Electronic Supplementary Information

Pd-catalyzed C(sp^2)–H carbonylation of 2-benzylpyridines for the synthesis of pyridoisoquinolinones

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I. General Information

All reagents were purchased without further purification unless otherwise noted. Reactions were monitored using thin-layer chromatography (TLC) on commercial silica gel plates (GF254). Visualization of the developed plates was performed under UV light (254 nm). Flash column chromatography was performed on silica gel (200-300 mesh). ^{1}H and ^{13}C NMR spectra were recorded on a 400 or 500 MHz spectrometer. Chemical shifts (δ) were reported in ppm referenced to an internal tetramethylsilane standard or the DMSO-d_6 residual peak (δ 2.50) for ^{1}H NMR. Chemical shifts of ^{13}C NMR were reported relative to DMSO-d_6 (δ 39.5). The following abbreviations were used to describe peak splitting patterns when appropriate: br = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. Coupling constants, *J*, were reported in Hertz unit (Hz). High resolution mass spectra (HRMS) were obtained on an ESI-LC-MS/MS spectrometer. Melting points were measured on a Mel-Temp apparatus and are uncorrected. IR was recorded on a Bruker Tensor 27 FT-IR spectrometer.

II. General Procedures

(a) General Procedure for the Synthesis of 2

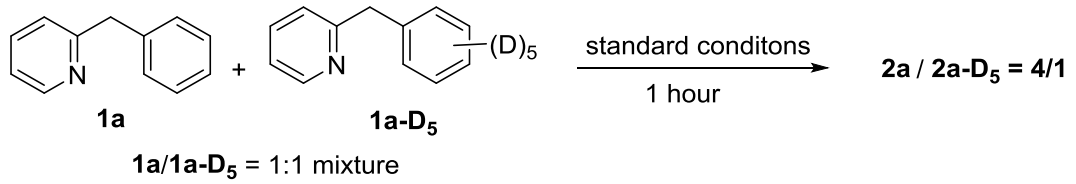
Substrates **1** were prepared according to literature reported procedures.[1a-c] To a Schlenk tube were added 2-benzyl heterocycle **1** (0.2 mmol)**,** $Pd(OAc)_2$ (0.02 mmol), and $Cu(TFA)_2xH_2O$ (0.24 mmol). Then, the tube was vacuumed and refilled with CO for 3 times followed by injection a solution of PivOH (0.2 mmol) in 2 mL of toluene. The reaction mixture was stirred at 110 ^{o}C for 6 h under a balloon pressure of CO. Finally, the reaction was cooled down to room temperature before addition of saturated aqueous NaCl (10 mL), NH_4OH (1 mL) and EtOAc (10 mL) to the reaction mixture. The aqueous phase was further extracted with EtOAc (3 × 10 mL). The combined organic layers were dried over anhydrous Na_2SO_4 and concentrated. The residue was purified by flash chromatography to provide the desired product **2**.

