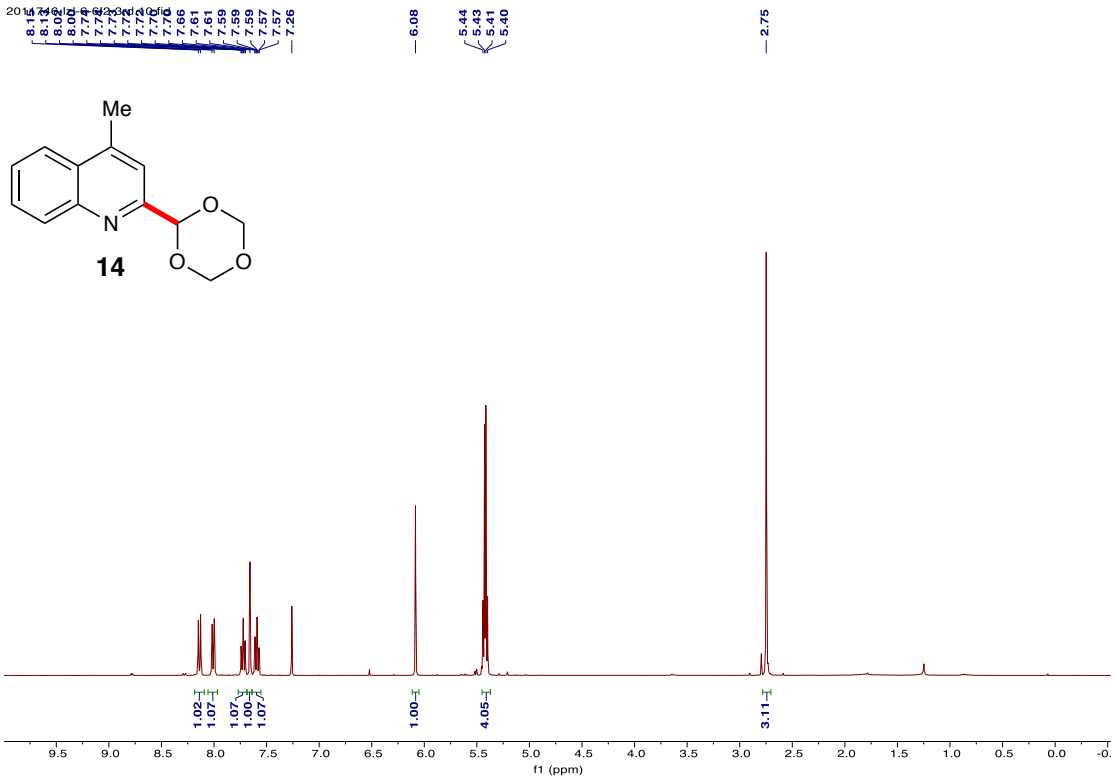
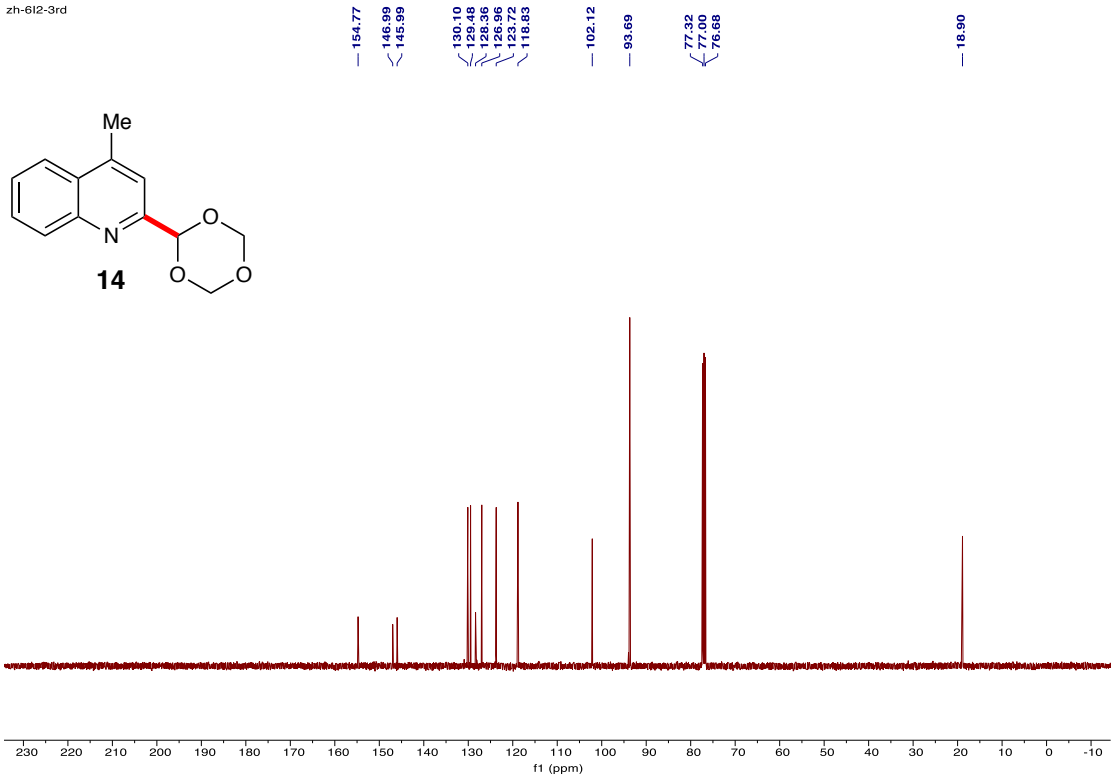
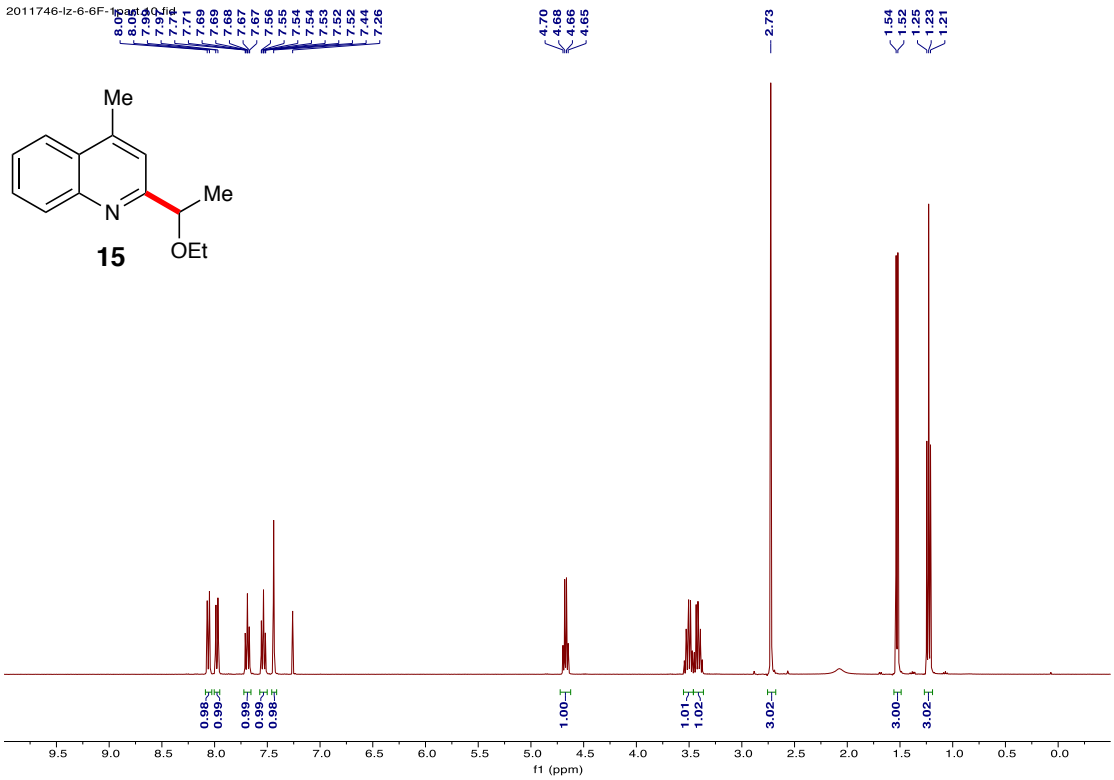
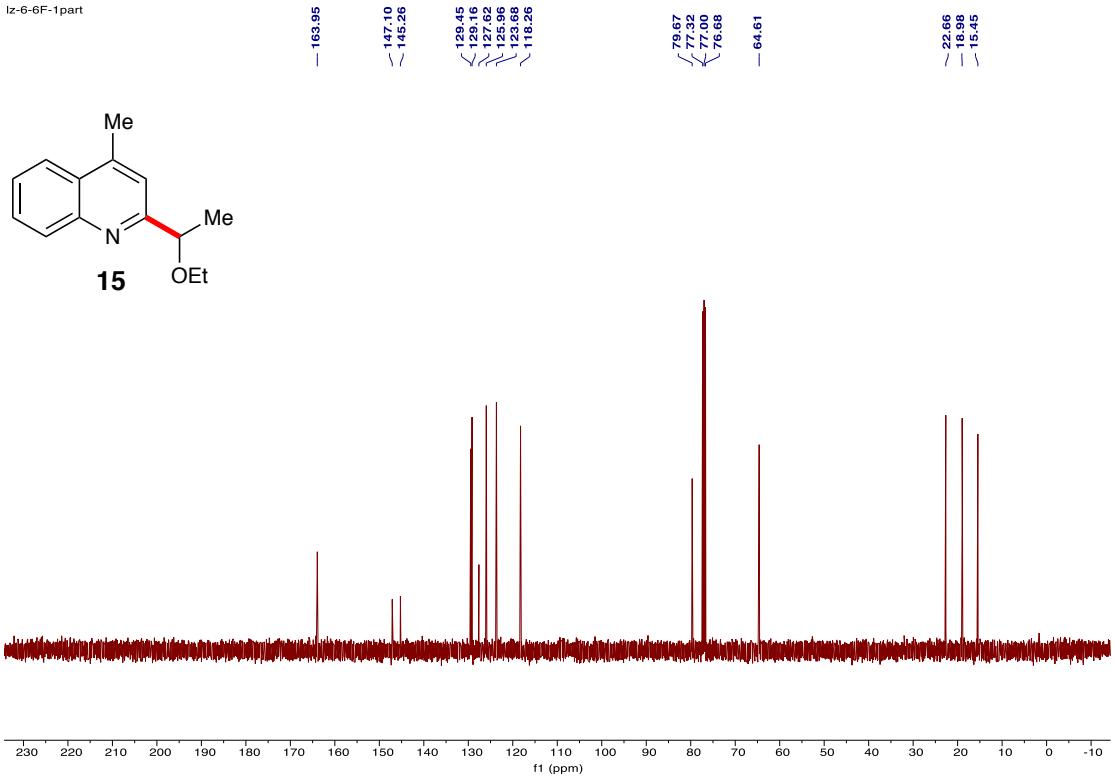
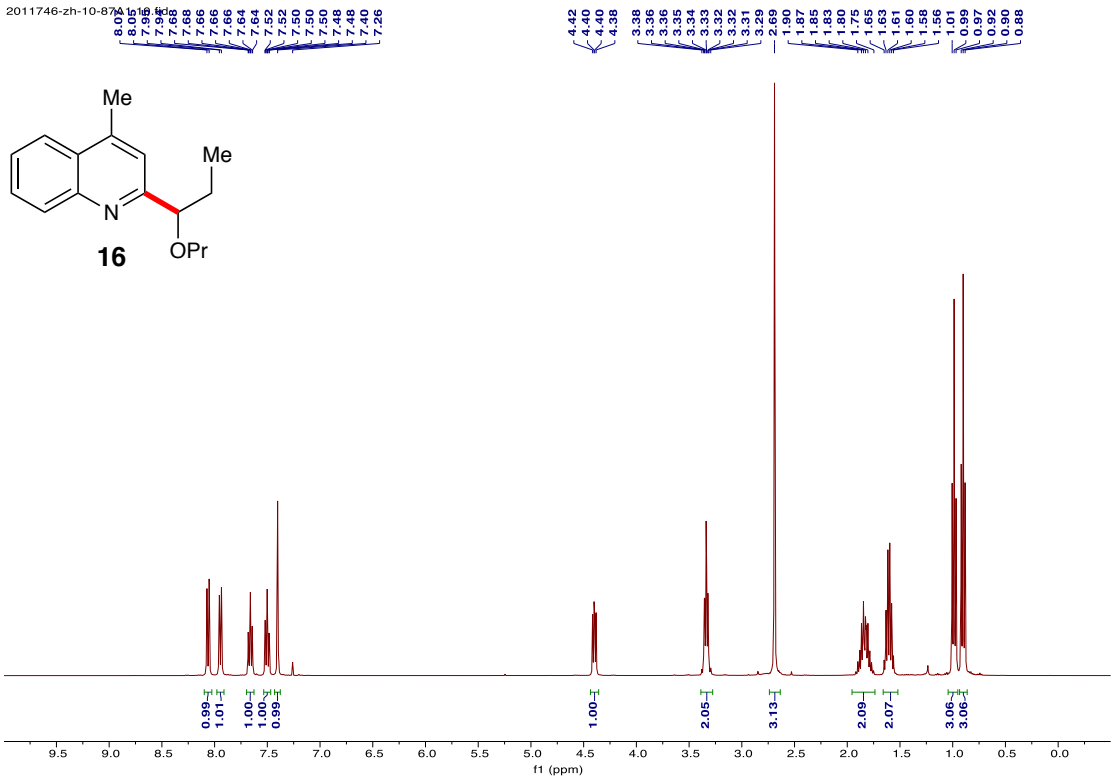
77.00
76.68
70.94
66.91
66.26
18.68
Me
N
O
O
13
f1 (ppm)

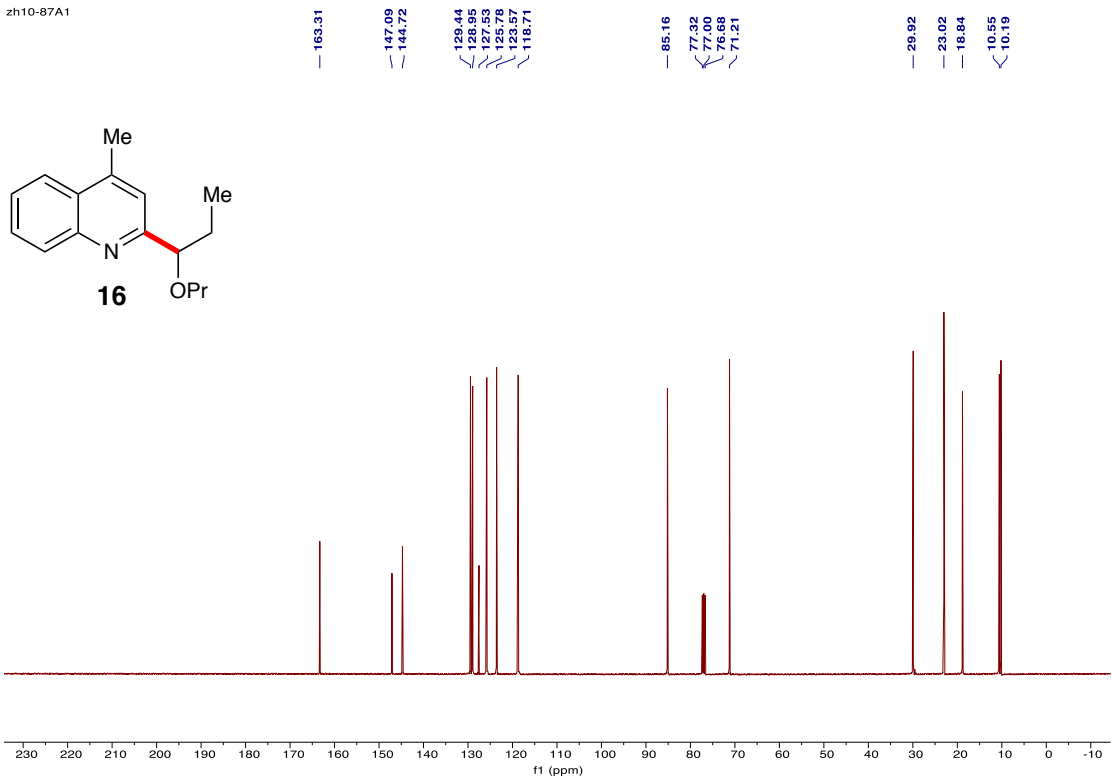

Me
N
O
O
O
14
f1 (ppm)

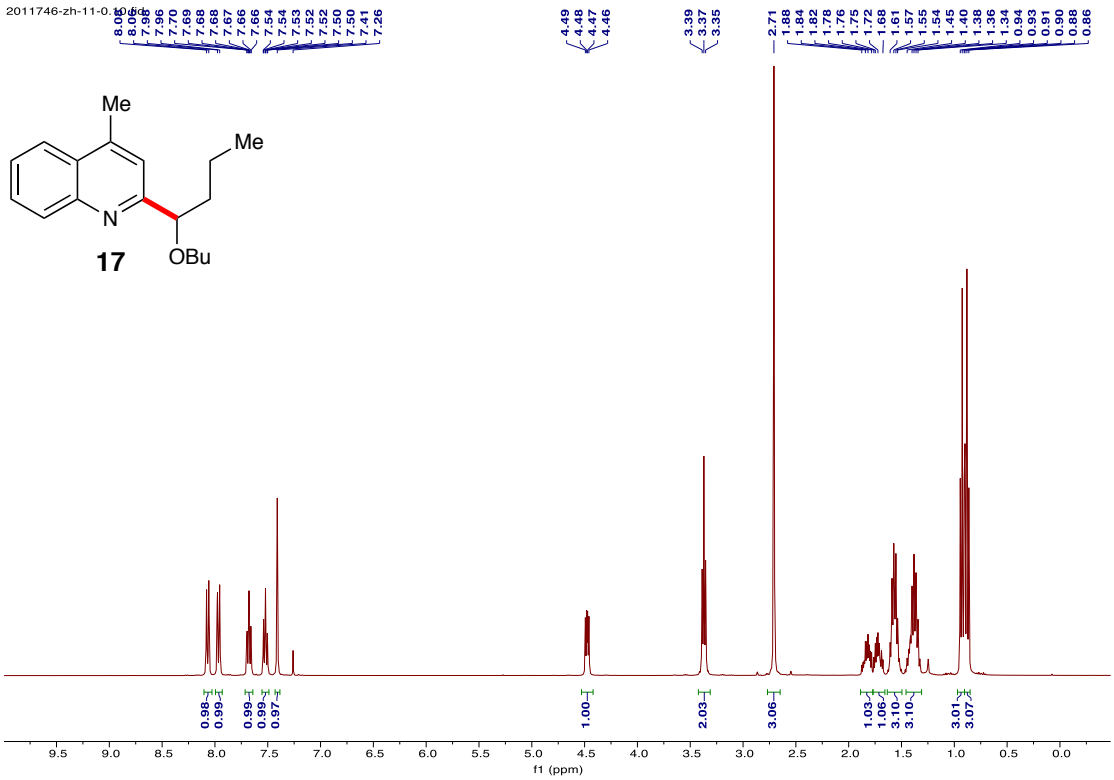

zh-6l2-3rd
Me
N
O
O
O
14
f1 (ppm)

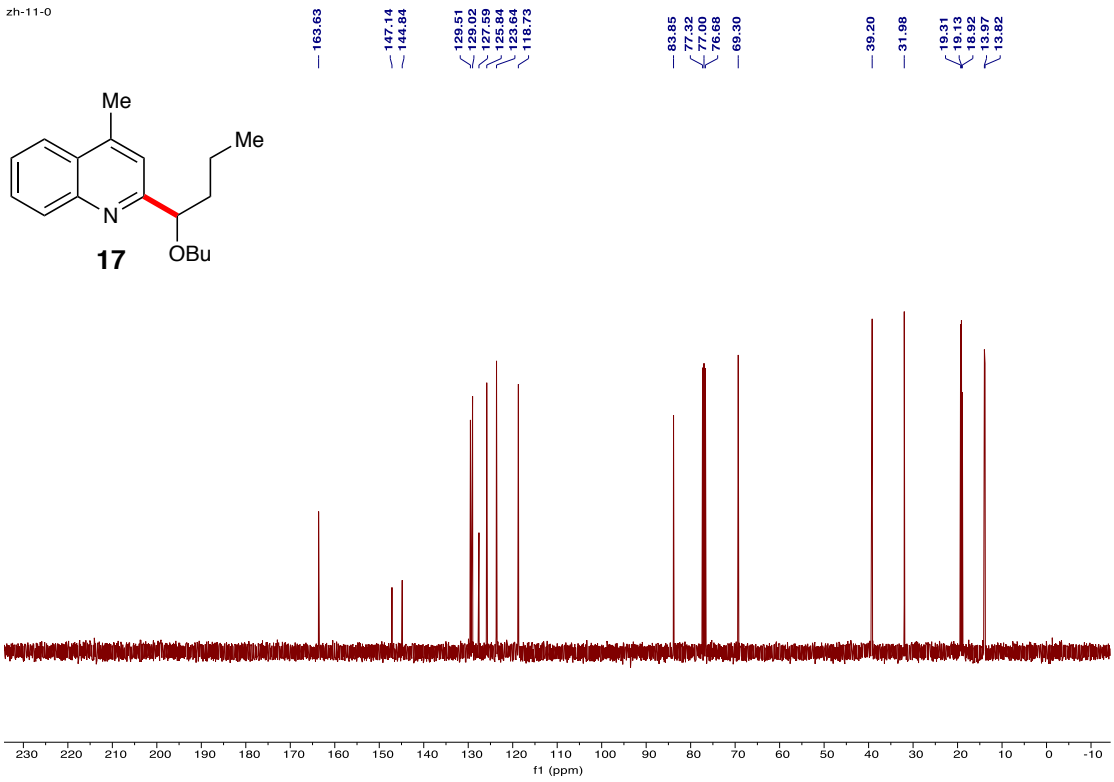

2011746-lz-6-6F-1part 10.fid
8.07
8.05
7.99
7.97
7.74
7.71
7.69
7.69
7.68
7.67
7.67
7.56
7.55
7.54
7.54
7.53
7.52
7.52
7.44
7.26
4.70
4.68
4.66
4.65
2.73
1.54
1.52
1.25
1.23
1.21
Me
Me
N
OEt
15
0.98
0.99
0.99
0.99
0.98
1.00
1.01
1.02
3.02
3.00
3.02
f1 (ppm)

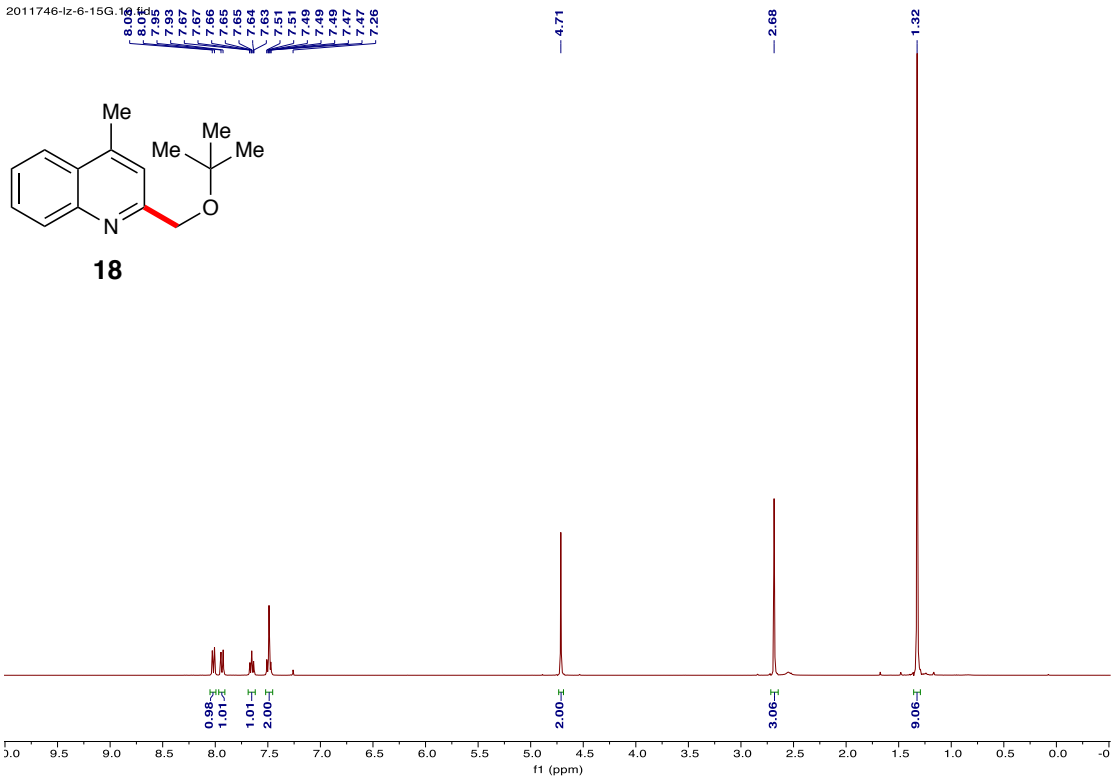

lz-6-6F-1part
163.95
147.10
145.26
129.45
129.16
127.62
125.96
123.68
118.26
79.67
77.32
77.00
76.68
64.61
22.66
18.98
15.45
Me
Me
N
OEt
15
f1 (ppm)

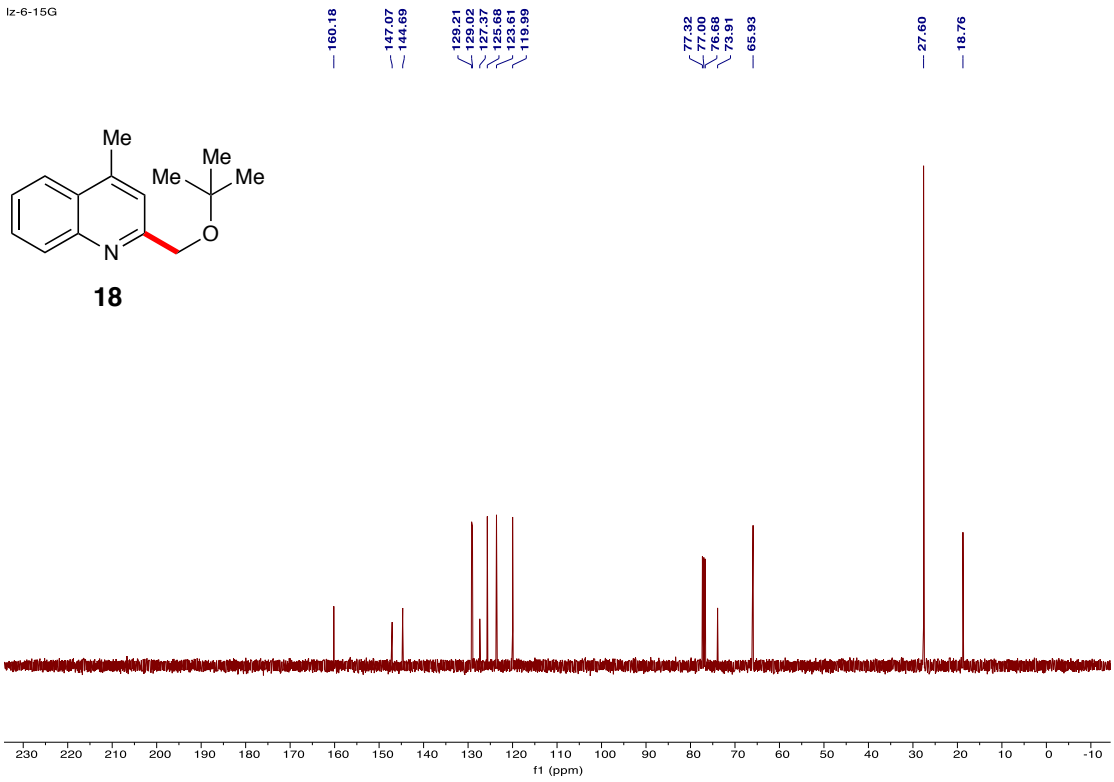

2011746-zh-10-87A1-1H.fid
8.07
8.05
7.95
7.94
7.68
7.68
7.66
7.66
7.66
7.64
7.64
7.52
7.52
7.50
7.50
7.50
7.48
7.48
7.40
7.26
4.42
4.40
4.40
4.38
3.38
3.36
3.36
3.35
3.34
3.33
3.32
3.32
3.31
3.29
2.69
1.90
1.87
1.85
1.83
1.80
1.75
1.65
1.63
1.61
1.60
1.58
1.56
1.01
0.99
0.97
0.92
0.90
0.88
Me
Me
N
OPr
16
0.99
1.01
1.00
1.00
0.99
1.00
2.05
3.13
2.09
2.07
3.06
3.06
9.5
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
0.0
f1 (ppm)

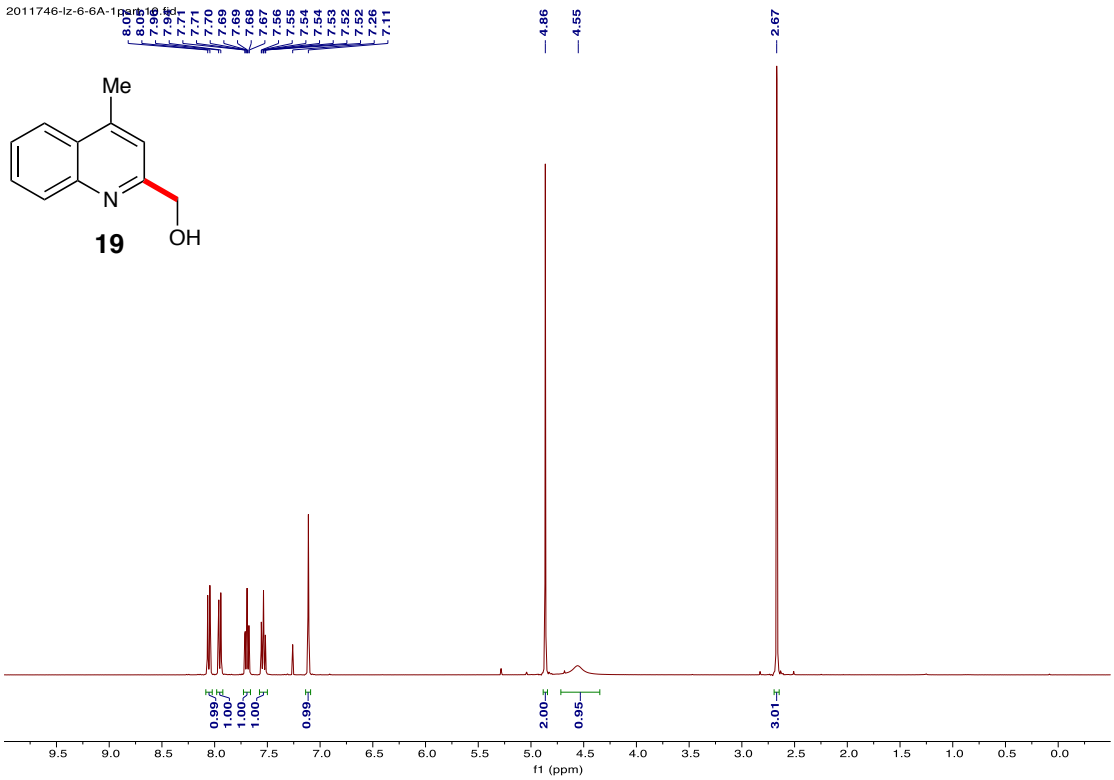

zh10-87A1
163.31
147.09
144.72
129.44
128.95
127.53
125.78
123.57
118.71
85.16
77.32
77.00
76.68
71.21
29.92
23.02
18.84
10.55
10.19
Me
Me
N
OPr
16
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

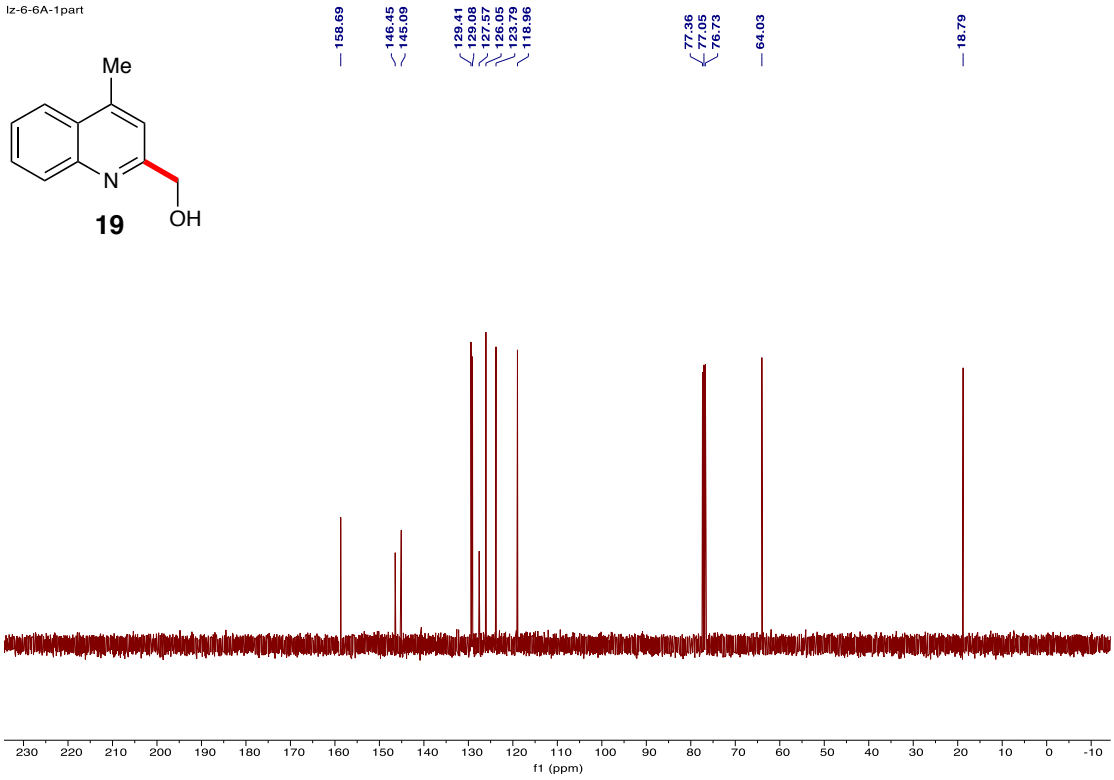

2011746-zh-11-0.10.fid
8.08
8.06
7.98
7.96
7.70
7.69
7.68
7.68
7.67
7.66
7.66
7.54
7.54
7.53
7.52
7.50
7.50
7.41
7.26
4.49
4.48
4.47
4.46
3.39
3.37
3.35
2.71
1.84
1.84
1.82
1.78
1.76
1.75
1.72
1.68
1.61
1.57
1.55
1.54
1.45
1.40
1.38
1.36
1.34
0.94
0.93
0.91
0.90
0.88
0.86
Me
Me
N
OBu
17
0.98
0.99
0.99
0.99
0.97
1.00
2.03
3.06
1.03
1.06
3.10
3.10
3.01
3.07
f1 (ppm)

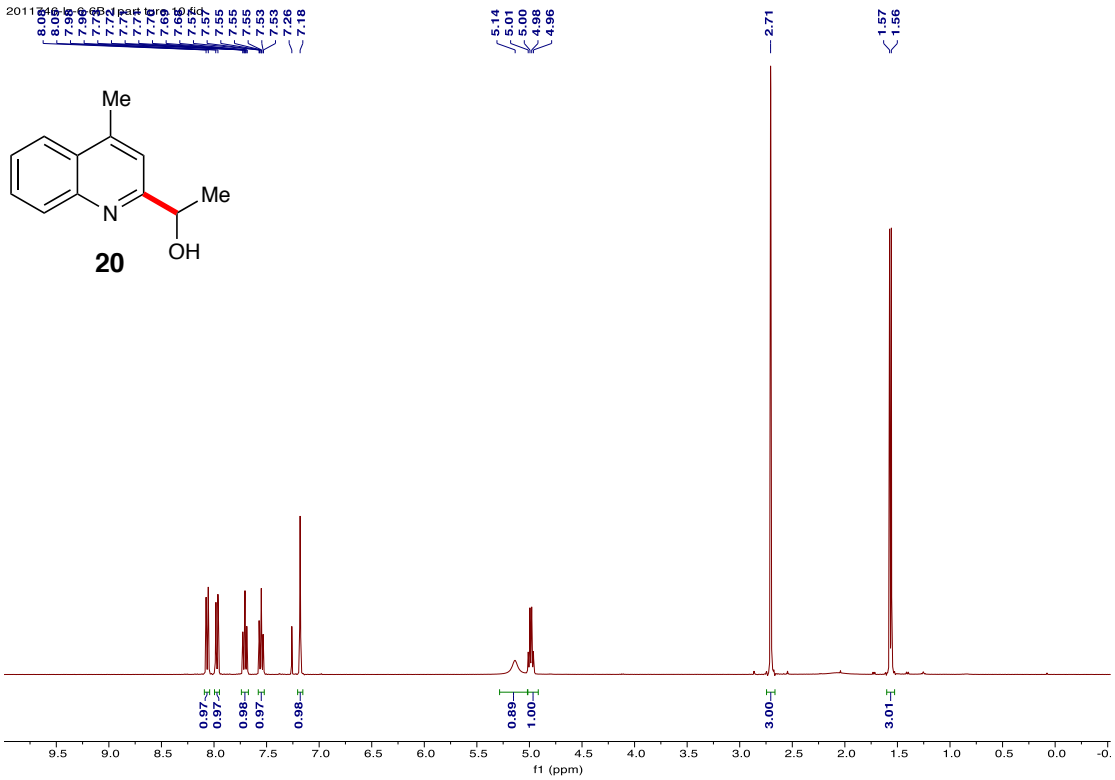

zh-11-0
163.63
147.14
144.84
129.51
129.02
127.59
125.84
123.64
118.73
83.85
77.32
77.00
76.68
69.30
39.20
31.98
19.31
19.13
18.92
13.97
13.82
Me
Me
N
OBu
17
f1 (ppm)

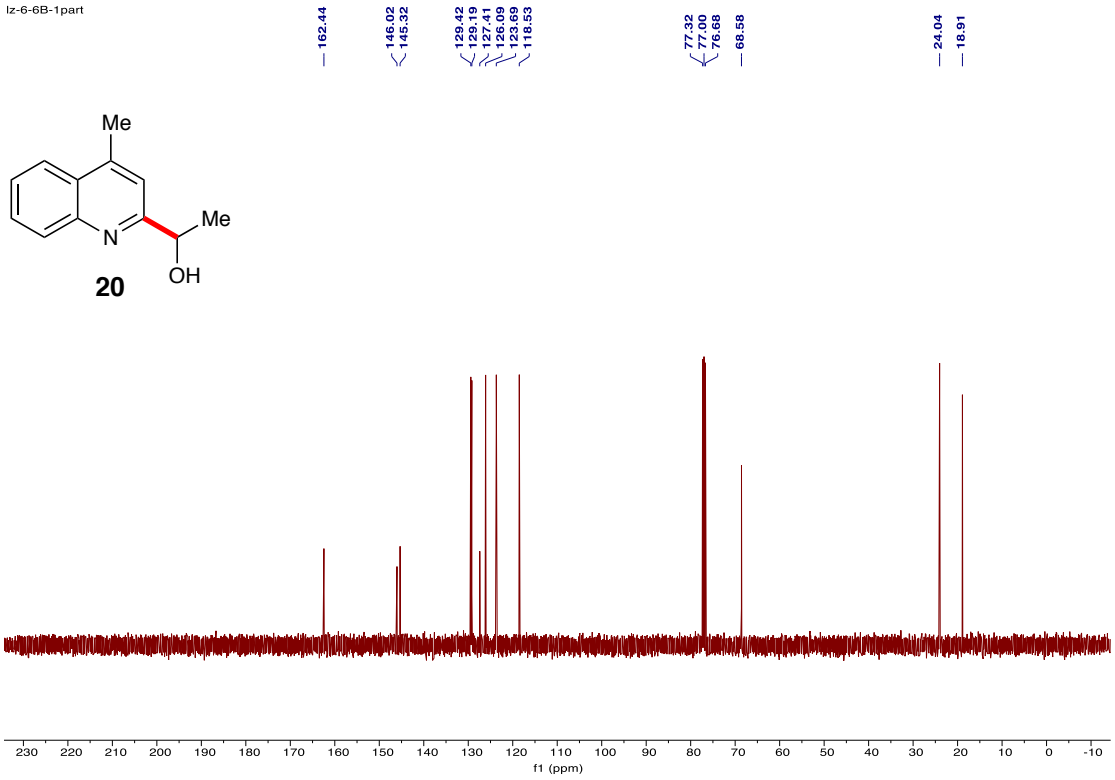

2011746-lz-6-15G.10.fid
8.04
8.03
7.95
7.93
7.67
7.66
7.65
7.64
7.63
7.51
7.49
7.49
7.49
7.47
7.47
7.26
4.71
2.68
1.32
Me
Me
Me
Me
N
O
18
0.98
1.01
1.01
2.00
2.00
3.06
9.06
f1 (ppm)

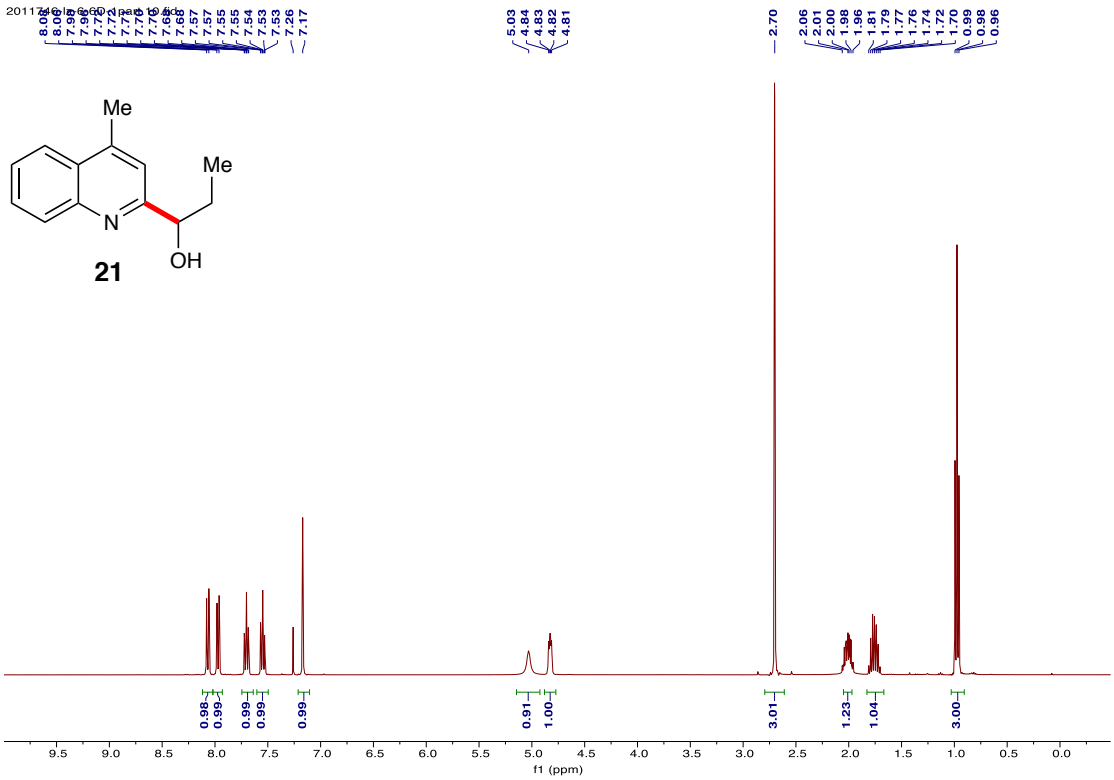

lz-6-15G
160.18
147.07
144.69
129.21
129.02
127.37
125.68
123.61
119.99
77.32
77.00
76.68
73.91
65.93
27.60
18.76
Me
Me
Me
Me
N
O
18
f1 (ppm)

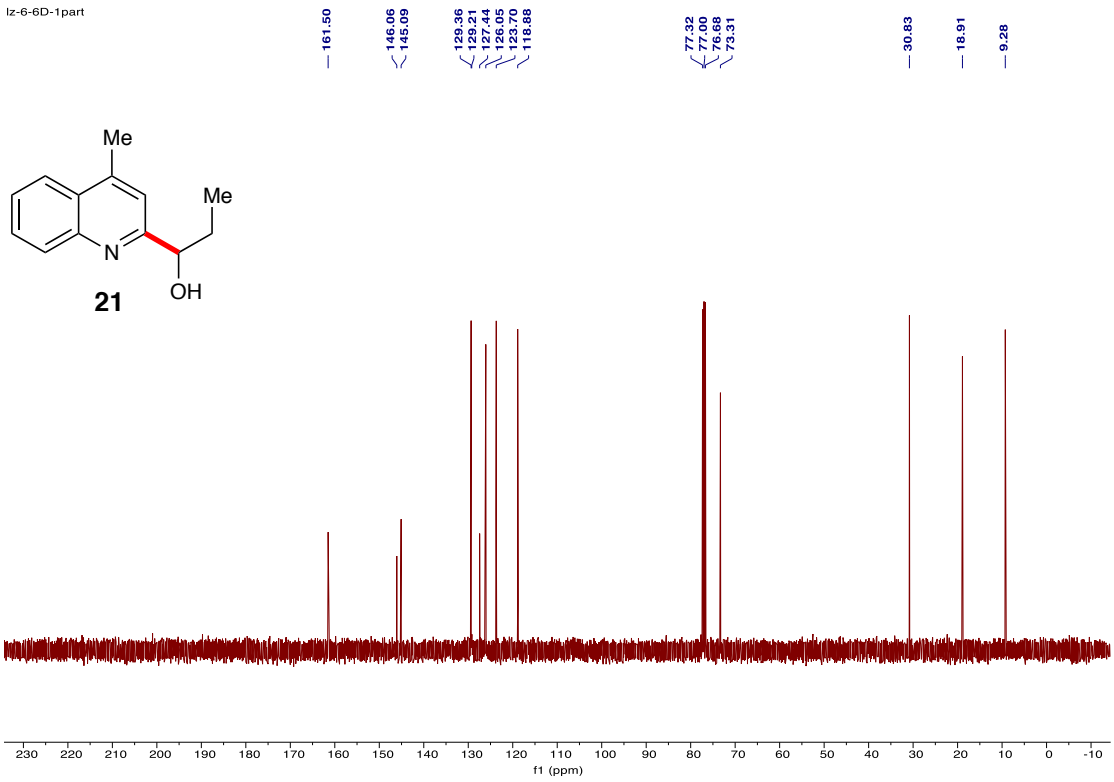

2011746-lz-6-6A-1part10.fid
8.07
8.05
7.96
7.94
7.77
7.71
7.70
7.69
7.69
7.68
7.67
7.56
7.55
7.54
7.54
7.53
7.52
7.52
7.26
7.11
4.86
4.55
2.67
Me
N
OH
19
0.99
1.00
1.00
1.00
0.99
2.00
0.95
3.01
f1 (ppm)