(b) Isotope labelling experiments

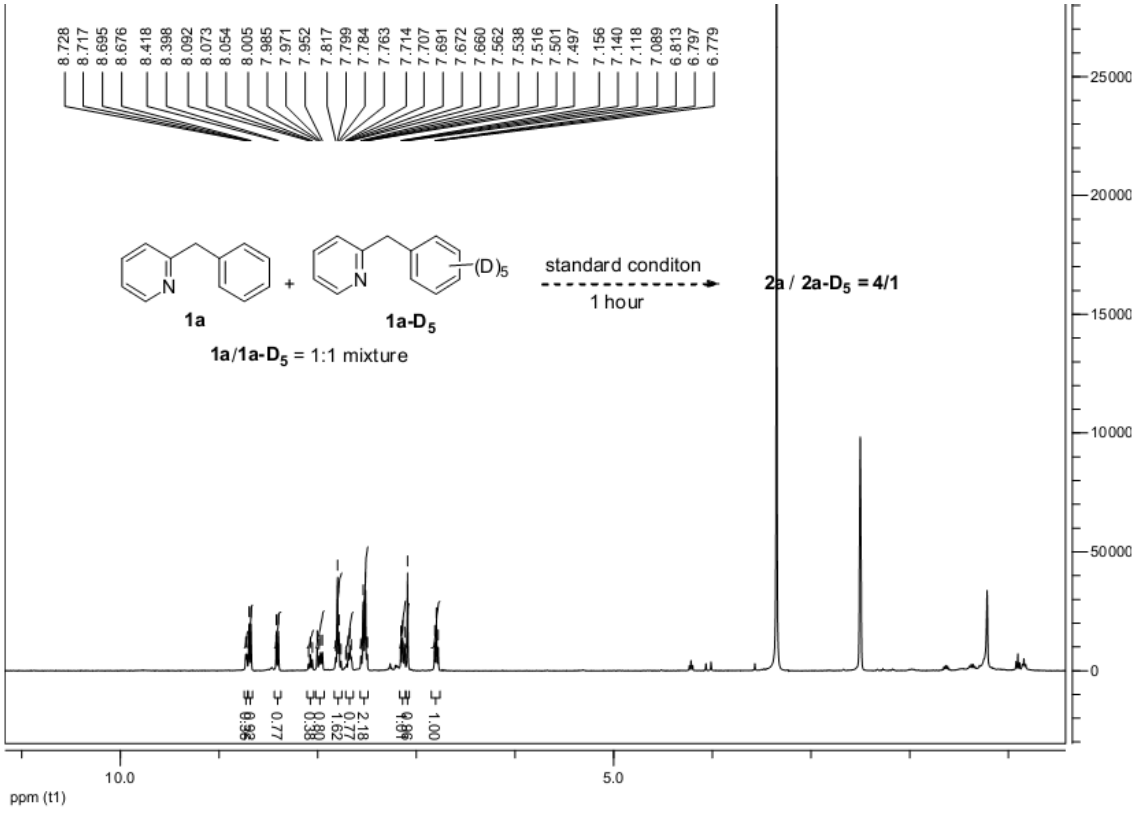
Deuterated substrate **1a-D_5** was synthesized according to the literature reported method[1a] using potassium 2-(pyridin-2-yl)acetate and pentadeuterated bromobenzene as substrates. **1a-D_5** was obtained in 80% yield as a liquid. ^{1}H NMR (400 MHz,

$CDCl_3$): 8.55 (d, *J* = 4.8 Hz, 1H); 7.57 (t, *J* = 7.6 Hz, 1H), 7.11 (dd, *J* = 7.6, 4.8 Hz, 2H); 4.16 (s, 2H).

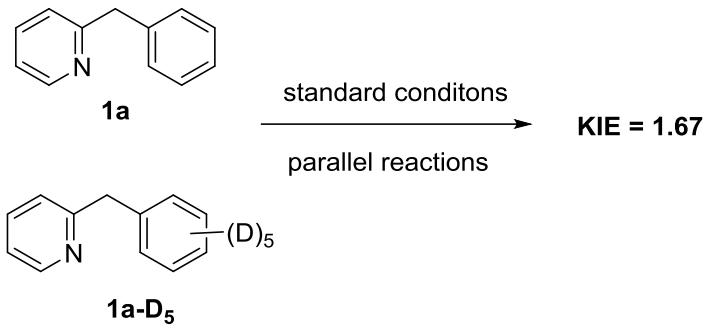
Intermolecular competition reaction of a mixture of **1a** and **1a-D$_5$**:



A mixture of **1a** (0.1 mmol), **1a-D$_5$** (0.1 mmol), $Pd(OAc)_2$ (0.02 mmol), PivOH (0.2 mmol), $Cu(TFA)_2xH_2O$ (0.24 mmol) in Toluene (2 mL) was stirred at 110 ℃ under balloon pressure of CO for 1.0 h. The mixture was diluted with EtOAc (20 mL), washed with saturated aqueous NaCl (10 mL), NH_4·OH (1 mL) and EtOAc (10 mL). The organic layer was dried over anhydrous Na_2SO_4. The concentrated residue was purified by column chromatography to give a mixture of **2a** and **2a-D$_4$** in a ratio of 4:1 as determined by ^{1}H NMR in a total yield of 38%.