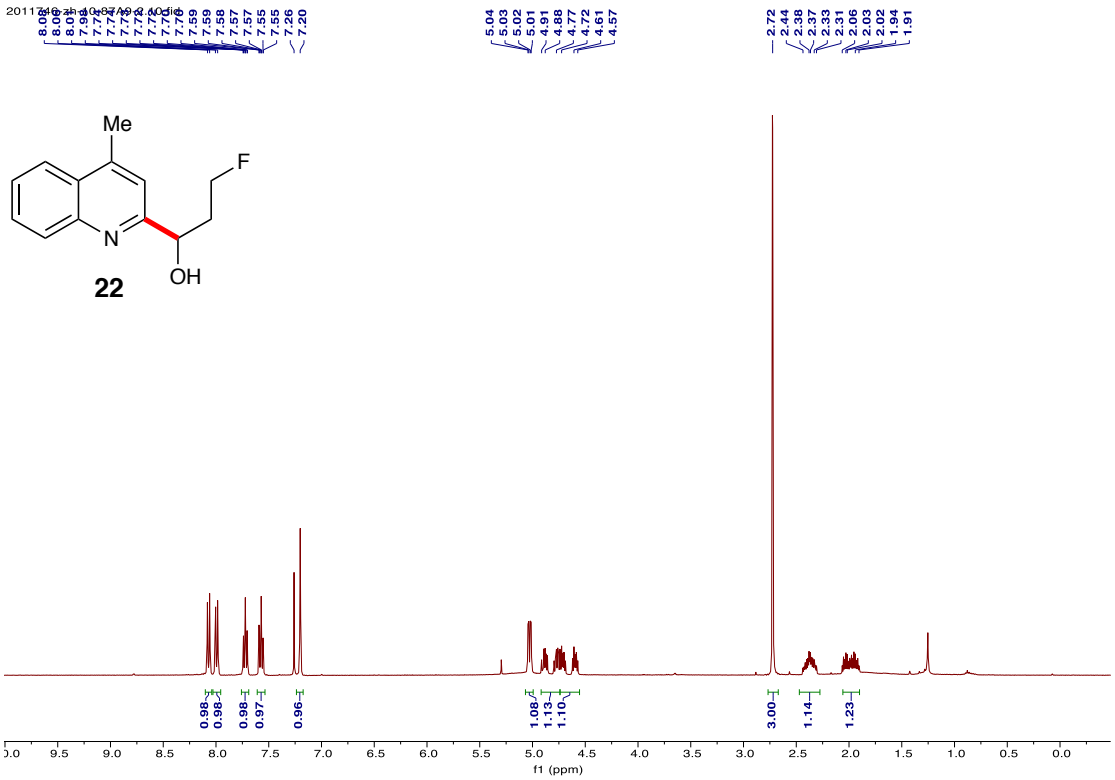

lz-6-6A-1part
158.69
146.45
145.09
129.41
129.08
127.57
126.05
123.79
118.96
77.36
77.05
76.73
64.03
18.79
Me
N
OH
19
f1 (ppm)

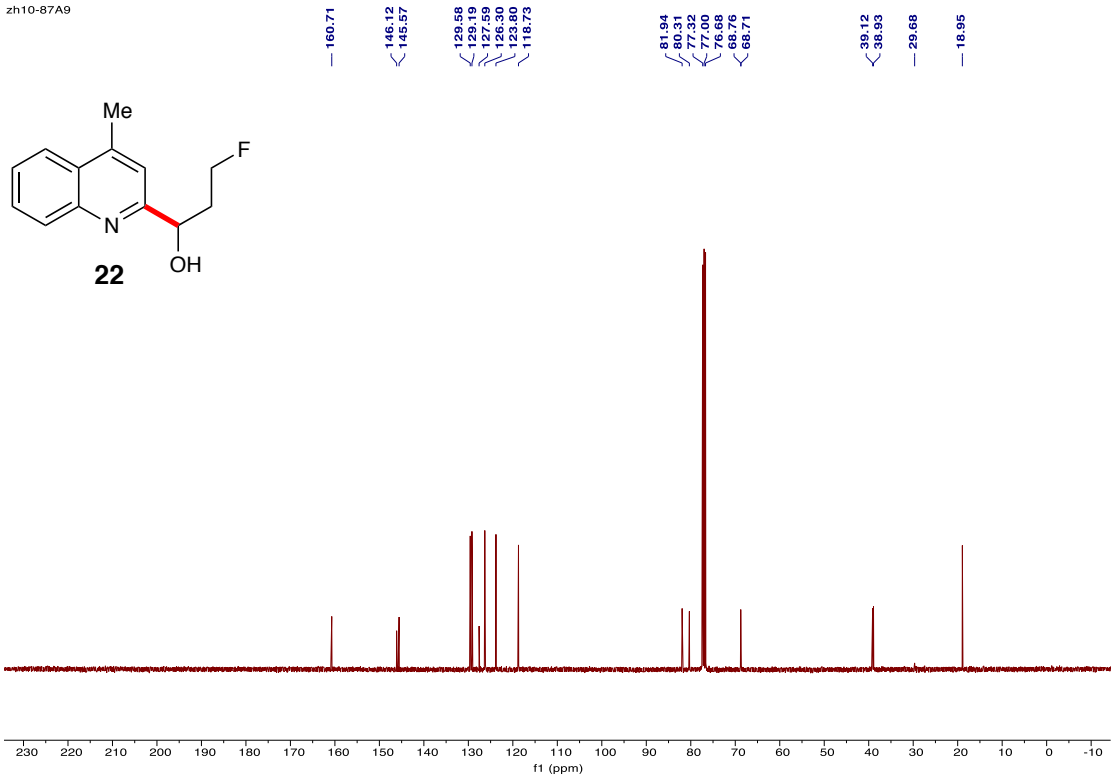

Me
Me
N
OH
20
8.09
8.08
7.98
7.96
7.74
7.72
7.71
7.70
7.69
7.68
7.67
7.57
7.55
7.55
7.55
7.53
7.53
7.26
7.18
5.14
5.01
5.00
4.98
4.96
2.71
1.57
1.56
0.97
0.97
0.98
0.97
0.98
0.89
1.00
3.00
3.01
f1 (ppm)

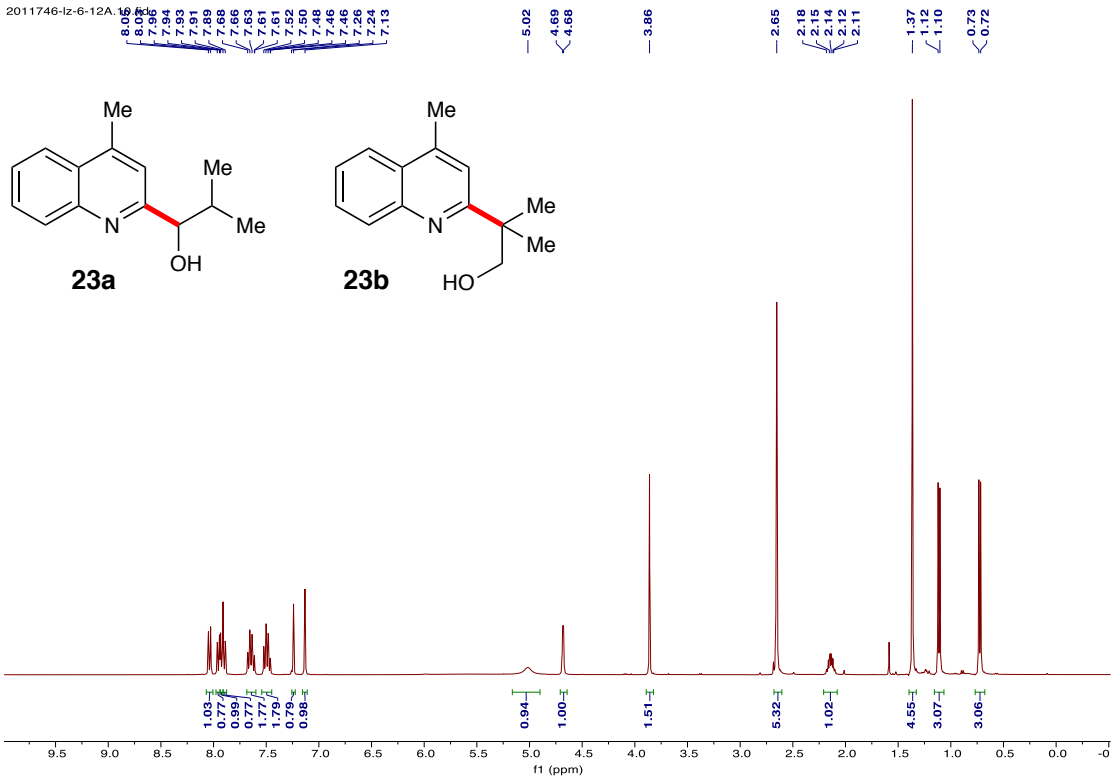

lz-6-6B-1part
Me
Me
N
OH
20
162.44
146.02
145.32
129.42
129.19
127.41
126.09
123.69
118.53
77.32
77.00
76.68
68.58
24.04
18.91
f1 (ppm)

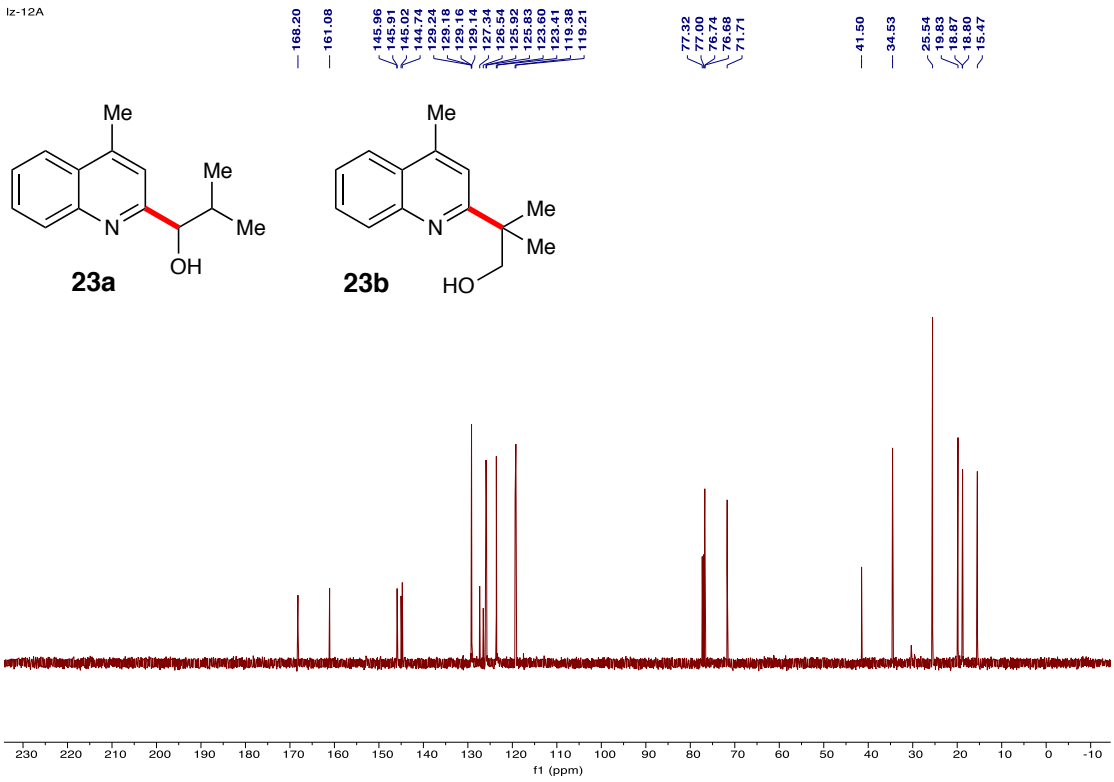

Me
Me
N
OH
21
f1 (ppm)

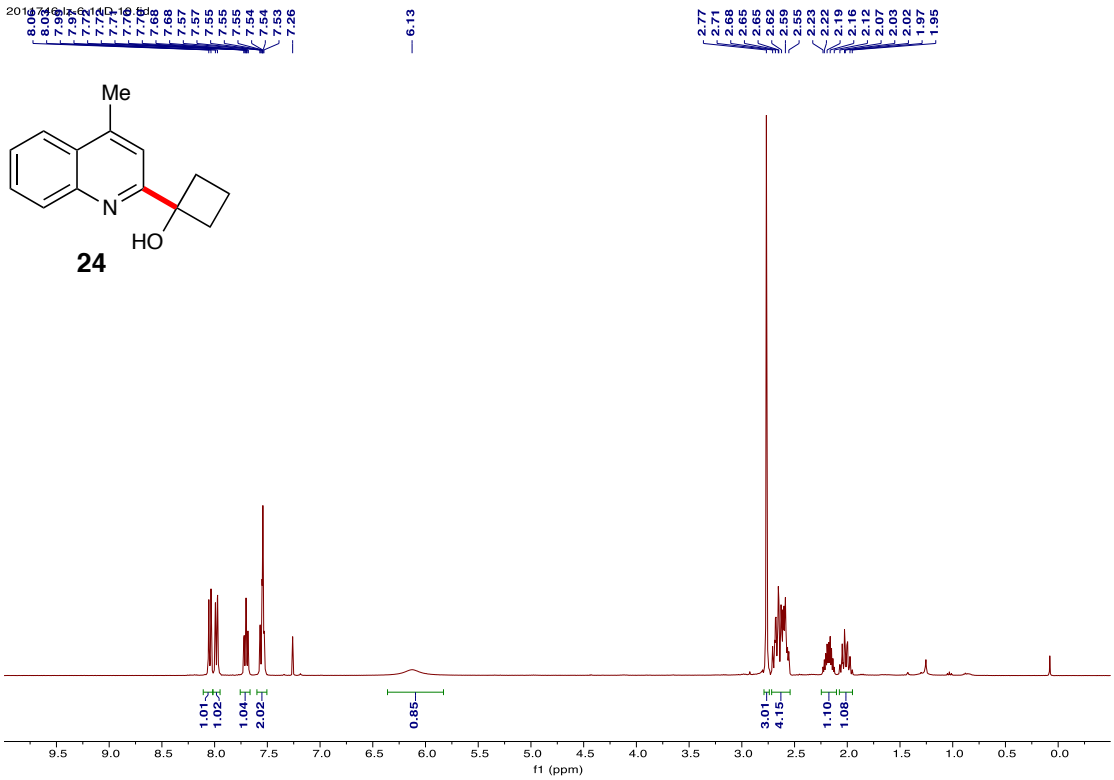

lz-6-6D-1part
Me
Me
N
OH
21
f1 (ppm)

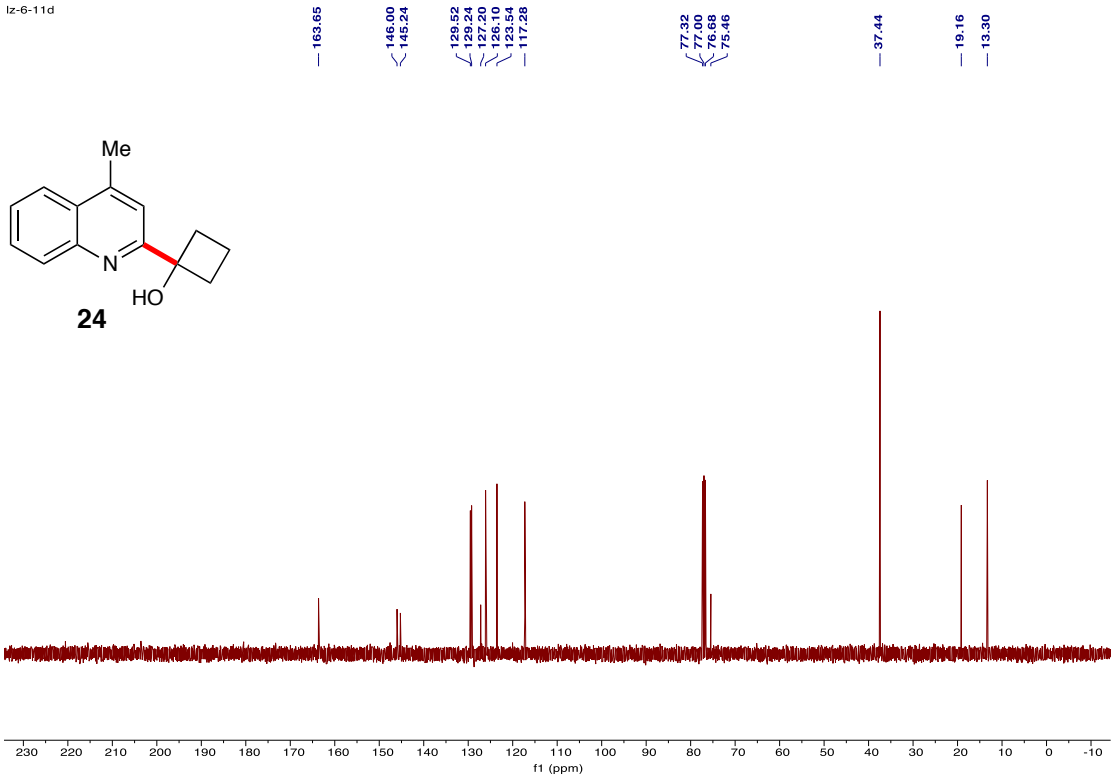

Me
F
N
OH
22
f1 (ppm)

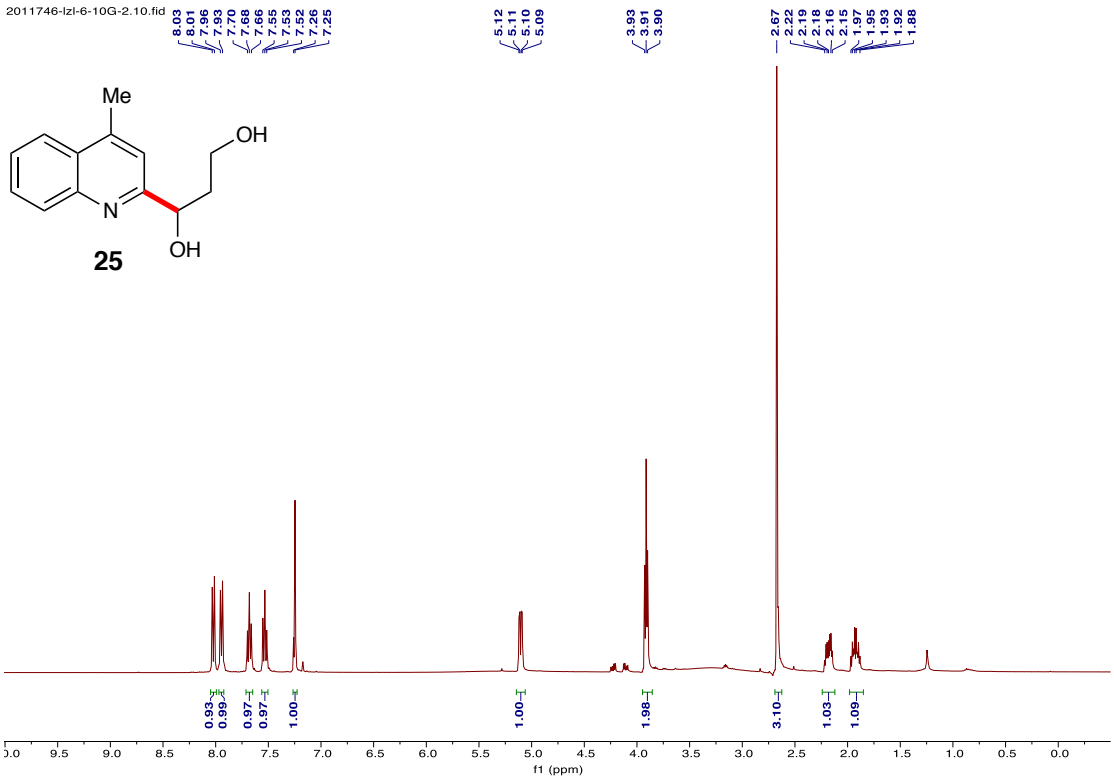

zh10-87A9
Me
F
N
OH
22
f1 (ppm)

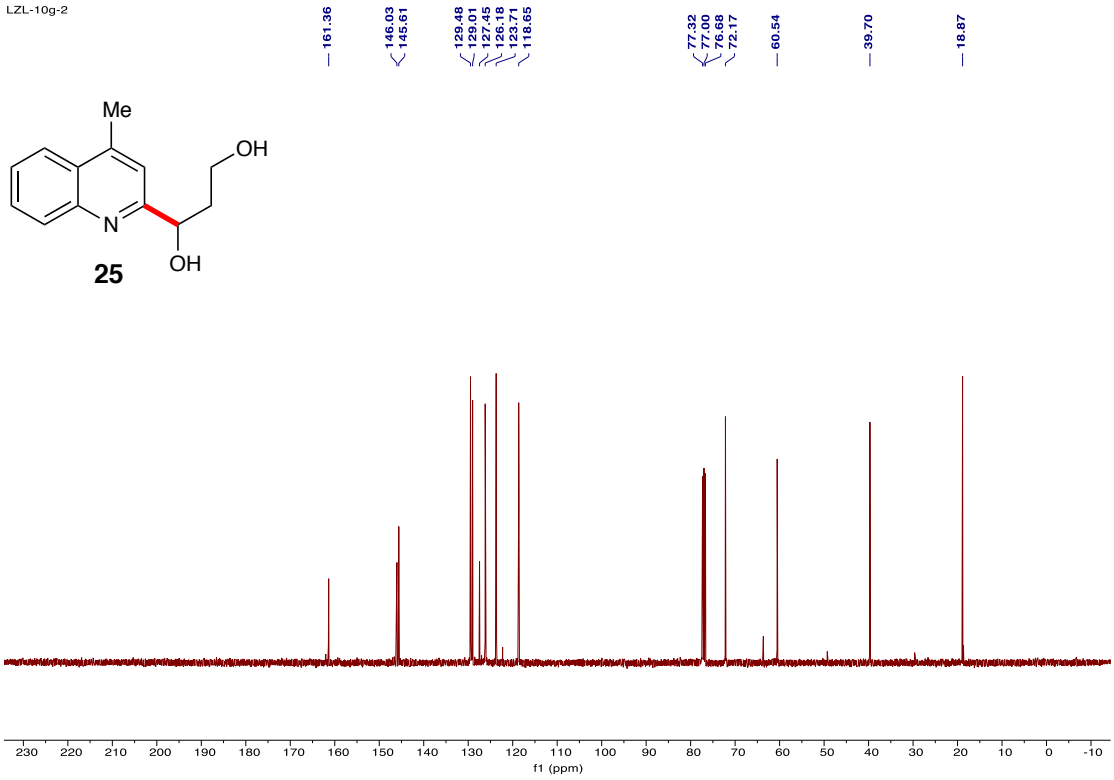

2011746-lz-6-12A.10.fid
8.05
8.03
7.96
7.94
7.93
7.91
7.89
7.68
7.66
7.63
7.61
7.61
7.52
7.50
7.48
7.46
7.46
7.26
7.24
7.13
5.02
4.69
4.68
3.86
2.65
2.18
2.15
2.14
2.12
2.11
1.37
1.12
1.10
0.73
0.72
Me
Me
Me
N
OH
23a
Me
Me
Me
N
HO
23b
1.03
0.77
0.99
0.77
1.77
1.79
0.79
0.98
0.94
1.00
1.51
5.32
1.02
4.55
3.07
3.06
f1 (ppm)