Parallel competition reactions of **1a** and **1a-D$_5$**:



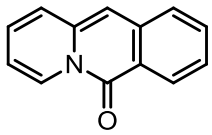
Two identical reactions were set side by side with **1a** and **1a-D_5** as substrates respectively. A tube was charged with **1a** (0.2 mmol), $Pd(OAc)_2$ (0.02 mmol), PivOH (0.2 mmol), and $Cu(TFA)_2xH_2O$ (0.24 mmol). Toluene (2.0 mL) was added to the mixture and it was stirred at 110 ℃ under balloon pressure of CO. Take 0.1 mL of reaction solution from the tube in 10, 20, 30, 40 and 50 min, respectively. Each of the aliquot was treated by following the procedure mentioned above. The crude reaction mixture was analyzed by ^{1}H NMR. In a parallel experiment, the same reaction was performed using **1a-D_5** (0.1 mmol) as a substrate with the identical operation. The yields of **2a** and **2a-D_4** of the 10 samples were plotted against reaction time. The ratio of product formation was determined to be 1.67 by comparing the slopes.

Time (x min)	10	20	30	40	50	Equation (C=Ax+B)	KIE=(A_y/A_z)
Yield of **2a** (y %)	2	4	7	10	13	y = 0.25x-0.4	1.67
Yield of 2**a-D_4** (z %)	1	2	4	6	10	z = 0.15x-0.4	

Figure S1: the relationship between reaction time and yield.

III. Analytical Data

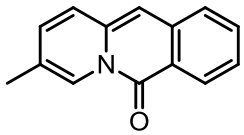
6*H*-pyrido[1,2-*b*]isoquinolin-6-one (**2a**)



Yellow solid, 34 mg, 87% yield, eluent: petroleum ether/ethyl acetate 15/1. ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.68 (dd, *J* = 7.5, 0.5 Hz, 1H), 8.41 (d, *J* = 8.5 Hz, 1H),7.81-7.77 (m, 2H), 7.53-7.50 (m, 2H), 7.15-7.12 (m, 1H), 7.09 (s, 1H), 6.81-6.78

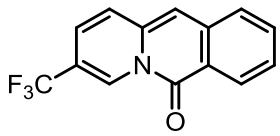
(m, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) 158.7, 137.6, 136.6, 133.0, 127.9, 127.1, 126.4, 126.3, 125.8, 125.5, 119.6, 113.1, 100.9; FT-IR (film) 3435, 3115, 3049, 2921, 1669, 1634, 1614, 1573, 1528, 1469, 1351, 1251, 1157, 1138, 800, 724, 670, 556 cm^{-1}; HRMS (ESI) calcd for $C_{13}H_9NO$ $[M+H]^+$ 196.0757, found 196.0758; mp 166-169 ^{o}C.

3-methyl-6*H*-pyrido[1,2-*b*]isoquinolin-6-one (**2b**)



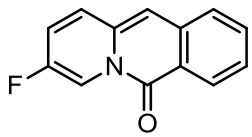
Yellow solid, 36 mg, 86% yield, eluent: petroleum ether/ethyl acetate 15/1.^{1}H NMR (400 MHz, DMSO-d_6) δ 8.53 (s, 1H), 8.40 (d, *J* = 8.4 Hz, 1H), 7.80-7.74 (m, 2H), 7.51-7.48 (m, 2H), 7.08 (s, 1H), 7.06-7.03 (m, 1H), 2.25 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d_6) 158.3, 136.6, 136.3, 132.6, 130.3, 127.8, 126.3, 125.9, 125.4, 122.3, 122.2, 119.6, 100.7, 18.5; FT-IR (film) 3425, 3101, 3056, 2918, 1661, 1635, 1611, 1576, 1533, 1471, 1460, 1282, 1244, 1038, 821, 745, 704, 673, 556 cm^{-1}; HRMS (ESI) calcd for $C_{14}H_{11}NO$ [M+H]+ 210.0913, found 210.0915; mp 93-96 ^{o}C.