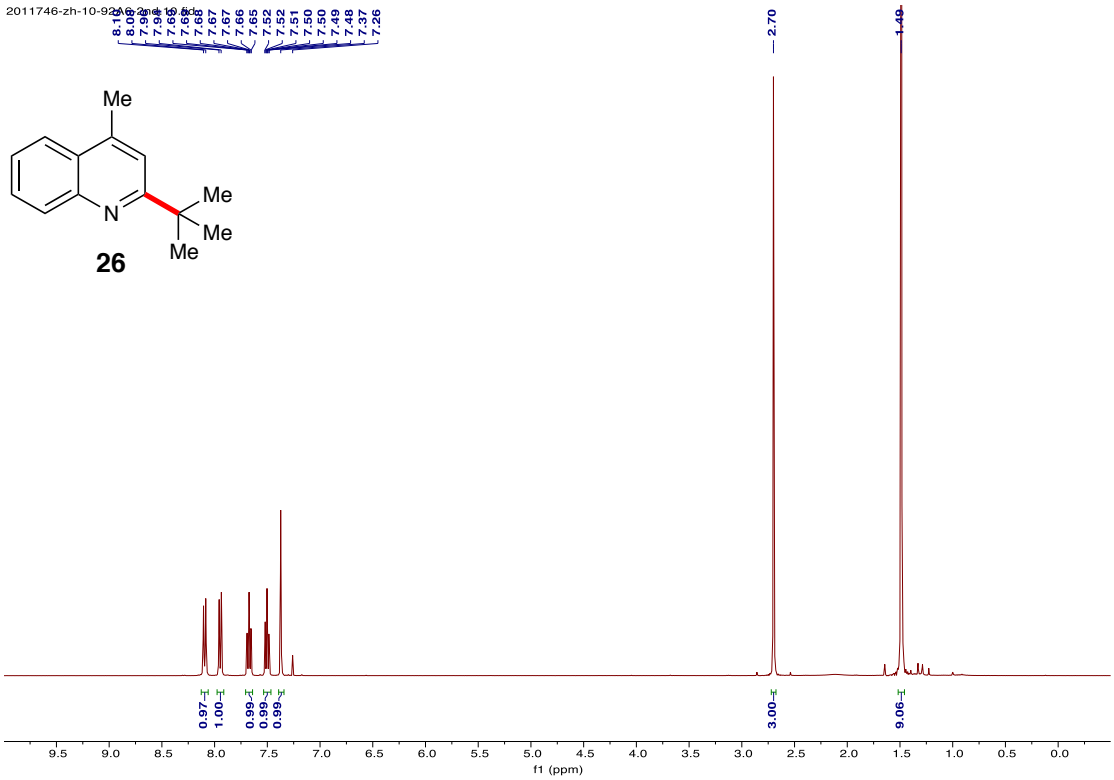

lz-12A
168.20
161.08
145.96
145.91
145.02
144.74
129.24
129.18
129.16
129.14
127.34
126.54
125.92
125.83
123.60
123.41
119.38
119.21
77.32
77.00
76.74
76.68
71.71
41.50
34.53
25.54
19.83
18.87
18.80
15.47
Me
Me
Me
N
OH
23a
Me
Me
Me
N
HO
23b
f1 (ppm)

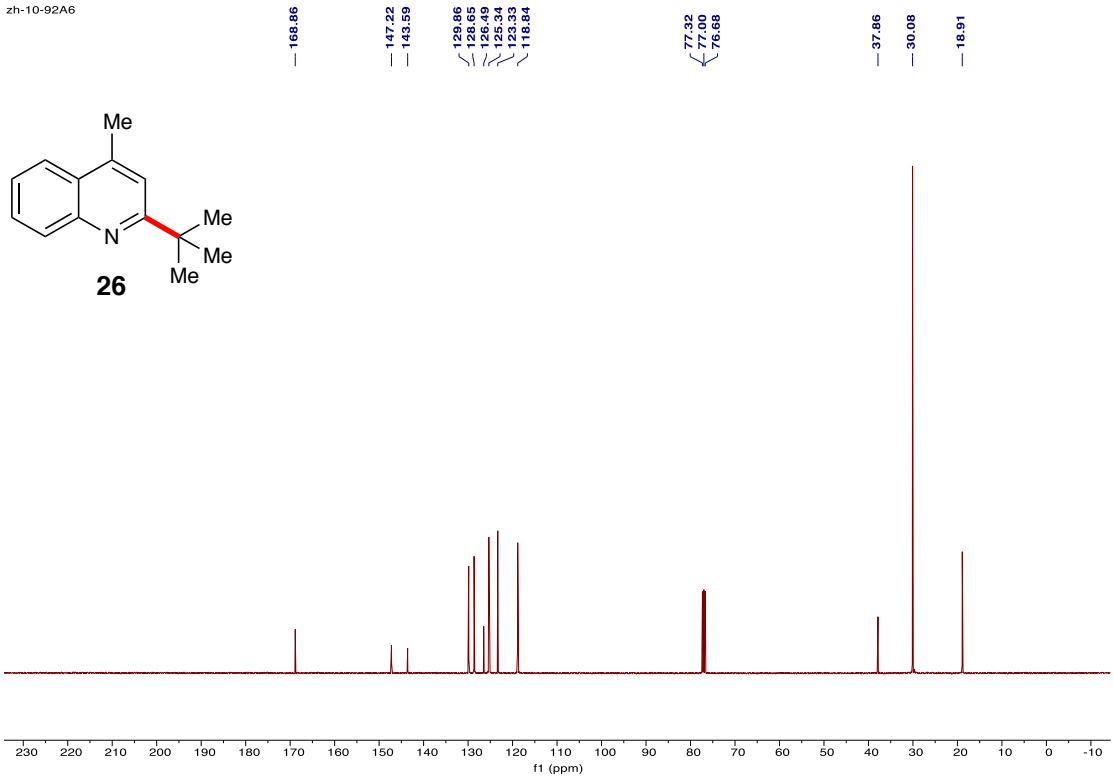

Me
N
HO
24
f1 (ppm)

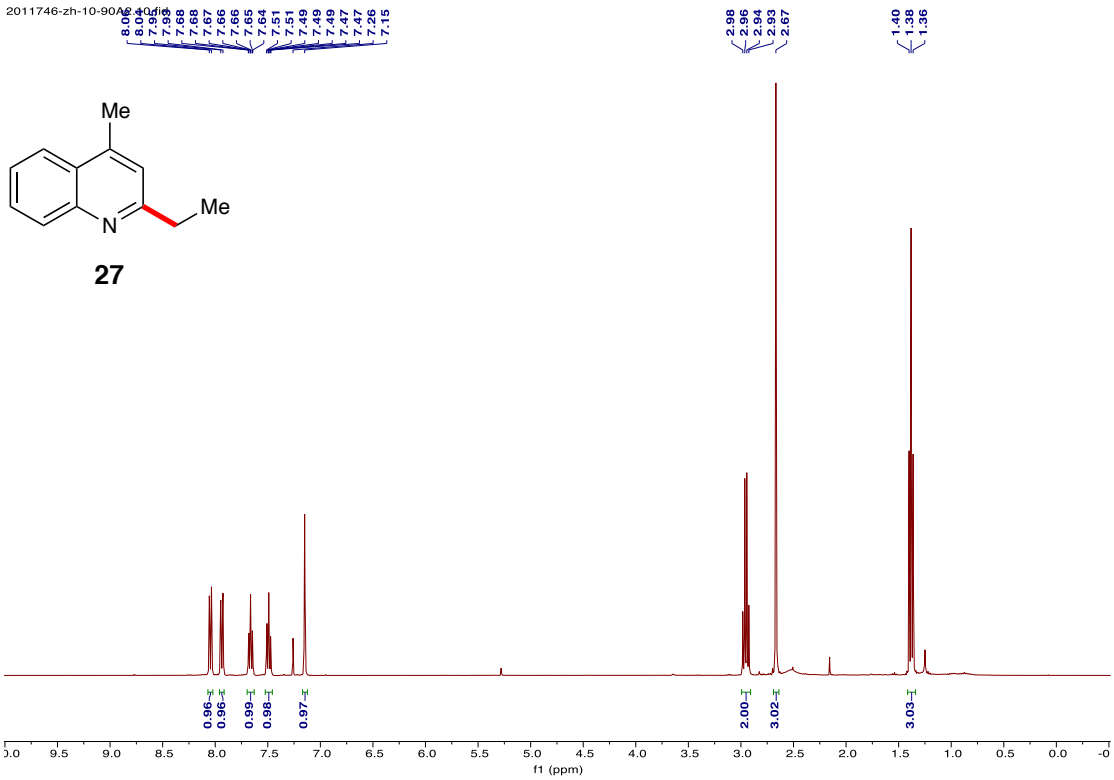

lz-6-11d
Me
N
HO
24
f1 (ppm)


2011746-lzl-6-10G-2.10.fid
8.03
8.01
7.96
7.93
7.70
7.68
7.66
7.55
7.53
7.52
7.26
7.25
5.12
5.11
5.10
5.09
3.93
3.91
3.90
2.67
2.22
2.19
2.18
2.16
2.15
1.97
1.95
1.93
1.92
1.88
Me
OH
N
OH
25
0.93
0.99
0.97
0.97
1.00
1.00
1.98
3.10
1.03
1.09
f1 (ppm)

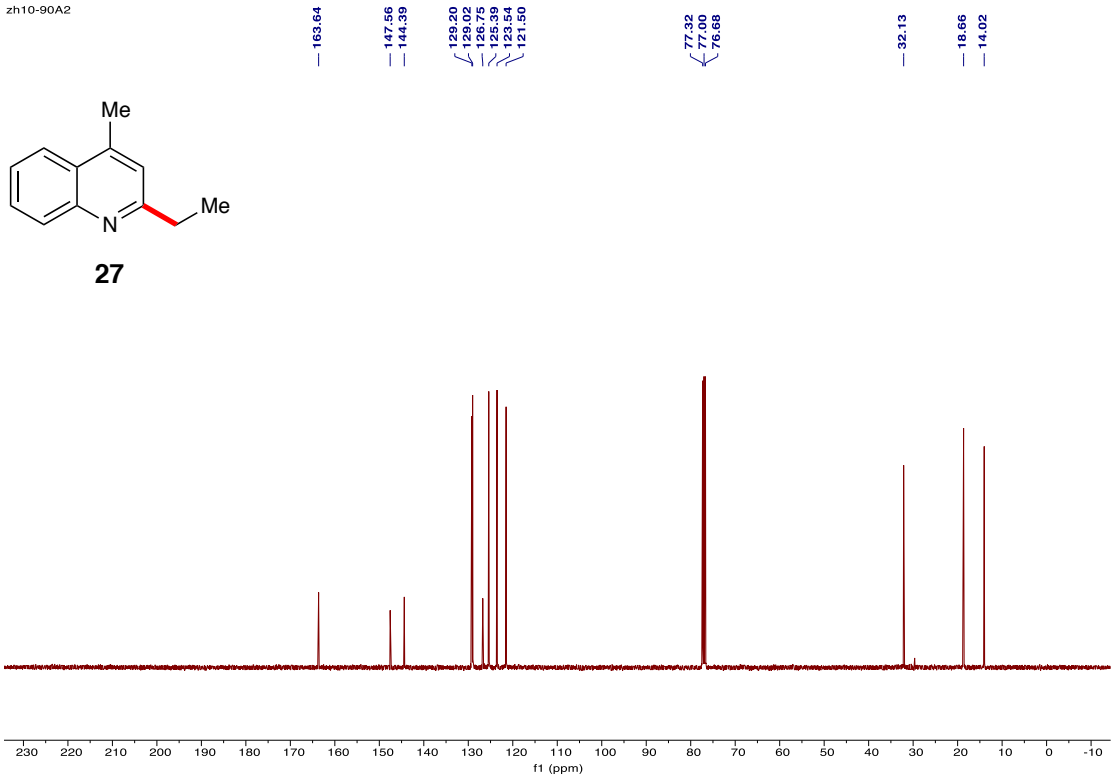

LZL-10g-2
161.36
146.03
145.61
129.48
129.01
127.45
126.18
123.71
118.65
77.32
77.00
76.68
72.17
60.54
39.70
18.87
Me
OH
N
OH
25
f1 (ppm)


2011746-zh-10-92A6
8.19
8.08
7.96
7.94
7.68
7.68
7.67
7.67
7.66
7.65
7.52
7.51
7.50
7.50
7.49
7.48
7.37
7.26
2.70
1.49
Me
N
Me
Me
Me
26
0.97
1.00
0.99
0.99
0.99
3.00
9.06
f1 (ppm)

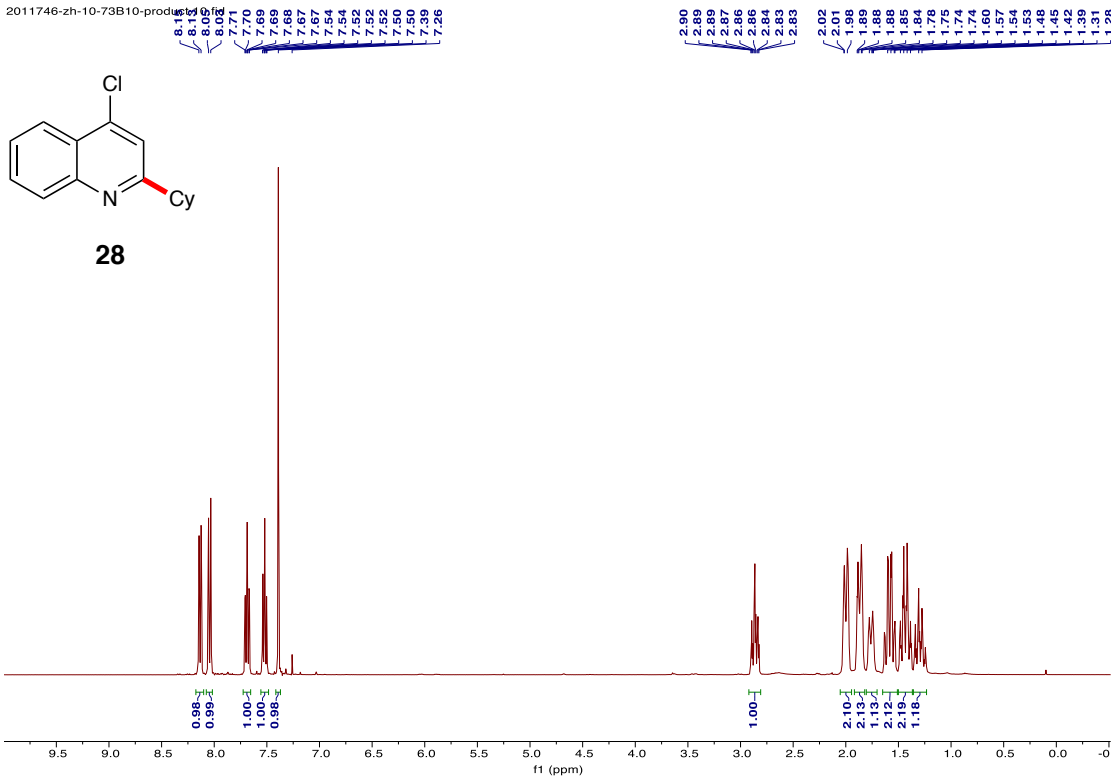

zh-10-92A6
168.86
147.22
143.59
129.86
128.65
126.49
125.34
123.33
118.84
77.32
77.00
76.68
37.86
30.08
18.91
Me
N
Me
Me
Me
26
f1 (ppm)

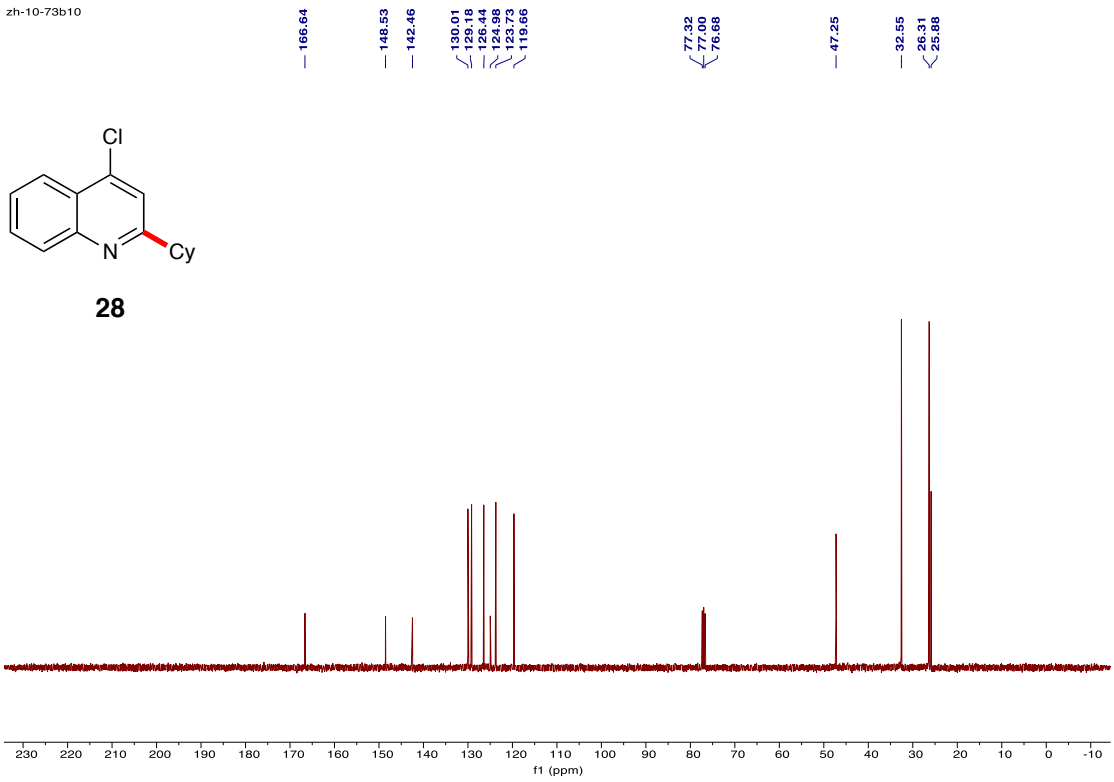

2011746-zh-10-90A2-10.fid
8.06
8.04
7.95
7.93
7.68
7.68
7.67
7.66
7.66
7.65
7.64
7.51
7.51
7.49
7.49
7.49
7.47
7.47
7.26
7.15
2.98
2.96
2.94
2.93
2.67
1.40
1.38
1.36
Me
Me
N
0.96
0.96
0.99
0.98
0.97
2.00
3.02
3.03
f1 (ppm)

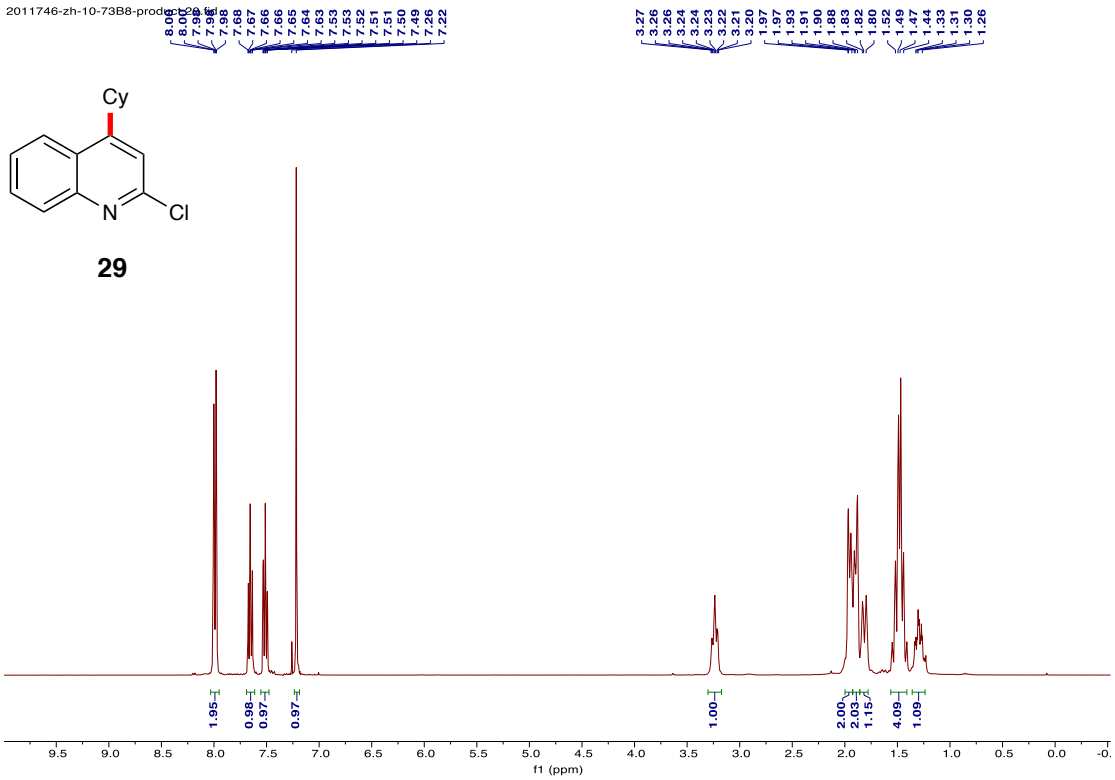
27

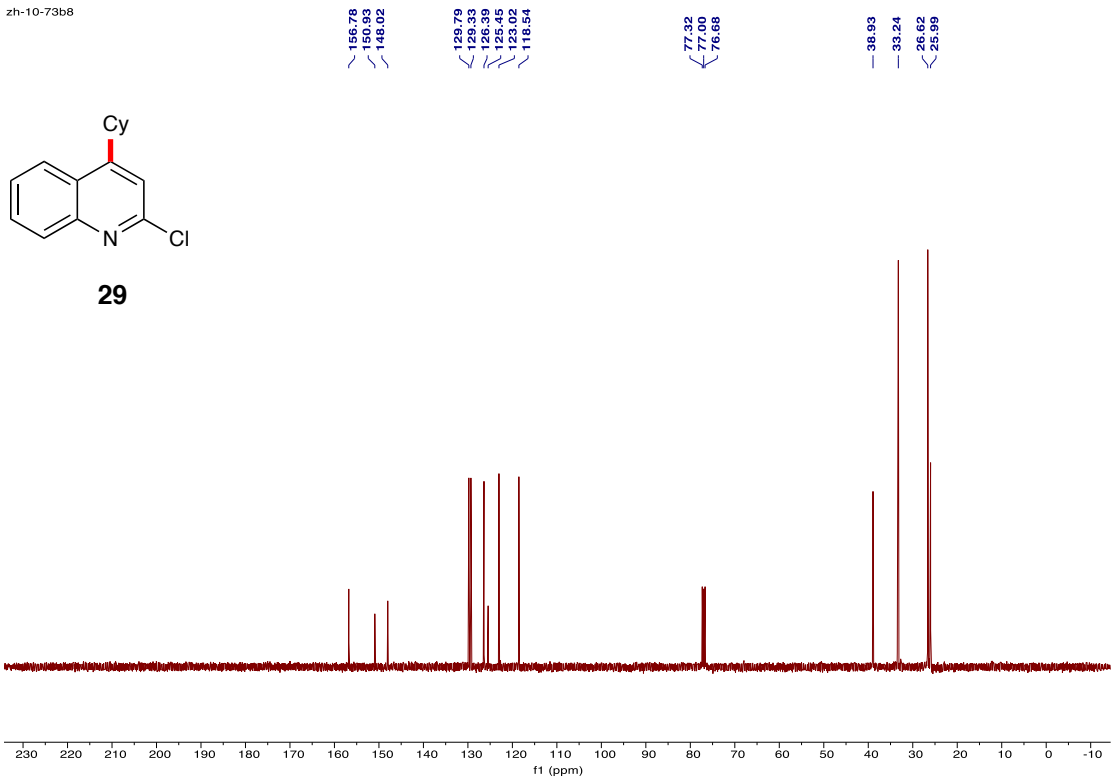

zh10-90A2
163.64
147.56
144.39
129.20
129.02
126.75
125.39
123.54
121.50
77.32
77.00
76.68
32.13
18.66
14.02
Me
Me
N
f1 (ppm)

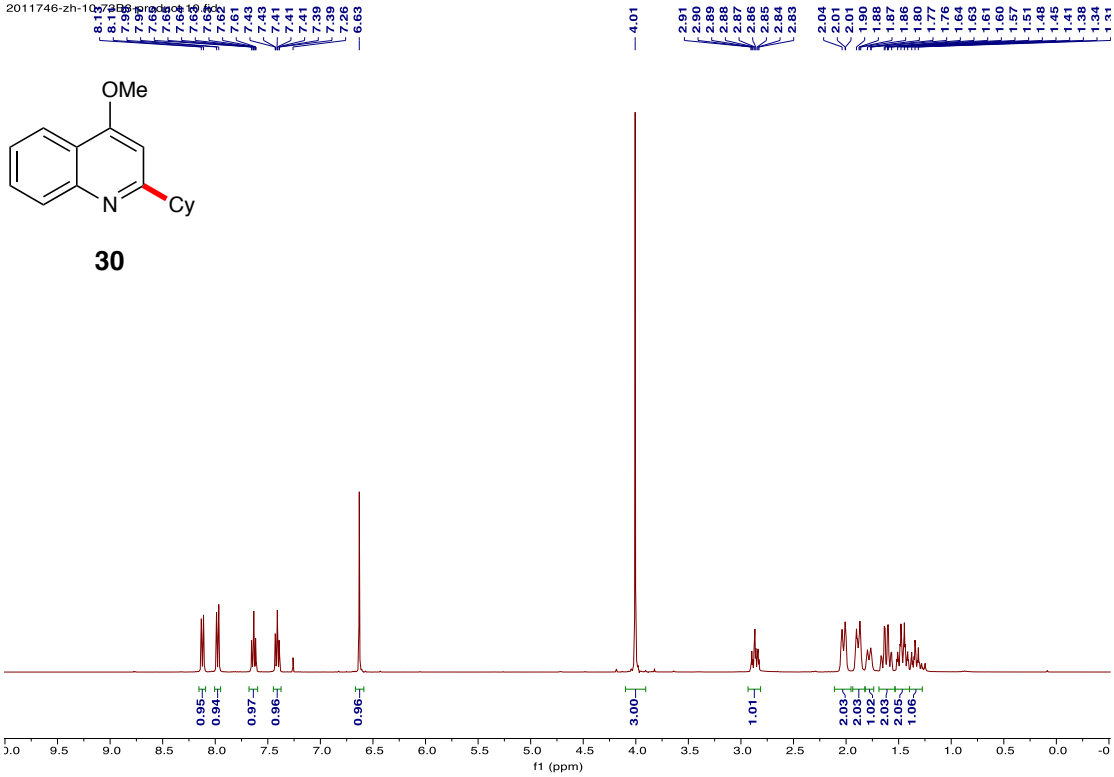
27

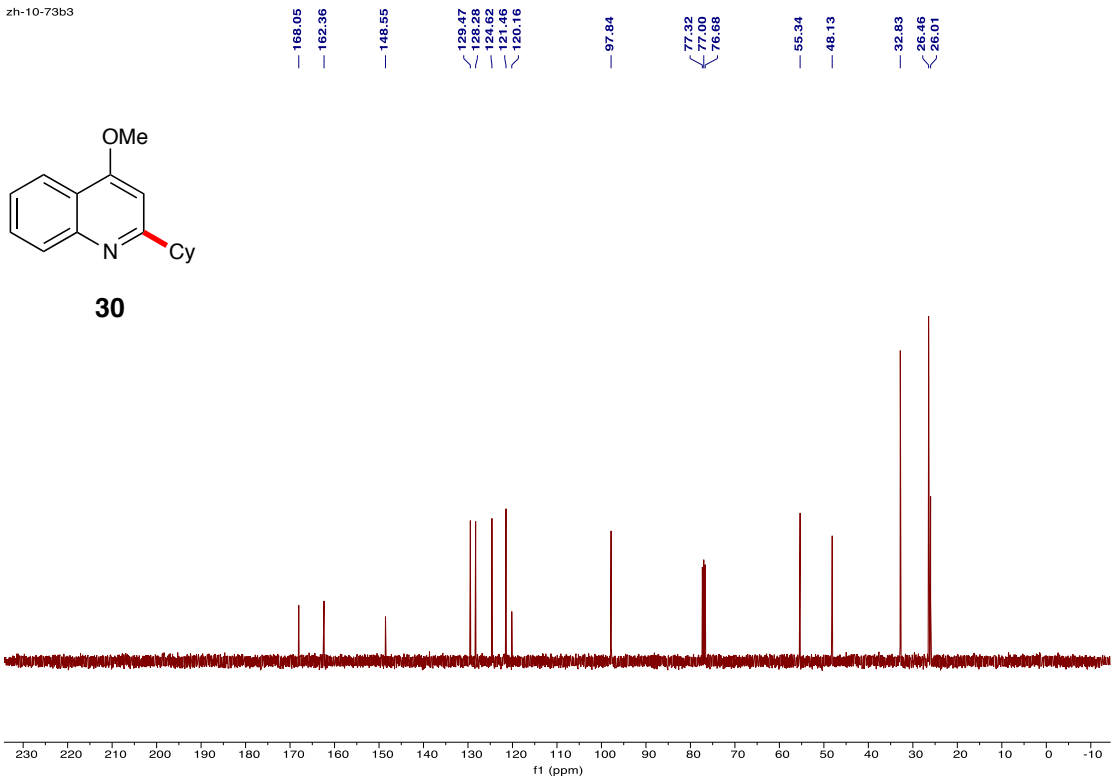

2011746-zh-10-73B10-product-1H-fid
Cl
N
Cy
28
f1 (ppm)

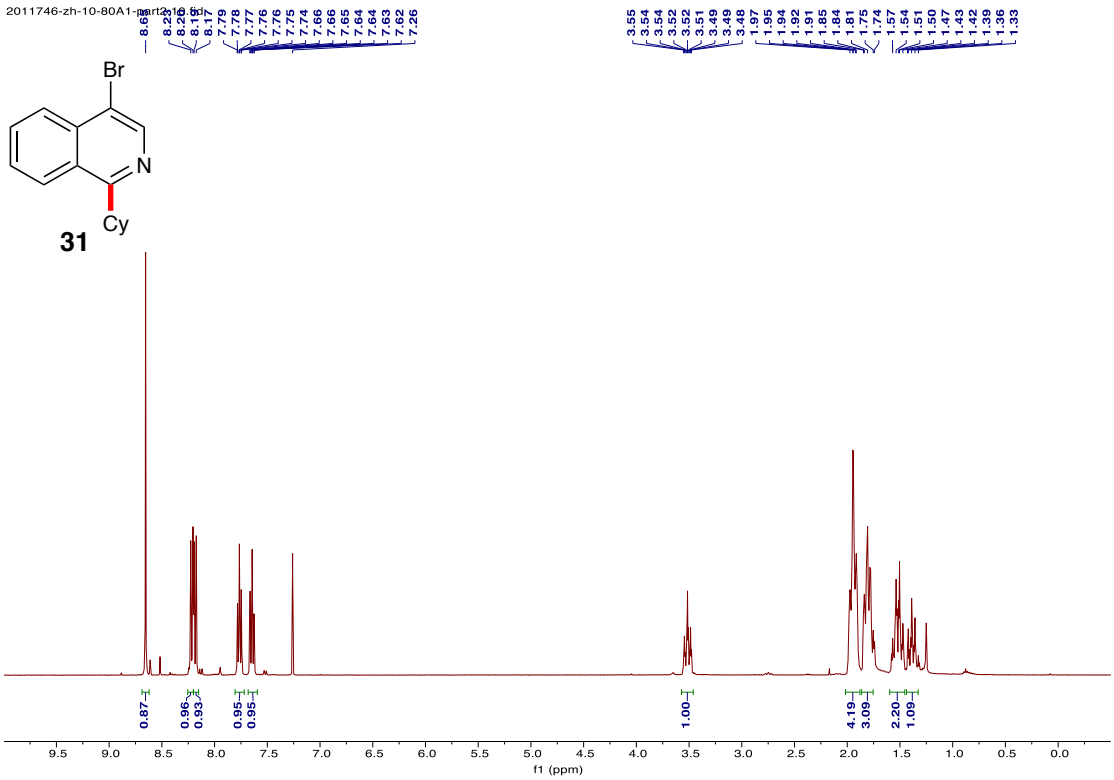

zh-10-73b10
Cl
N
Cy
28
f1 (ppm)

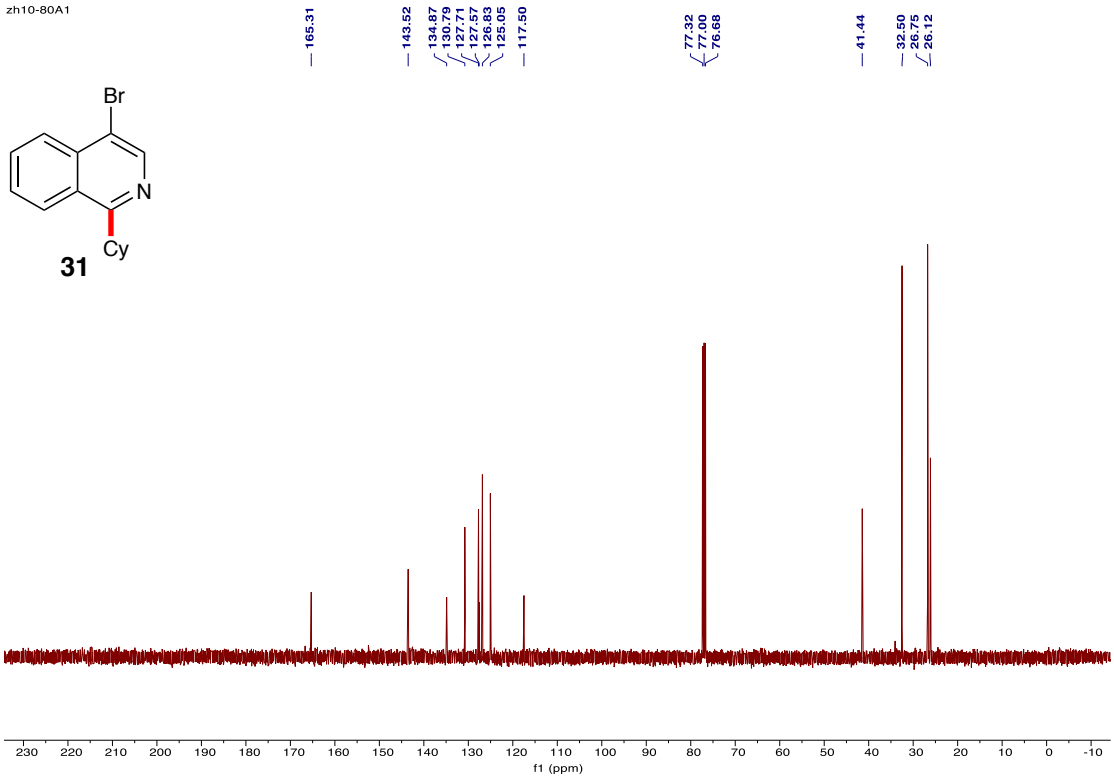

2011746-zh-10-73B8-product 29.fid
8.00
8.00
7.98
7.98
7.68
7.67
7.66
7.66
7.65
7.64
7.63
7.53
7.53
7.52
7.51
7.51
7.50
7.49
7.26
7.22
3.27
3.26
3.26
3.24
3.24
3.23
3.22
3.21
3.20
1.97
1.97
1.93
1.91
1.90
1.88
1.83
1.82
1.80
1.52
1.49
1.47
1.44
1.33
1.31
1.30
1.26
Cy
N
Cl
29
1.95
0.98
0.97
0.97
1.00
2.00
2.03
1.15
4.09
1.09
f1 (ppm)

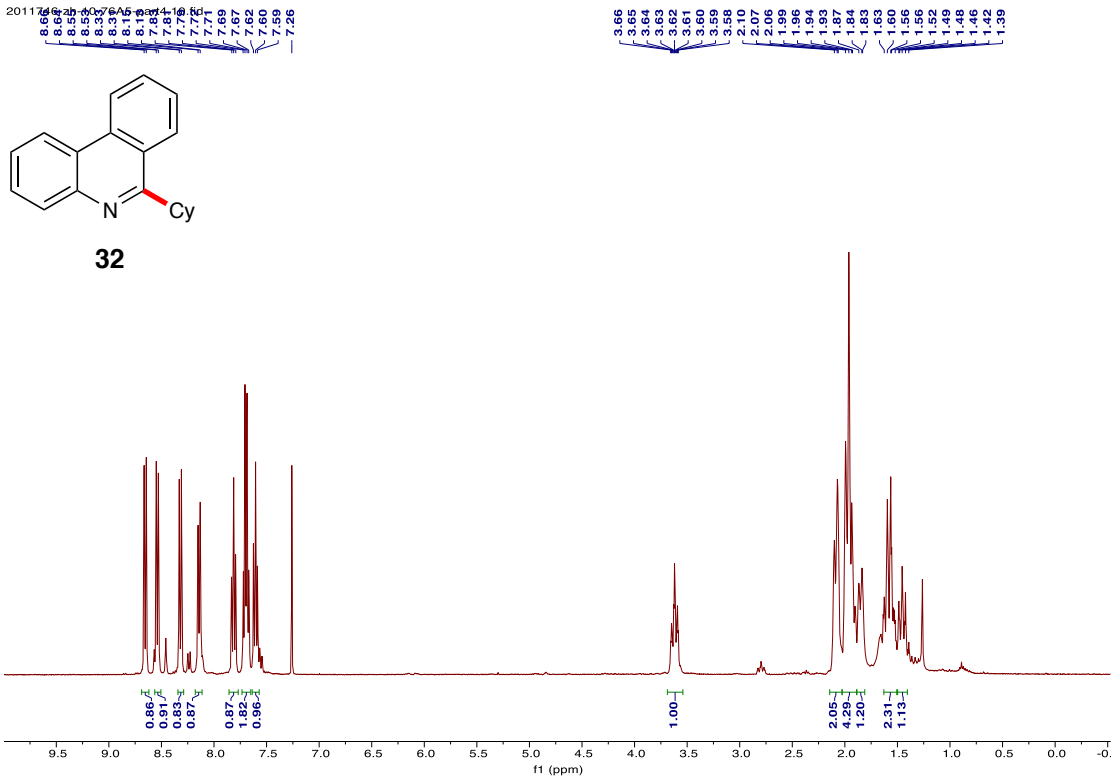

zh-10-73b8
156.78
150.93
148.02
129.79
129.33
126.39
125.45
123.02
118.54
77.32
77.00
76.68
38.93
33.24
26.62
25.99
Cy
N
Cl
29
f1 (ppm)

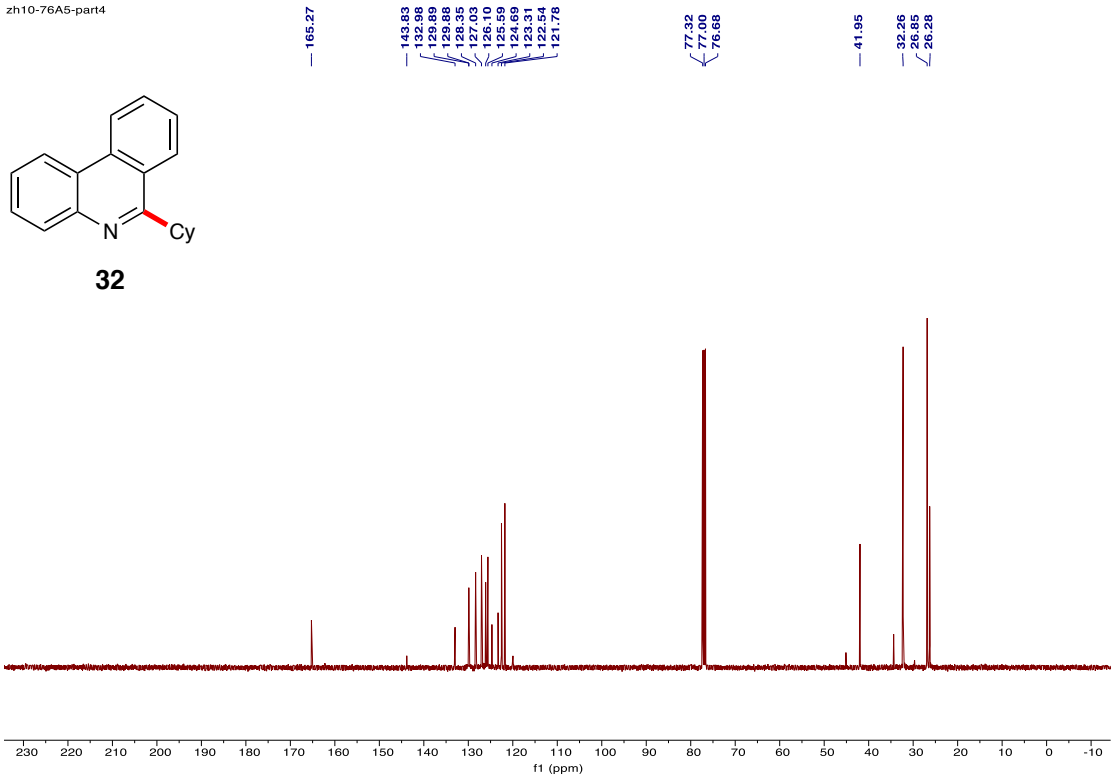

OMe
N
Cy
30
4.01
0.95
0.94
0.97
0.96
0.96
3.00
1.01
2.03
2.03
1.02
2.03
2.05
1.06
f1 (ppm)

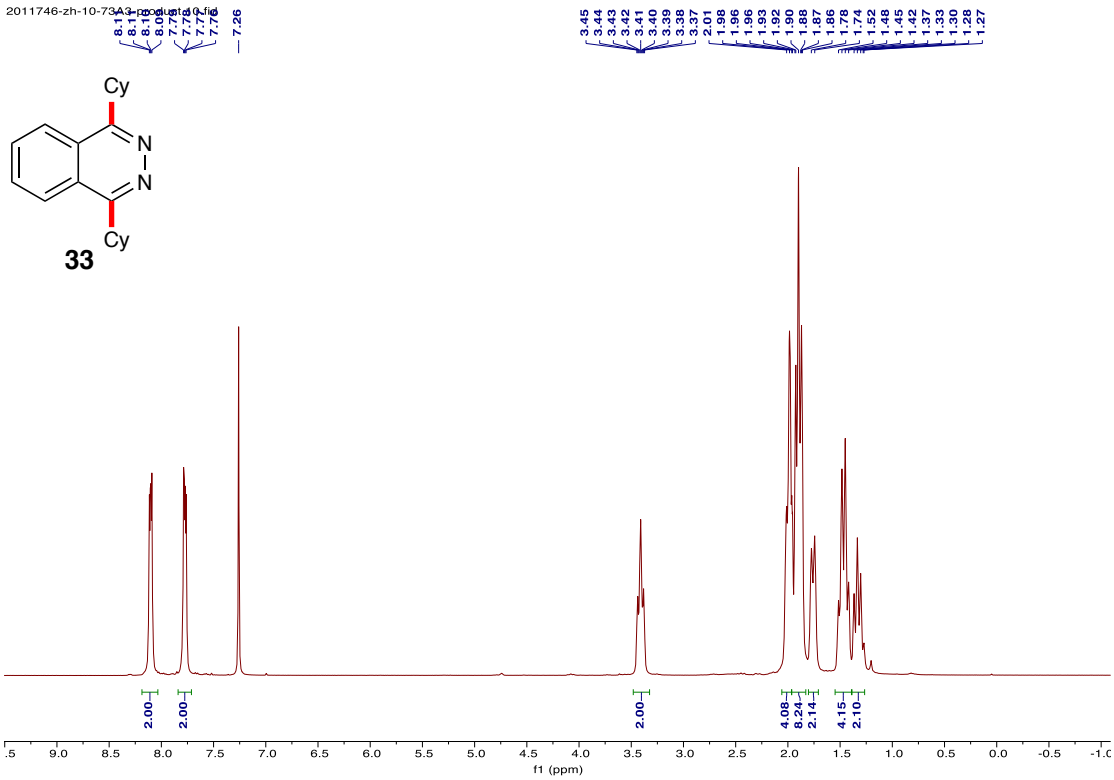

zh-10-73b3
168.05
162.36
148.55
129.47
128.28
124.62
121.46
120.16
97.84
77.32
77.00
76.68
55.34
48.13
32.83
26.46
26.01
OMe
N
Cy
30
f1 (ppm)

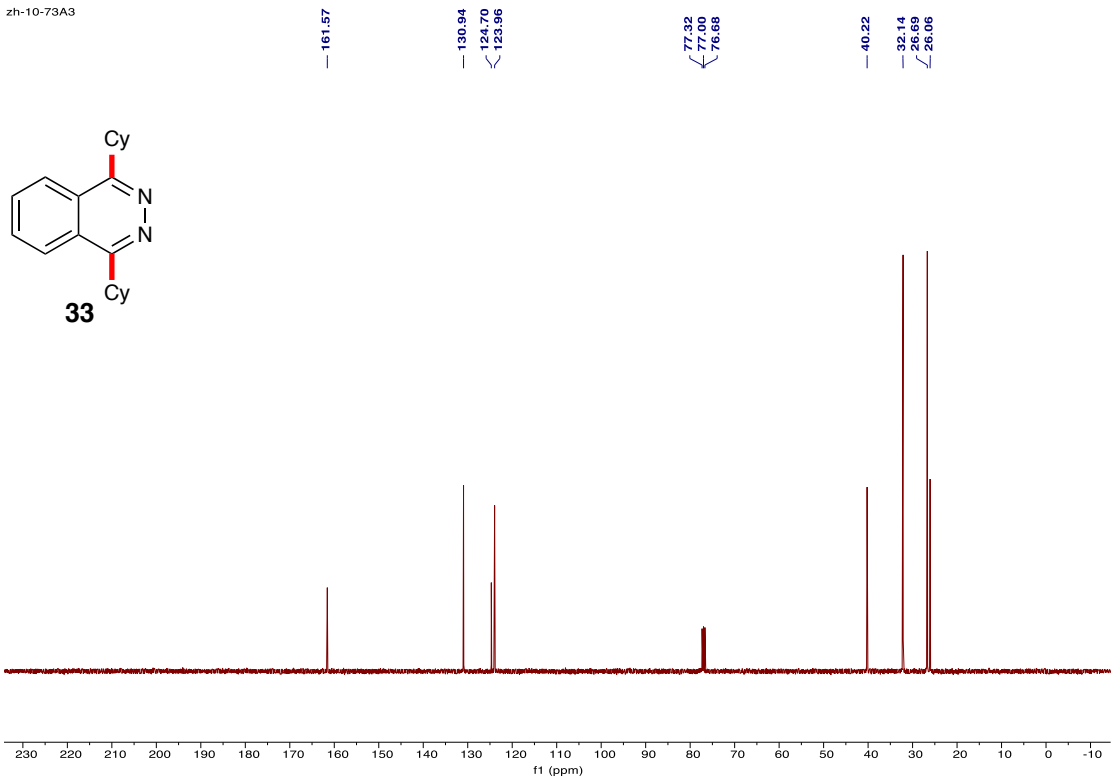

2011746-zh-10-80A1-part2-10.fid
Br
N
Cy
31
f1 (ppm)


zh10-80A1
Br
N
Cy
31
f1 (ppm)

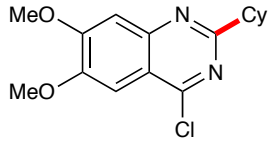

N
Cy
32
f1 (ppm)


zh10-76A5-part4
N
Cy
32
f1 (ppm)


2011746-zh-10-73A3
Cy
N
N
Cy
33
f1 (ppm)

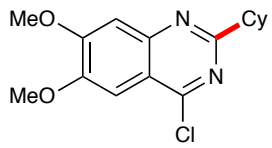

zh-10-73A3
Cy
N
N
Cy
33
f1 (ppm)