3-(trifluoromethyl)-6H-pyrido[1,2-*b*]isoquinolin-6-one (**2c**)



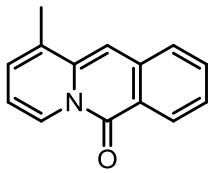
Yellow solid, 38 mg, 72% yield, eluent: petroleum ether/ethyl acetate 12/1. ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.92 (s, 1H), 8.41 (dd, *J* = 8.0, 3.0 Hz, 1H), 7.86 (t, *J* =8.0 Hz, 2H), 7.65-7.63 (m, 2H), 7.61-7.60 (m, 2H), 7.21-7.17 (m, 2H); ^{13}C NMR (125 MHz, DMSO-d_6) 159.0, 136.7, 136.3, 133.9, 128.4, 128.2, 126.9, 126.8, 125.9 (q, *J* = 6.6 Hz), 125.0, 120.7, 120.4, 114.8 (q, *J* = 34.9 Hz), 102.7; FT-IR (film) 3441, 3105, 3064, 2919, 1670, 1645, 1613, 1587, 1540, 1471, 1334, 1153, 1123, 1056, 969, 896, 753, 685, 670, 612 cm^{-1}; HRMS (ESI) calcd for $C_{14}H_8F_3NO$ [M+H]+ 264.0631, found 264.0633; mp 189-191 ^{o}C.

3-fluoro-6*H*-pyrido[1,2-*b*]isoquinolin-6-one (**2d**)



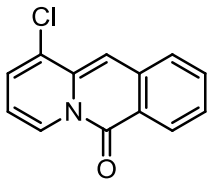
Yellow solid, 31 mg, 73% yield, eluent: petroleum ether/ethyl acetate 15/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.74 (dd, *J* = 6.0, 2.0 Hz, 1H), 8.60 (d, *J* = 4.4 Hz, 1H), 7.73-7.68 (m, 2H), 7.56-7.52 (m, 1H), 7.33-7.29 (m, 1H), 6.97-6.92 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) 135.8, 135.3, 132.4, 128.2, 127.8 (d, *J* = 7.3 Hz), 125.9 (d, *J* = 5.6 Hz), 120.4 (d, *J* = 28.3 Hz), 120.0, 111.3 (d, *J* = 42.8 Hz), 102.5; FT-IR (film) 3424, 3112, 3074, 2925, 2853, 1652, 1612, 1575, 1526, 1473, 1457, 1426, 1220, 1154, 1127, 1109, 815, 803, 743, 697, 670, 561 cm^{-1}; HRMS (ESI) calcd for $C_{13}H_8FNO$ $[M+H]^+$ 214.0663, found 214.0664; mp 159-162 ^{o}C.

1-methyl-6*H*-pyrido[1,2-*b*]isoquinolin-6-one (**2e**)



Yellow solid, 26 mg, 62% yield, eluent: petroleum ether/ethyl acetate 15/1. ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.66 (d, *J* = 7.5 Hz, 1H), 8.38 (d, *J* = 8.0 Hz, 1H), 7.86 (d, *J* = 7.5 Hz, 1H), 7.79-7.76 (m, 1H), 7.53-7.49 (m, 1H), 7.05 (d, *J* = 4.0 Hz, 2H), 6.74 (t, *J* = 7.0 Hz, 1H); 2.40 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d_6) 159.1, 137.8, 136.3, 132.9, 132.2, 127.8, 126.8, 126.6, 125.7, 124.5, 119.4, 112.5, 98.1, 19.2; FT-IR (film) 3423, 3118, 2976, 2919, 2851, 1655, 1637, 1614, 1569, 1472, 1454, 1439, 1372, 1271, 1249, 1166, 1133, 1070, 801, 740, 707, 674, 559 cm^{-1}; HRMS (ESI) calcd for $C_{14}H_{11}NO$ [M+H]+ 210.0913, found 210.0913; mp 119-121 ^{o}C.

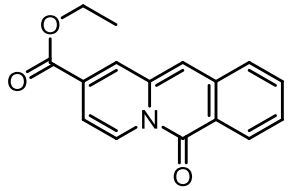
1-chloro-6*H*-pyrido[1,2-*b*]isoquinolin-6-one (**2f**)



Yellow solid, 36 mg, 79% yield, eluent: petroleum ether/ethyl acetate 10/1. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.70 (d, *J* = 7.6 Hz, 1H), 8.41 (d, *J* = 8.0 Hz, 1H), 7.96 (d, *J* = 8.0 Hz, 1H), 7.87-7.83 (m, 1H), 7.60 (t, *J* = 6.8 Hz, 1H), 7.45 (d, *J* = 7.2 Hz, 1H), 7.36 (s, 1H), 6.76 (t, *J* = 7.2 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) 158.9, 136.1, 134.7, 133.5, 128.1, 128.0, 127.3, 127.3, 126.8, 125.9, 119.9, 111.3, 99.0; FT-IR (film) 3090, 2923, 2852, 1688, 1631, 1609, 1582, 1550, 1522, 1440, 1405, 1164, 1148, 805, 742, 696, 670, 554 cm^{-1}; HRMS (ESI) calcd for $C_{13}H_8ClNO$ [M+H]+ 230.0367, found

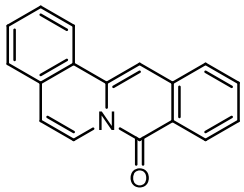
230.0365; mp 141-144 ^{o}C.

ethyl 6-oxo-6*H*-pyrido[1,2-*b*]isoquinoline-2-carboxylate (**2g**)



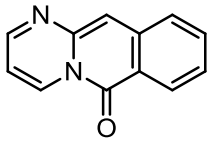
Yellow solid, 50 mg, 93% yield, eluent: petroleum ether/ethyl acetate 8/1. ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.64 (d, *J* = 8.0 Hz, 1H), 8.43 (d, *J* = 8.0 Hz, 1H), 8.13 (s, 1H), 7.84 (d, *J* = 3.5 Hz, 2H), 7.63-7.60 (m, 1H), 7.41 (s, 1H), 6.96 (d, *J* = 8.0 Hz, 1H), 4.34 (q, *J* = 7.0 Hz, 2H), 1.34 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (125 MHz, DMSO-d_6) 164.2, 158.5, 136.1, 136.0, 133.4, 130.5, 128.0, 127.4, 127.1, 127.1, 126.7, 121.2, 110.0, 105.7, 61.9, 14.6; FT-IR (film) 1711, 1651, 1637, 1614, 1578, 1469, 1437, 1293, 1241, 1097, 1023, 740, 699, 666, 572 cm^{-1}; HRMS (ESI) calcd for $C_{16}H_{13}NO_3$ [M+H]+ 268.0968, found 268.0969; mp 132-135 ^{o}C.

8*H*-isoquinolino[3,2-*a*]isoquinolin-8-one (**2h**)



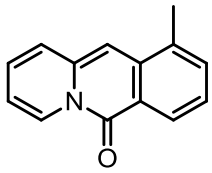
Yellow solid, 30 mg, 57% yield, eluent: petroleum ether/ethyl acetate 10/1. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.54 (d, *J* = 8.0 Hz, 2H), 8.40 (d, *J* = 8.4 Hz, 1H), 8.00 (s, 1H), 7.90 (d, *J* = 8.0 Hz, 1H), 7.84 (t, *J* = 7.2 Hz, 1H), 7.69-7.56 (m, 4H), 7.02 (d, *J* = 8.0 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) 158.9, 135.8, 134.9, 133.0, 130.3, 129.5, 128.6, 127.5, 127.2, 126.8, 126.7, 126.2, 124.0, 122.6, 120.9, 112.6, 98.7; FT-IR (film) 3442, 3122, 3058, 3020, 1657, 1614, 1587, 1548, 1458, 1445, 1328, 1285, 1269, 1136, 1103, 1085, 1025, 975, 761, 742, 686, 529 cm^{-1}; HRMS (ESI) calcd for $C_{17}H_{11}NO$ [M+H]+ 246.0913, found 246.0916; mp 149-152 ^{o}C.