2011746-zh-11-5A10.10.fid

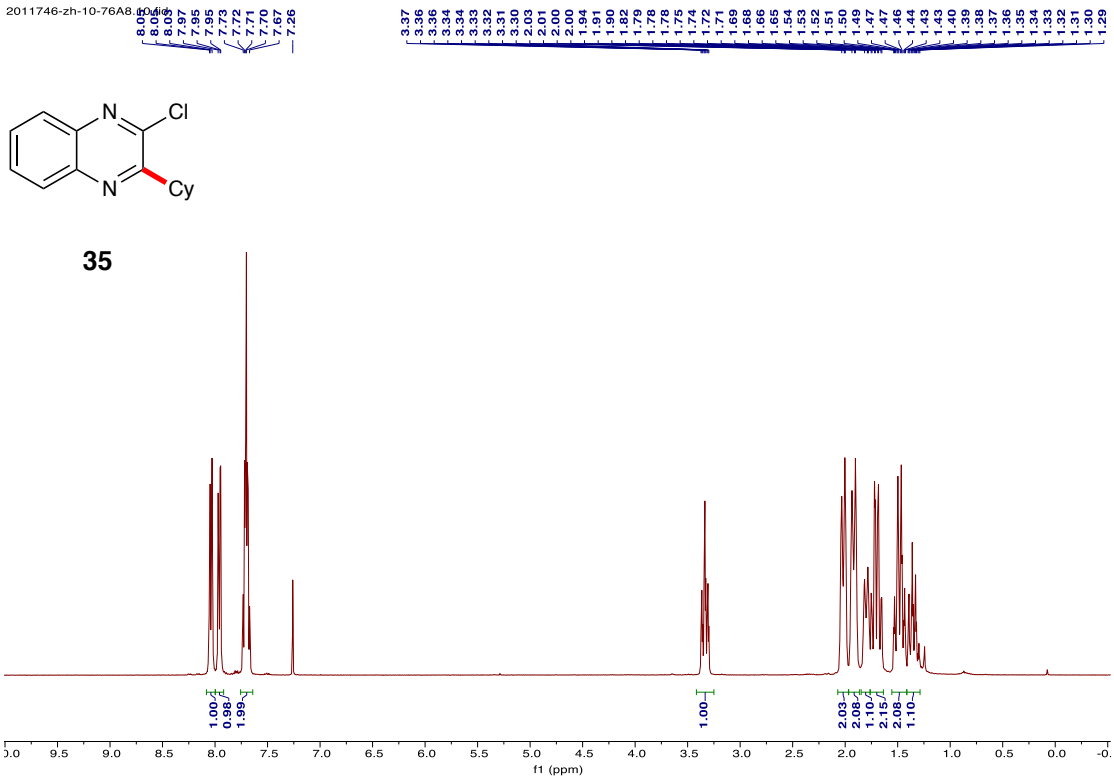


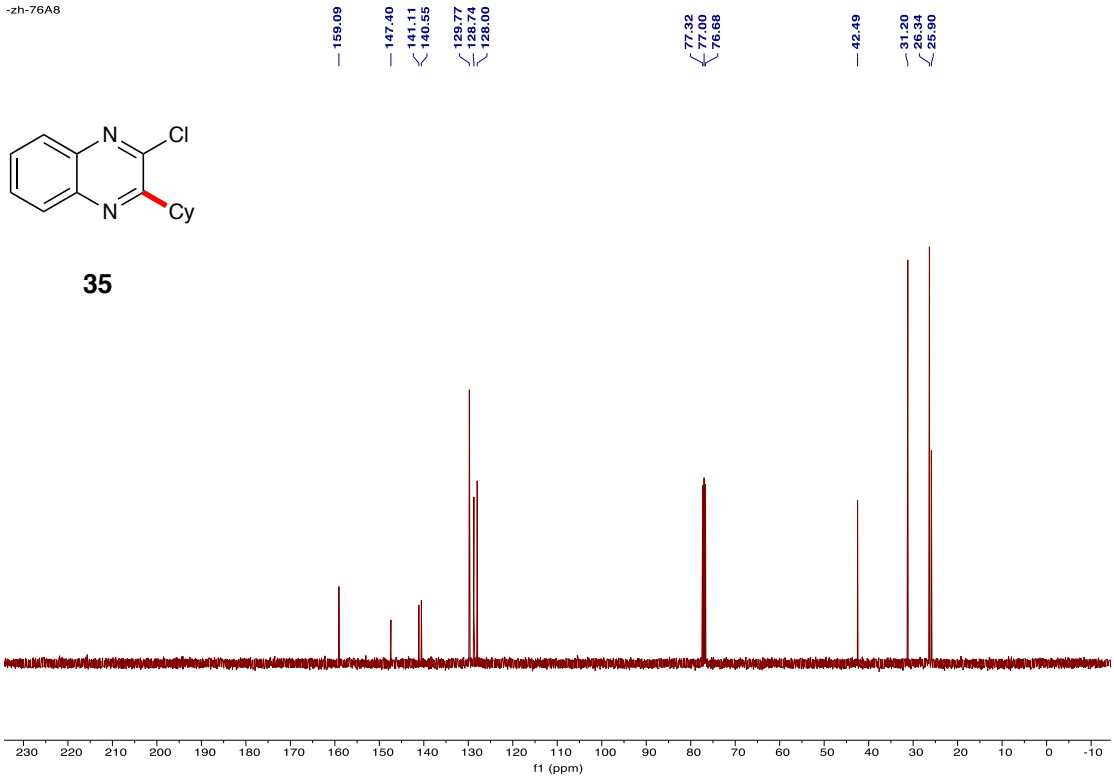
34

zh11-5A10

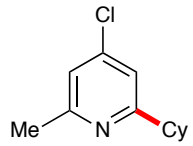


34


2011746-zh-10-76A8-1H.fid
8.05
8.04
8.03
7.97
7.95
7.95
7.73
7.72
7.71
7.70
7.67
7.26
3.37
3.36
3.36
3.34
3.34
3.33
3.32
3.31
3.30
2.03
2.01
2.00
2.00
1.94
1.91
1.90
1.82
1.79
1.78
1.78
1.75
1.74
1.72
1.71
1.69
1.68
1.66
1.65
1.54
1.53
1.52
1.51
1.50
1.49
1.47
1.47
1.46
1.44
1.43
1.43
1.40
1.39
1.38
1.37
1.36
1.35
1.34
1.33
1.32
1.31
1.30
1.29
N
Cl
N
Cy
35
1.00
0.98
1.99
1.00
2.03
2.08
1.10
2.15
2.08
1.10
f1 (ppm)

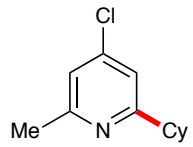

-zh-76A8
159.09
147.40
141.11
140.55
129.77
128.74
128.00
77.32
77.00
76.68
42.49
31.20
26.34
25.90
N
Cl
N
Cy
35
f1 (ppm)

2011746-zh-10-71A3.20.fid



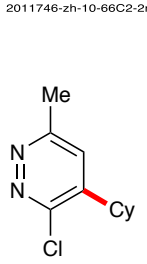
36

-zh-10-71A3



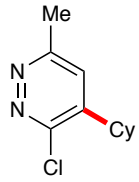
36

2011746-zh-10-66C2-2nd.10.fid



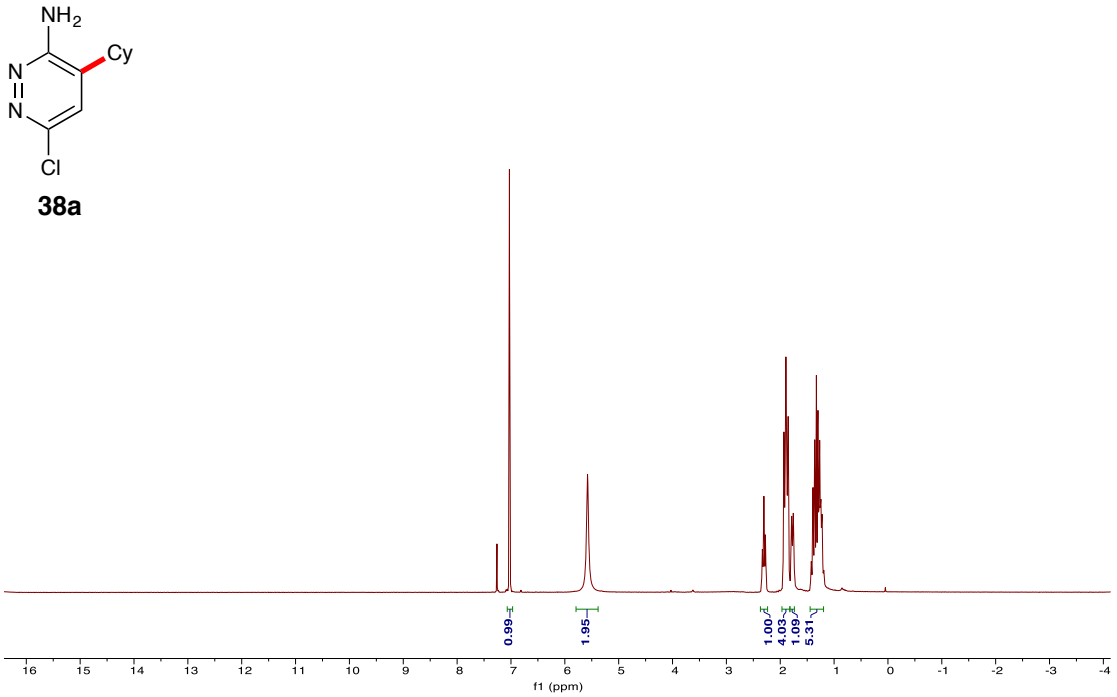
37

zh10-75A5



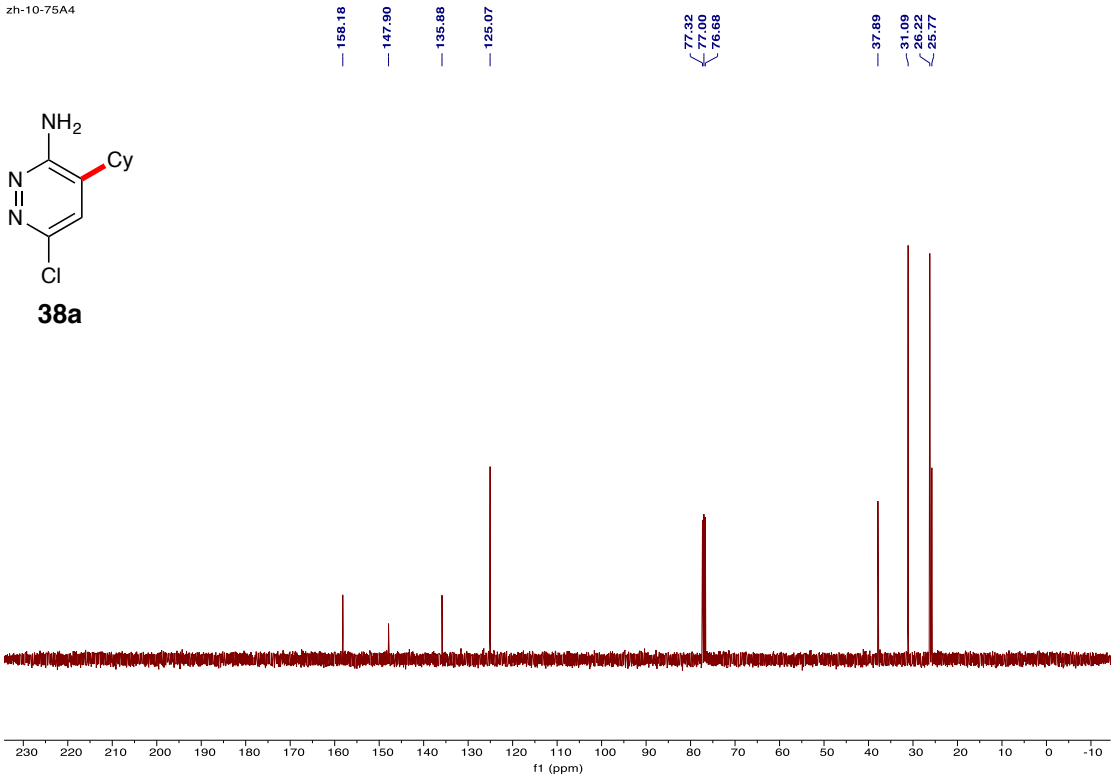
37

2011746-zh-10-75A4-3.10.fid



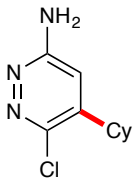
38a

zh-10-75A4



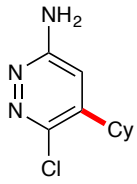
38a

2011746-zh-10-79A7-2nd.10.fid

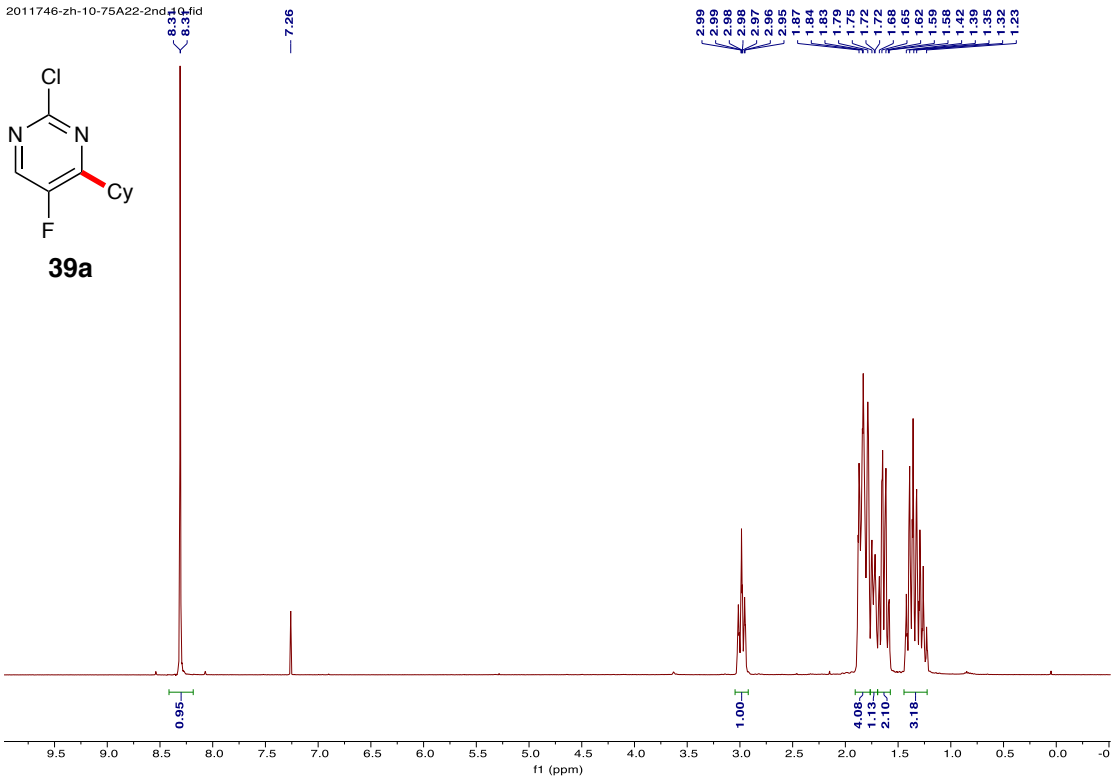


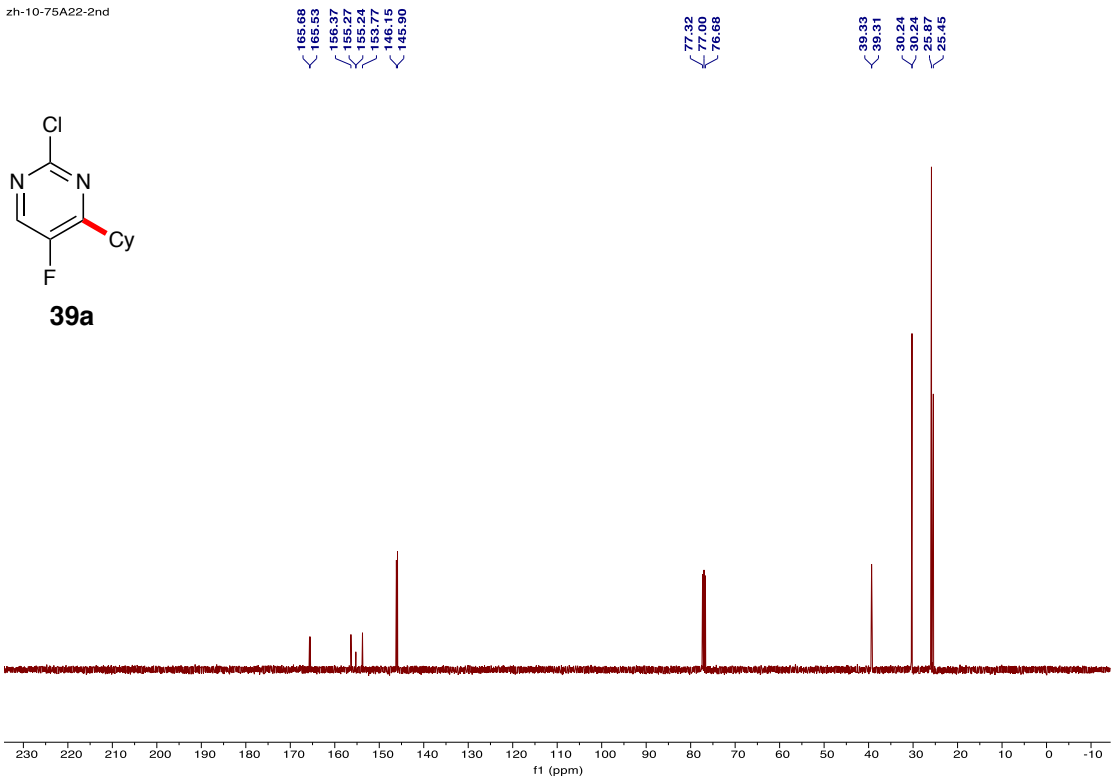
38b

zh-10-79A7-2nd

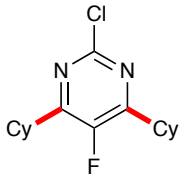


38b


2011746-zh-10-75A22-2nd-10.fid
8.39
8.39
7.26
2.99
2.99
2.98
2.98
2.97
2.96
2.95
1.87
1.84
1.83
1.79
1.75
1.72
1.72
1.68
1.65
1.62
1.59
1.58
1.42
1.39
1.35
1.32
1.23
Cl
N
N
Cy
F
39a
0.95
1.00
4.08
1.13
2.10
3.18
f1 (ppm)

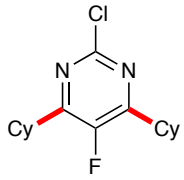

zh-10-75A22-2nd
165.68
165.53
156.37
155.27
155.24
153.77
146.15
145.90
77.32
77.00
76.68
39.33
39.31
30.24
30.24
25.87
25.45
Cl
N
N
Cy
F
39a
f1 (ppm)

2011746-zh-10-75A22-1st.10.fid



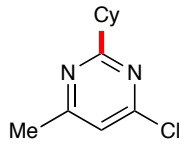
39b

zh-10-75A22-1st



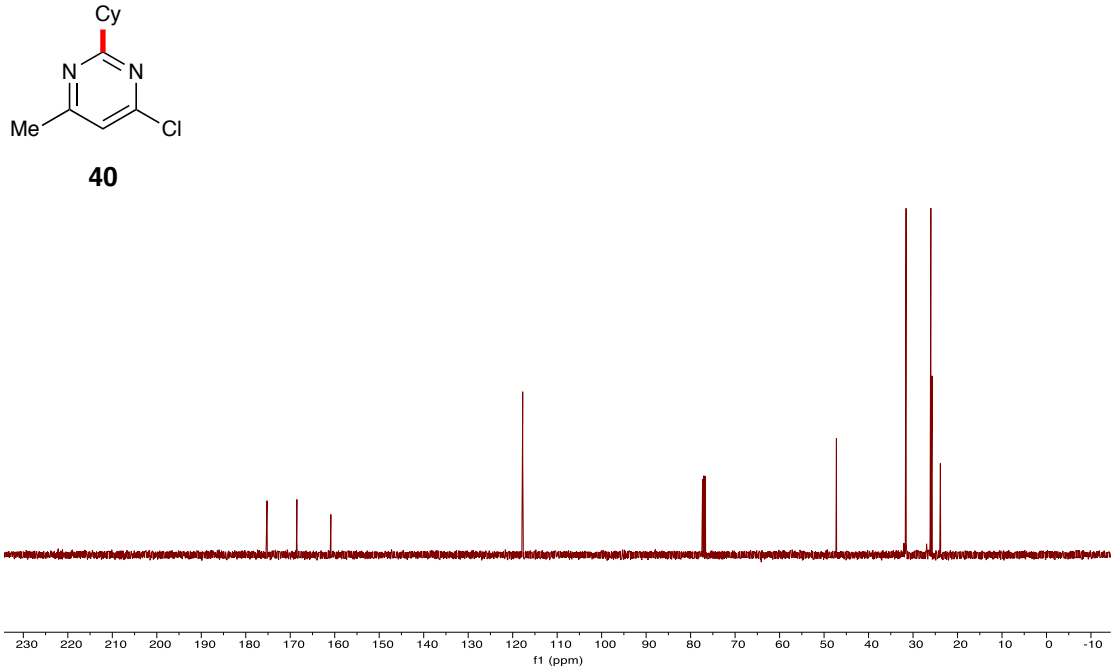
39b

2011746-zh-10-75A23-product.10.fid



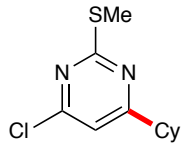
40

zh-10-75A23



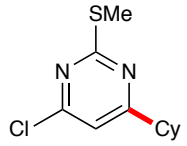
40

2011746-zh-10-75A24-part1.10.fid



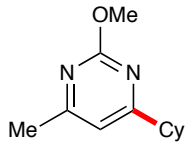
41

zh10-75A24



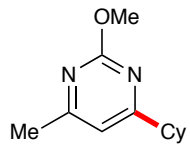
41

2011746-zh-10-75A25-product.10.fid

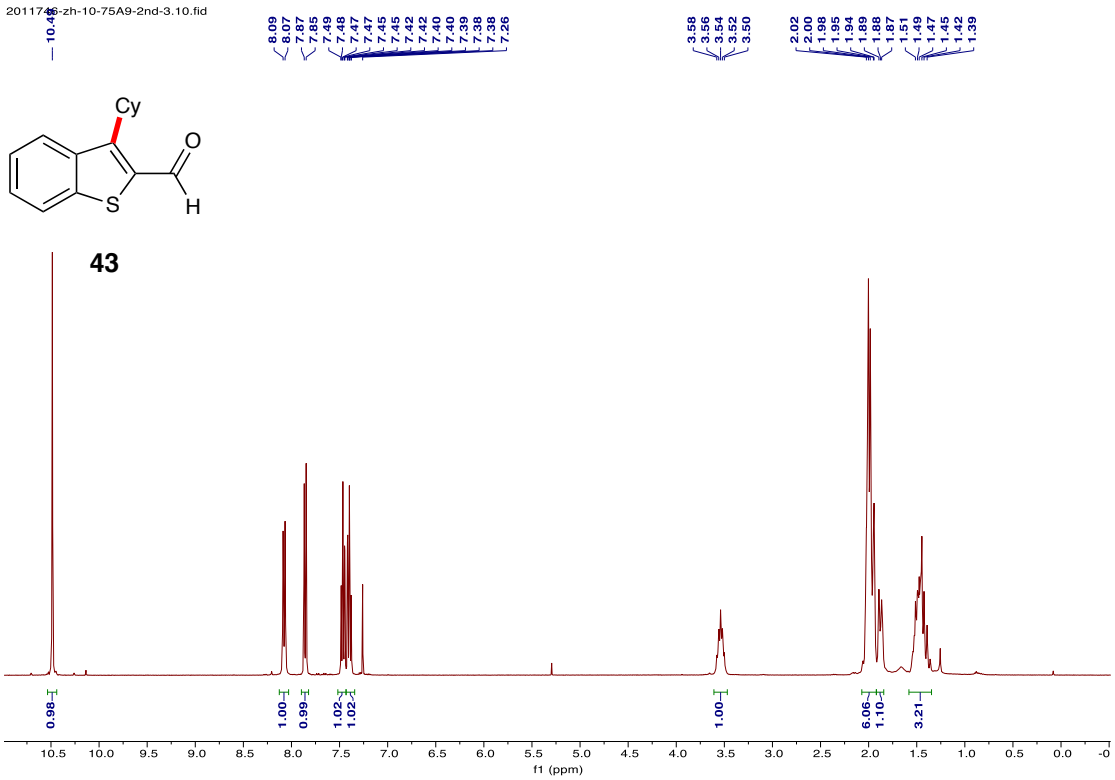


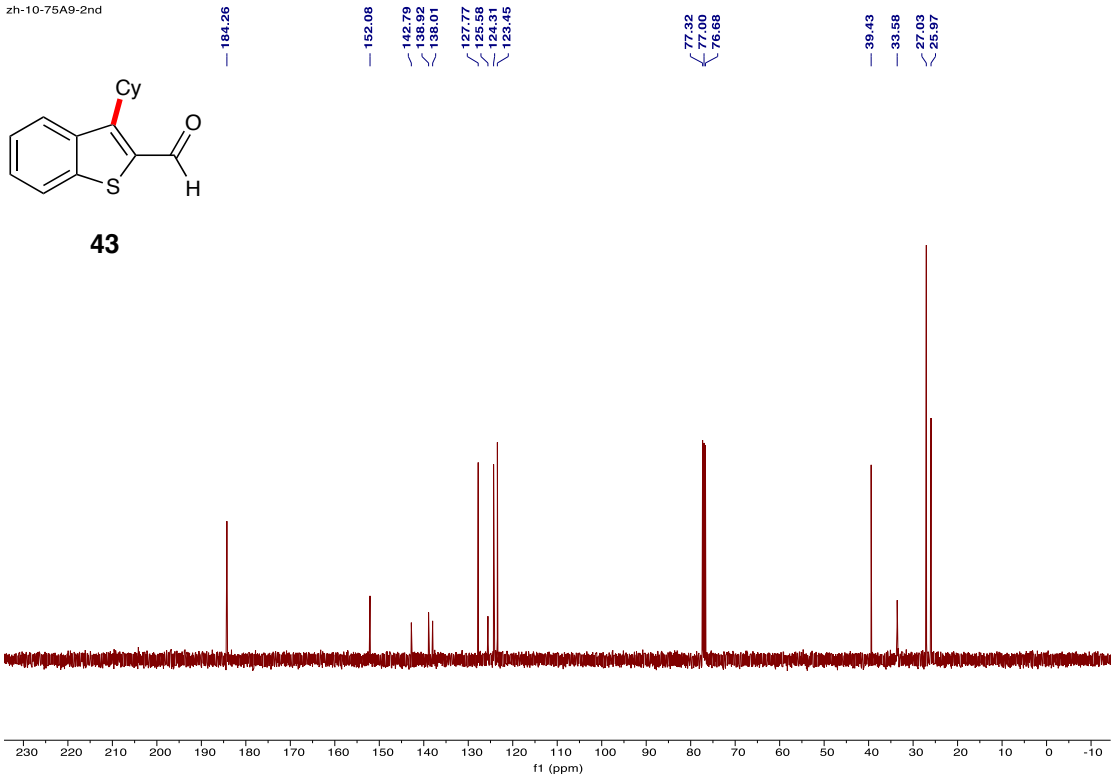
42

zh-10-75A25

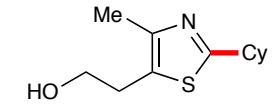


42


2011748-zh-10-75A9-2nd-3.10.fid
10.46
8.09
8.07
7.87
7.85
7.49
7.48
7.47
7.47
7.45
7.45
7.42
7.42
7.40
7.40
7.39
7.38
7.38
7.26
3.58
3.56
3.54
3.52
3.50
2.02
2.00
1.98
1.95
1.94
1.89
1.88
1.87
1.51
1.49
1.47
1.45
1.42
1.39
Cy
O
S
H
43
0.98
1.00
0.99
1.02
1.02
1.00
6.06
1.10
3.21
10.5 10.0 9.5 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 0.0 -0
f1 (ppm)

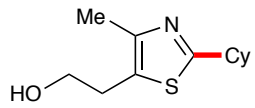

zh-10-75A9-2nd
184.26
152.08
142.79
138.92
138.01
127.77
125.58
124.31
123.45
77.32
77.00
76.68
39.43
33.58
27.03
25.97
Cy
O
S
H
43
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

2011746-zh-10-75A13-2nd.10.fid

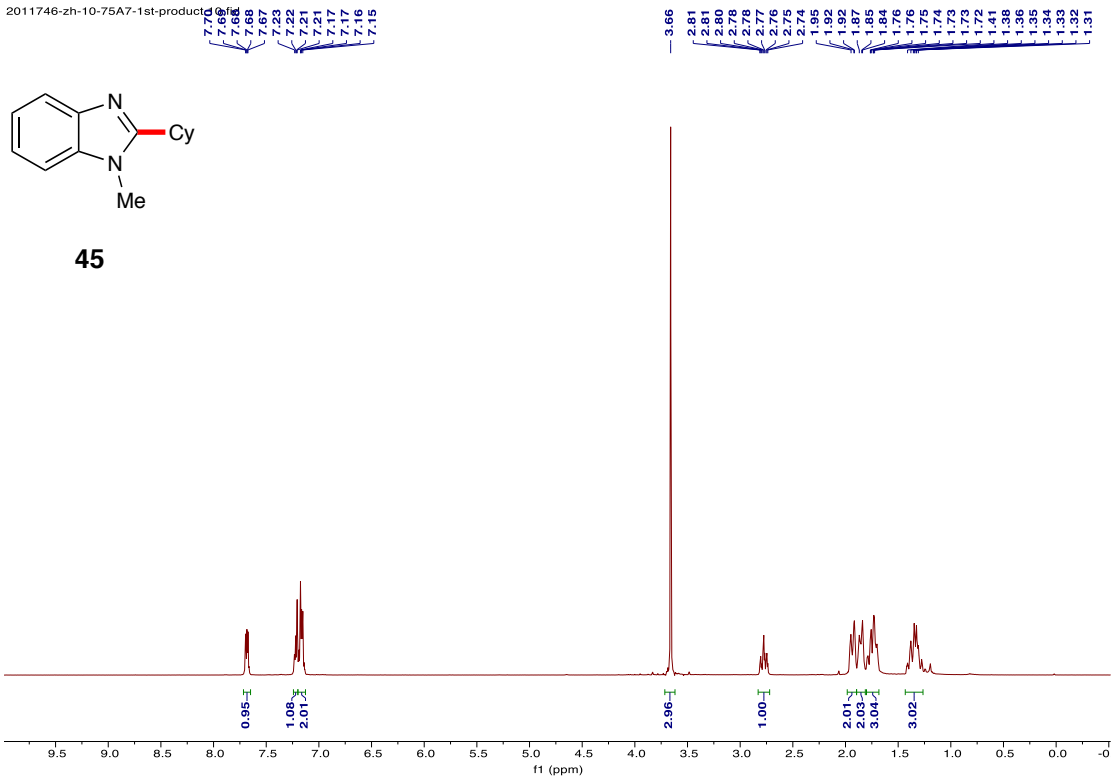


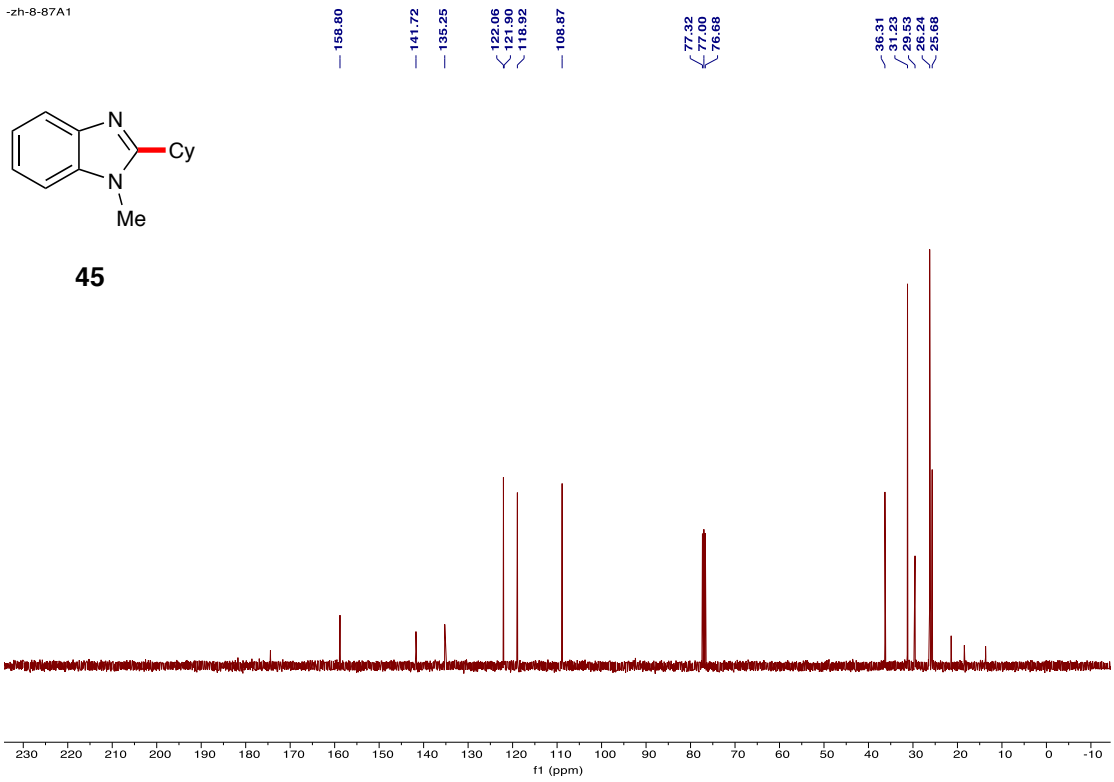
44

zh-10-75A13-2nd

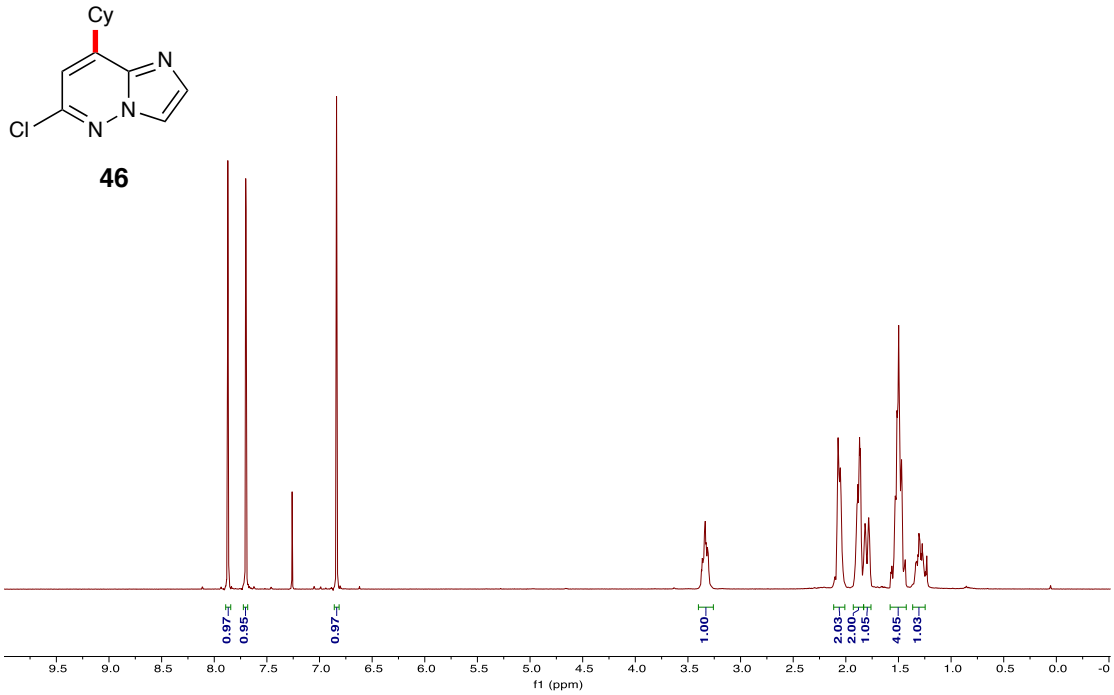


44


2011746-zh-10-75A7-1st-product-1H.fid
7.70
7.69
7.68
7.68
7.67
7.23
7.22
7.21
7.21
7.17
7.17
7.16
7.15
3.66
2.81
2.81
2.80
2.78
2.78
2.77
2.76
2.75
2.74
1.95
1.92
1.92
1.87
1.85
1.84
1.76
1.76
1.75
1.74
1.73
1.73
1.72
1.41
1.38
1.36
1.35
1.34
1.33
1.32
1.31
N
Cy
N
Me
45
0.95
1.08
2.01
2.96
1.00
2.01
2.03
3.04
3.02
f1 (ppm)

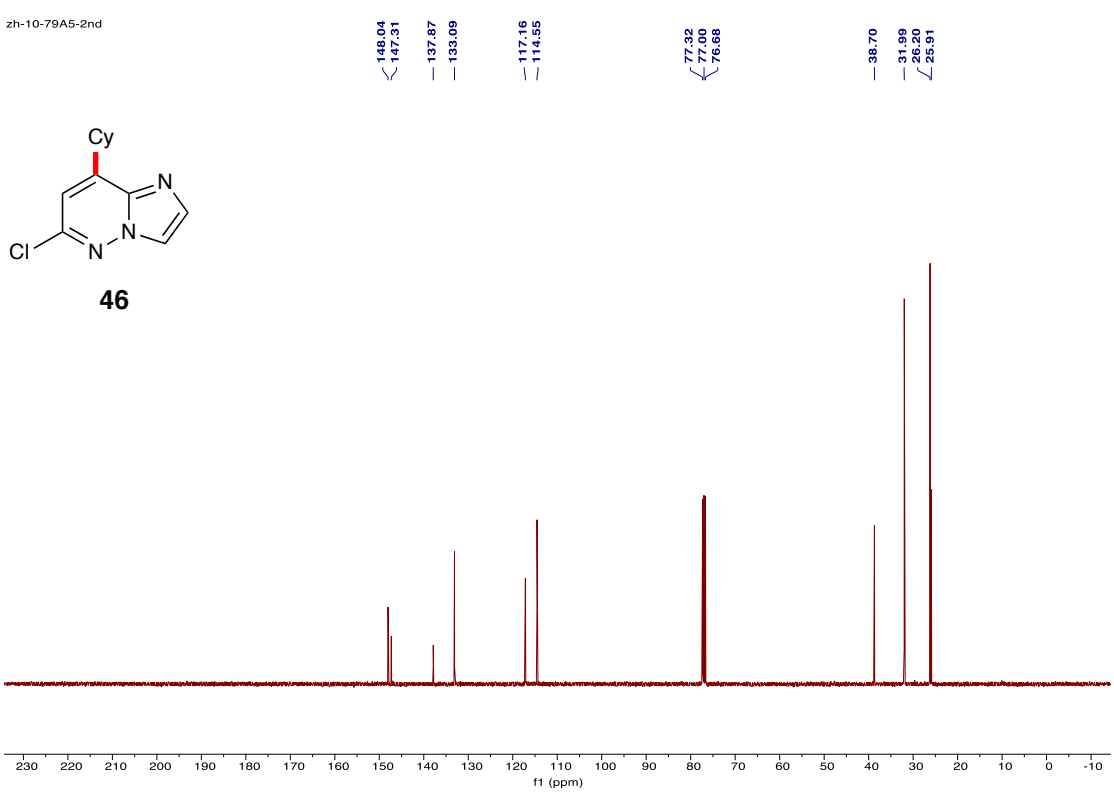

-zh-8-87A1
158.80
141.72
135.25
122.06
121.90
118.92
108.87
77.32
77.00
76.68
36.31
31.23
29.53
26.24
25.68
N
Cy
N
Me
45
f1 (ppm)

2011746-zh-10-79A5-2nd-1.10.fid

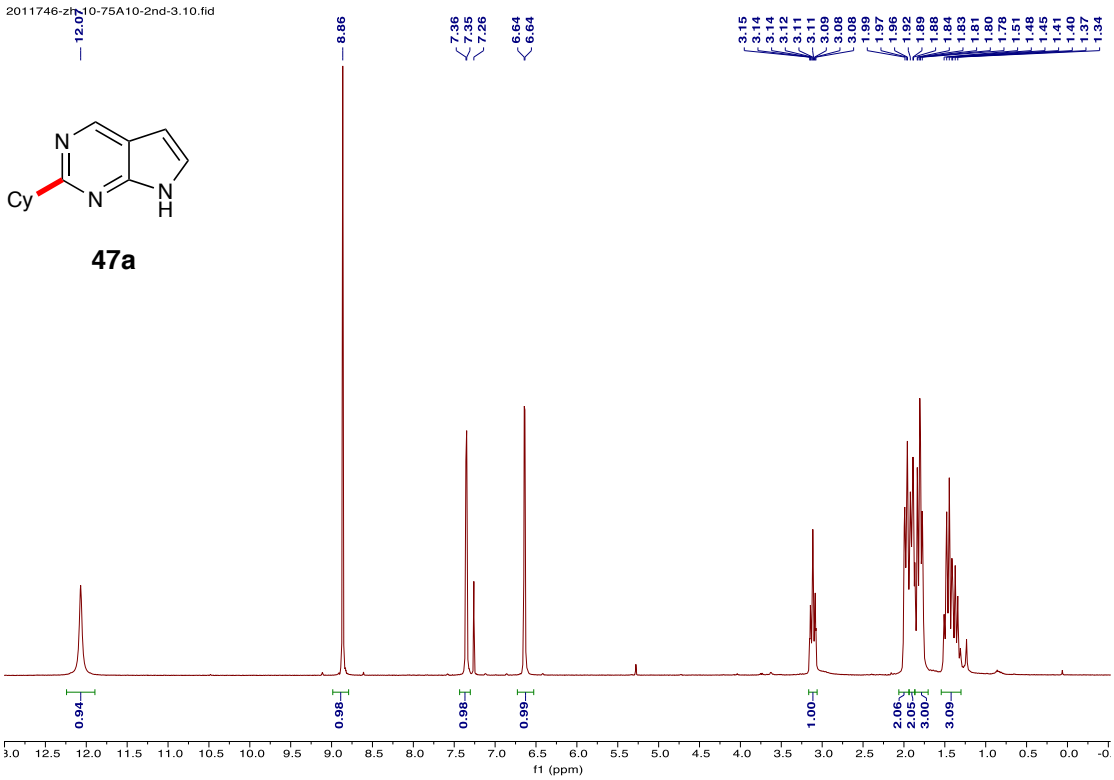


46

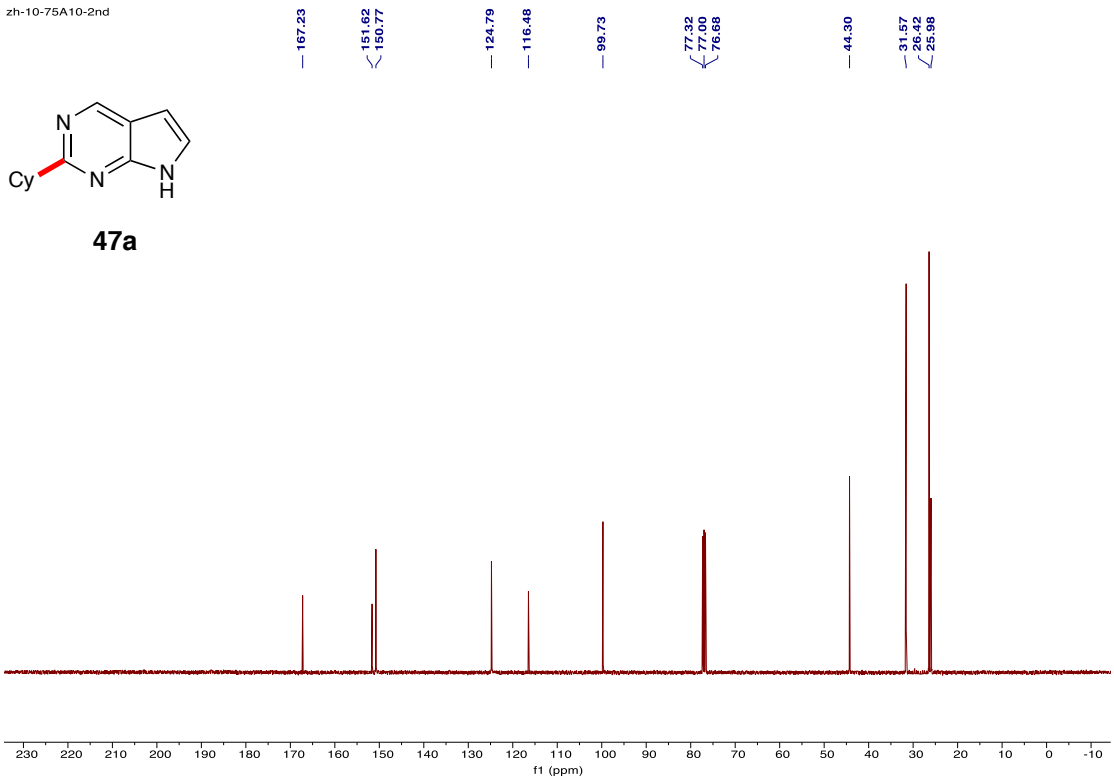
zh-10-79A5-2nd



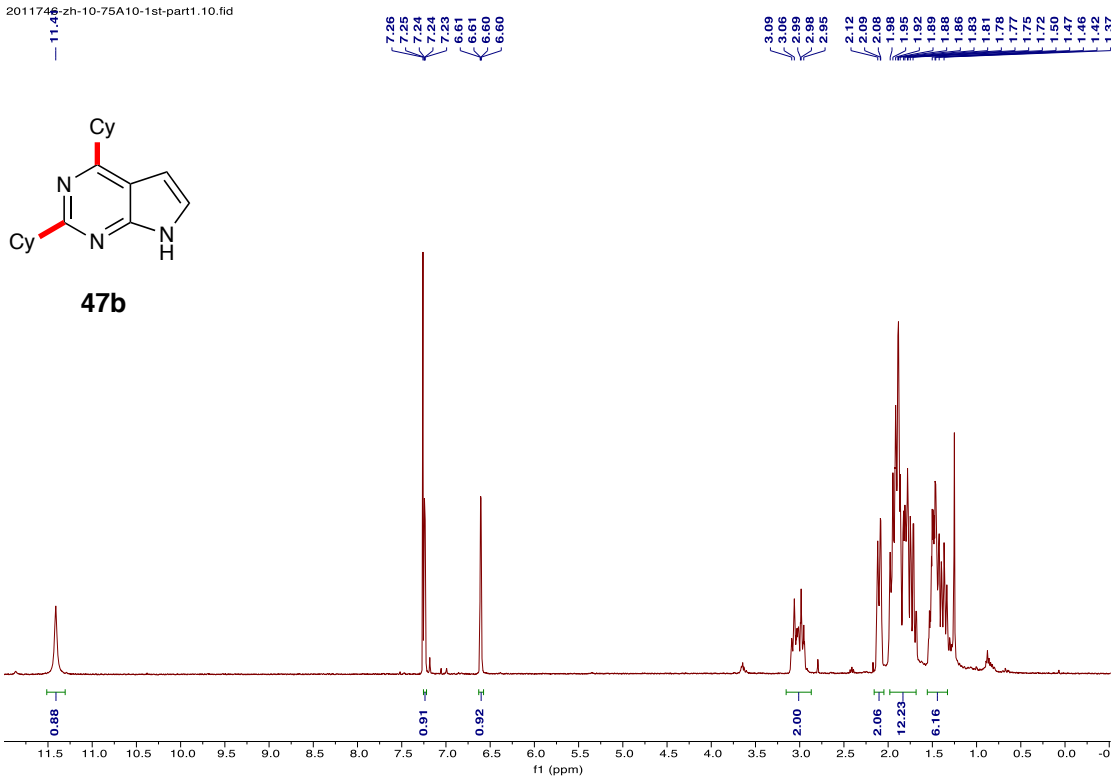
46

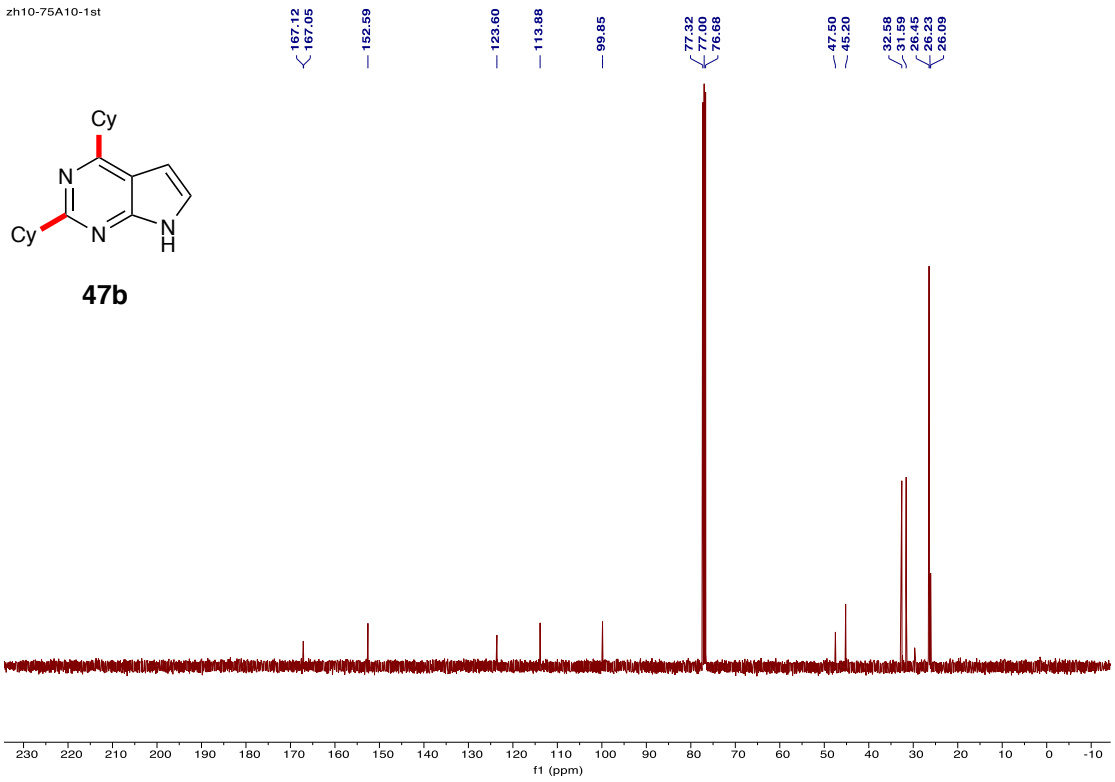

2011746-zh-10-75A10-2nd-3.10.fid
12.07
8.86
7.36
7.35
7.26
6.64
6.64
3.15
3.14
3.14
3.12
3.11
3.11
3.09
3.08
3.08
1.99
1.97
1.96
1.92
1.89
1.88
1.84
1.83
1.81
1.80
1.78
1.51
1.48
1.45
1.41
1.40
1.37
1.34
Cy
N
N
N
H
0.94
0.98
0.98
0.99
1.00
2.06
2.05
3.00
3.09
f1 (ppm)

47a

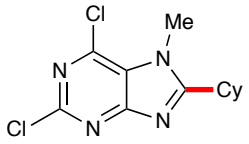

zh-10-75A10-2nd
167.23
151.62
150.77
124.79
116.48
99.73
77.32
77.00
76.68
44.30
31.57
26.42
25.98
Cy
N
N
N
H
f1 (ppm)

47a


2011746-zh-10-75A10-1st-part1.10.fid
11.46
7.26
7.25
7.24
7.24
7.23
6.61
6.61
6.60
6.60
3.09
3.06
2.99
2.98
2.95
2.12
2.09
2.08
1.98
1.95
1.92
1.89
1.88
1.86
1.83
1.81
1.78
1.77
1.75
1.72
1.50
1.47
1.46
1.42
Cy
N
Cy
N
N
H
47b
0.88
0.91
0.92
2.00
2.06
12.23
6.16
f1 (ppm)

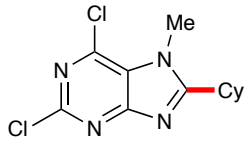

zh10-75A10-1st
167.12
167.05
152.59
123.60
113.88
99.85
77.32
77.00
76.68
47.50
45.20
32.58
31.59
26.45
26.23
26.09
Cy
N
Cy
N
N
H
47b
f1 (ppm)

2011746-zh-10-75A18-2.10.fid

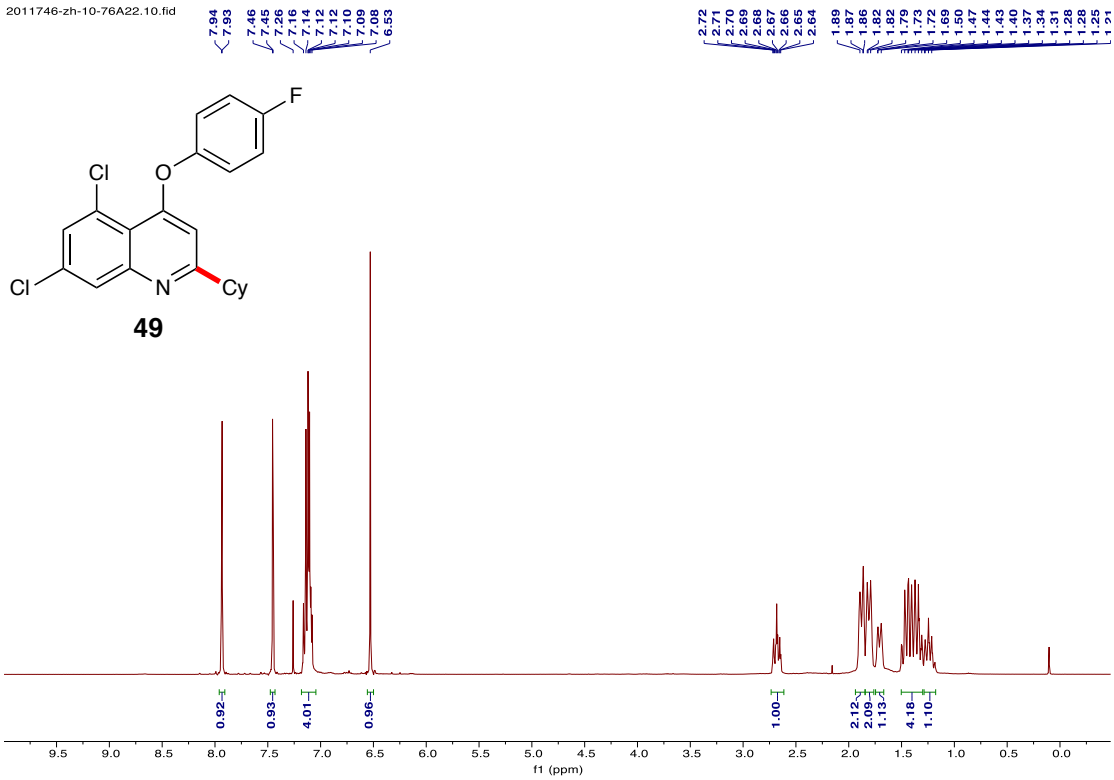


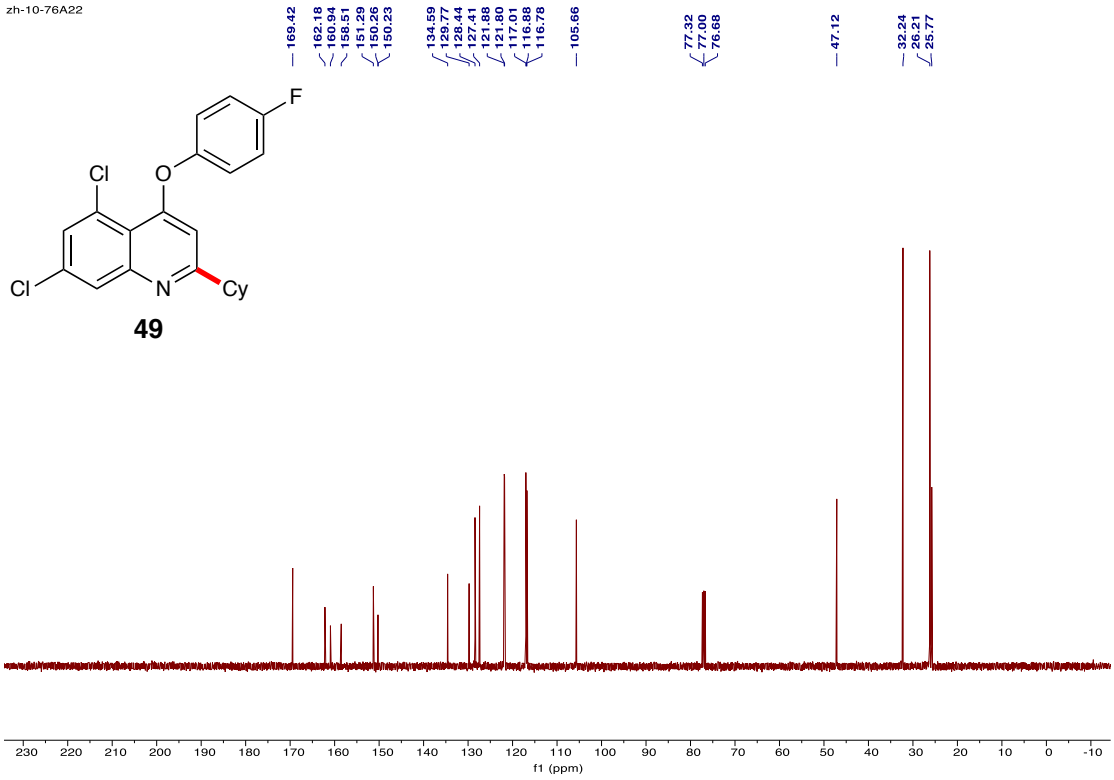
48

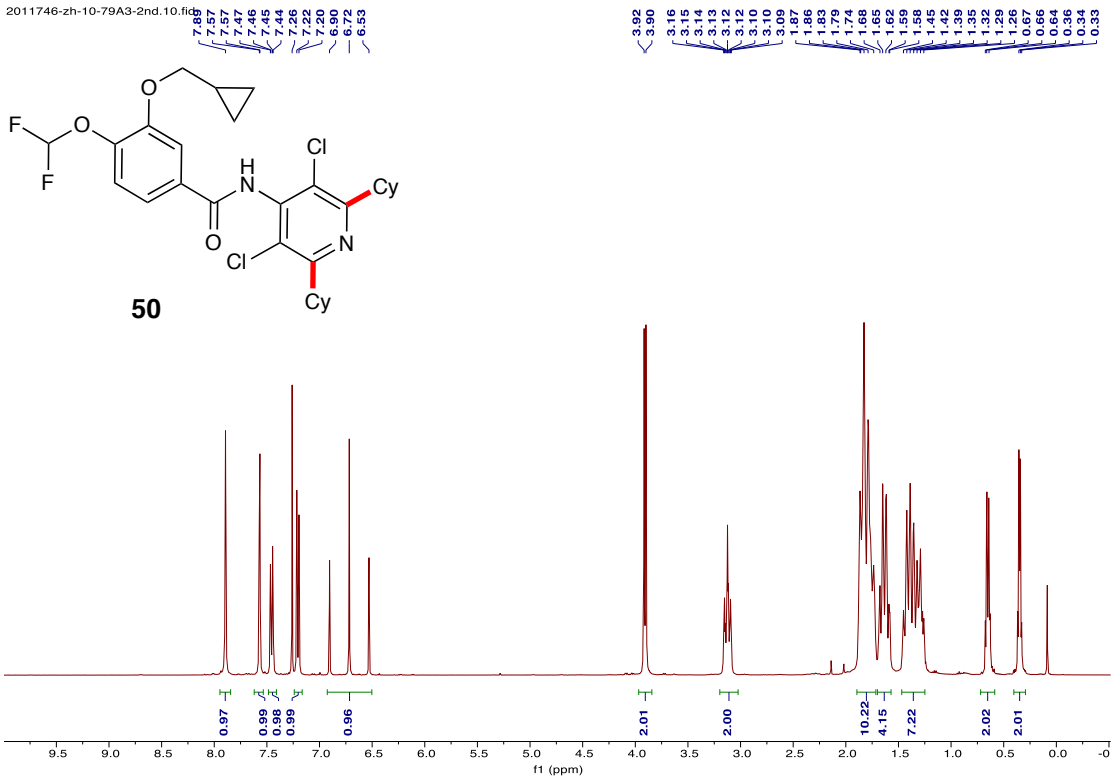
zh10-75A18

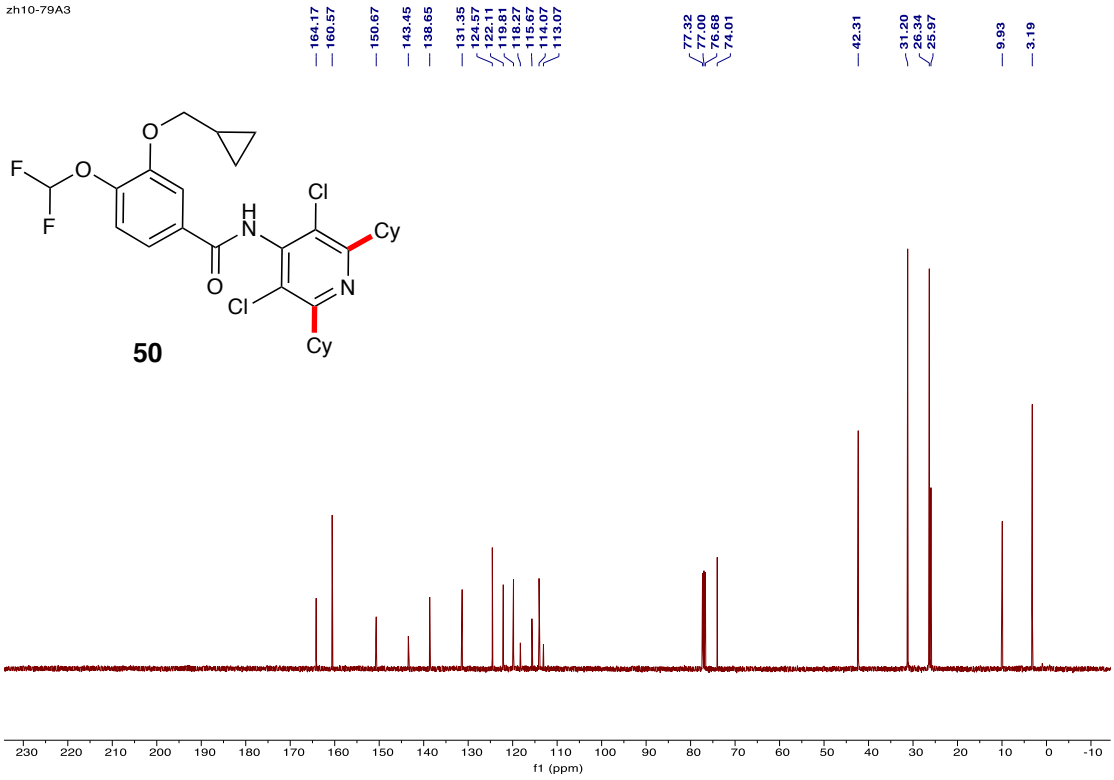


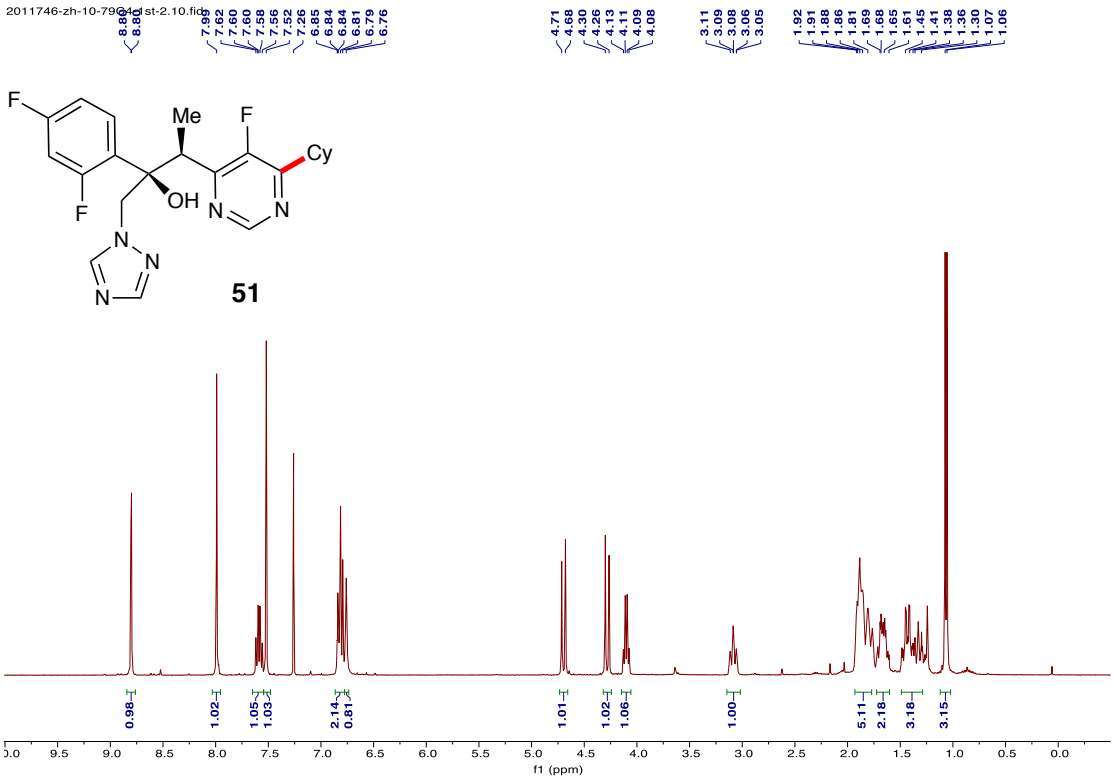
48

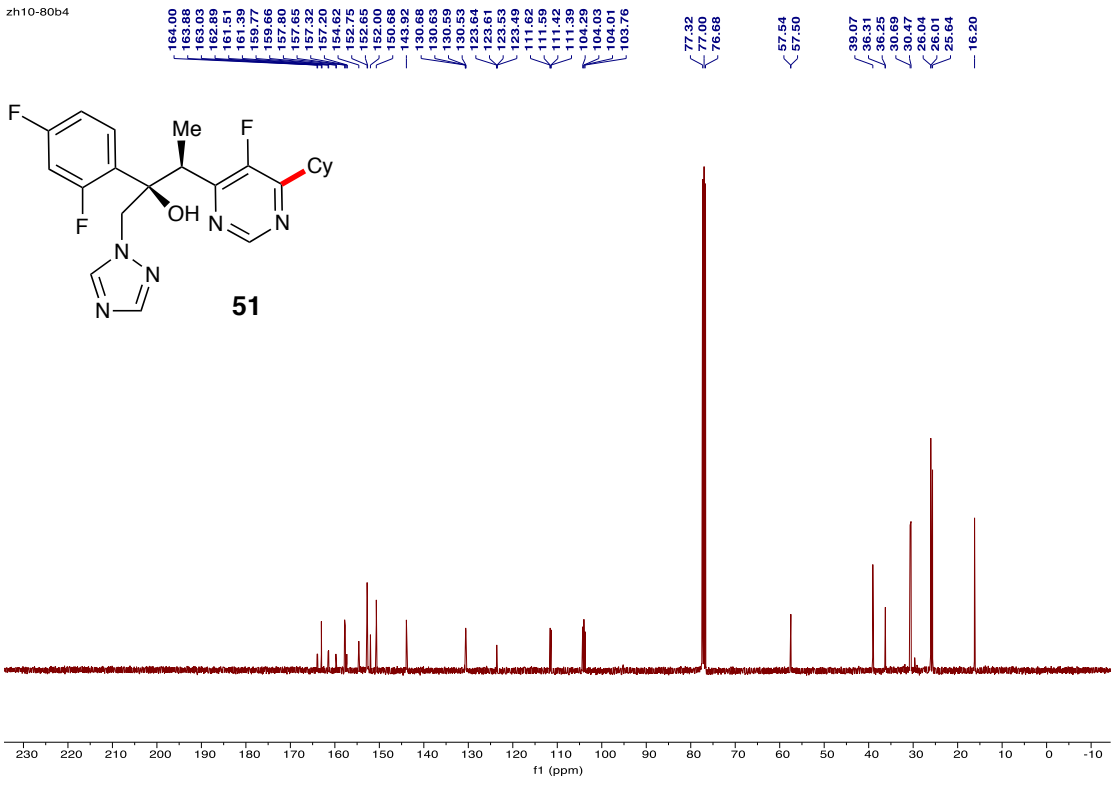

2011746-zh-10-76A22.10.fid
7.94
7.93
7.46
7.45
7.26
7.16
7.14
7.12
7.12
7.10
7.09
7.08
6.53
2.72
2.71
2.70
2.69
2.68
2.67
2.66
2.65
2.64
1.89
1.87
1.86
1.82
1.82
1.79
1.73
1.72
1.69
1.50
1.47
1.44
1.43
1.40
1.37
1.34
1.31
1.28
1.28
1.25
1.21
F
Cl
O
Cl
N
Cy
49
0.92
0.93
4.01
0.96
1.00
2.12
2.09
1.13
4.18
1.10
f1 (ppm)


zh-10-76A22
169.42
162.18
160.94
158.51
151.29
150.26
150.23
134.59
129.77
128.44
127.41
121.88
121.80
117.01
116.88
116.78
105.66
77.32
77.00
76.68
47.12
32.24
26.21
25.77
F
Cl
O
Cl
N
Cy
49
f1 (ppm)


2011746-zh-10-79A3-2nd.10.fid
7.59
7.57
7.57
7.47
7.46
7.45
7.44
7.26
7.22
7.20
6.90
6.72
6.53
3.92
3.90
3.16
3.15
3.14
3.13
3.12
3.12
3.10
3.10
3.09
1.87
1.86
1.83
1.79
1.74
1.68
1.65
1.62
1.59
1.58
1.45
1.42
1.39
1.35
1.32
1.29
1.26
0.67
0.66
0.64
0.36
0.34
0.33
O
F
O
F
H
N
Cl
Cy
O
Cl
N
Cy
50
0.97
0.99
0.98
0.99
0.96
2.01
2.00
10.22
4.15
7.22
2.02
2.01
f1 (ppm)


zh10-79A3
164.17
160.57
150.67
143.45
138.65
131.35
124.57
122.11
119.81
118.27
115.67
114.07
113.07
77.32
77.00
76.68
74.01
42.31
31.20
26.34
25.97
9.93
3.19
O
F
O
F
H
N
Cl
Cy
O
Cl
N
Cy
50
f1 (ppm)


2011746-zh-10-79041st-2.10.fid
8.80
8.80
7.99
7.62
7.60
7.60
7.58
7.56
7.52
7.26
6.85
6.84
6.84
6.81
6.79
6.76
4.71
4.68
4.30
4.26
4.13
4.11
4.09
4.08
3.11
3.09
3.08
3.06
3.05
1.92
1.91
1.88
1.86
1.81
1.69
1.68
1.65
1.61
1.45
1.41
1.38
1.36
1.30
1.07
1.06
F
Me
F
Cy
OH
F
N
N
N
N
N
51
0.98
1.02
1.05
1.03
2.14
0.81
1.01
1.02
1.06
1.00
5.11
2.18
3.18
3.15
f1 (ppm)


zh10-80b4
164.00
163.88
163.03
162.89
161.51
161.39
159.77
159.66
157.65
157.60
157.32
157.20
154.62
152.75
152.65
152.00
150.68
143.92
130.68
130.63
130.59
130.53
123.64
123.61
123.53
123.49
111.62
111.59
111.42
111.39
104.29
104.03
104.01
103.76
77.32
77.00
76.68
57.54
57.50
39.07
36.31
36.25
30.69
30.47
26.04
26.01
25.64
16.20
F
Me
F
Cy
OH
F
N
N
N
N
N
51
f1 (ppm)