6*H*-pyrimido[1,2-*b*]isoquinolin-6-one (**2i**)



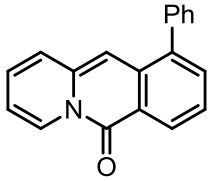
Orange solid, 24 mg, 61% yield, eluent: petroleum ether/ethyl acetate 10/1. ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.91 (dd, *J* = 7.5, 1.0 Hz, 1H), 8.45 (dd, *J* = 3.0, 2.0 Hz, 1H), 8.40 (d, *J* = 8.0 Hz, 1H), 7.90 (d, *J* = 8.0 Hz, 1H), 7.89-7.82 (m, 1H), 7.58-7.55 (m, 1H), 7.15 (s, 1H), 6.80 (dd, *J* = 7.5, 3.5 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) 158.7, 155.5, 142.9, 137.7, 134.0, 133.6, 128.0, 127.0, 126.2, 119.9, 109.1, 101.2; FT-IR (film) 3430, 1676, 1629, 1614, 1584, 1553, 1470, 1445, 1404, 1203, 936, 808, 747, 715, 677, 552 cm^{-1}; HRMS (ESI) calcd for $C_{12}H_8N_2O$ [M+H]+ 197.0709, found 197.0708; mp 188-191 ^{o}C.

10-methyl-6*H*-pyrido[1,2-*b*]isoquinolin-6-one (**2j**)



Yellow solid, 40 mg, 81% yield, eluent: petroleum ether/ethyl acetate 15/1. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.68 (d, *J* = 7.6 Hz, 1H), 8.28 (d, *J* = 8.4 Hz, 1H), 7.64-7.59 (m, 2H), 7.40 (dd, *J* = 8.0, 7.6 Hz, 1H), 7.17-7.13 (m, 2H), 6.80 (t, *J* = 6.4 Hz, 1H), 2.60 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d_6) 158.9, 137.4, 135.7, 133.2, 133.1, 127.1, 126.7, 125.9, 125.7, 125.1, 119.7, 113.3, 97.9, 19.3; FT-IR (film) 3424, 3113, 2967, 1923, 1659, 1631, 1606, 1583, 1525, 1467, 1456, 1433, 1387, 1377, 1279, 1212, 1212, 1212, 1163, 1138, 1007, 800, 732, 673, 561 cm^{-1}; HRMS (ESI) calcd for $C_{14}H_{11}NO$ [M+H]$^{+}$ 210.0913, found 214.0913; mp 153-156 ^{o}C.

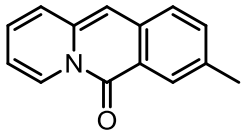
10-phenyl-6*H*-pyrido[1,2-*b*]isoquinolin-6-one (**2k**)



Yellow solid, 48 mg, 88% yield, eluent: petroleum ether/ethyl acetate 10/1. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.69 (d, *J* = 0.8 Hz, 1H), 8.48 (d, *J* = 8.0 Hz, 1H), 7.71 (dd, *J* = 7.2, 1.2 Hz, 1H), 7.60-7.47 (m, 7H), 7.11-7.08 (m, 1H), 6.88 (s, 1H), 6.83-6.79 (m, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) 158.8, 139.8, 138.3, 137.6, 134.4, 133.6, 133.0, 129.1, 128.1, 127.6, 127.2, 126.8, 125.8, 125.2, 120.2, 113.5, 98.6; FT-IR (film) 3424, 3115, 3033, 2919, 1947, 1658, 1635, 1595, 1574, 1432, 1420, 1388, 1339, 1181, 1077,

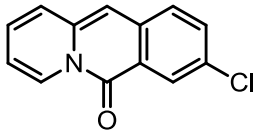
889, 847, 761, 737, 700, 681, 601, 563 cm^{-1}; HRMS (ESI) calcd for $C_{19}H_{13}NO$ $[M+H]^+$ 272.1070, found 272.1093; mp 151-153 ^oC.

8-methyl-6*H*-pyrido[1,2-*b*]isoquinolin-6-one (**2l**)



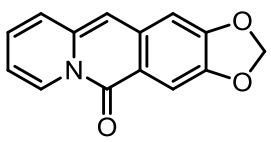
Yellow solid, 24 mg, 57% yield, eluent: petroleum ether/ethyl acetate 15/1. ^1H NMR (400 MHz, DMSO-d_6) δ 8.67 (d, *J* = 7.6 Hz, 1H), 8.21 (s, 1H), 7.73 (d, *J* = 8.4 Hz, 1H), 7.64 (dd, *J* = 8.4, 1.6 Hz, 1H), 7.50 (d, *J* = 8.8 Hz, 1H), 7.11-7.06 (m, 2H), 6.78-6.75 (m, 1H), 2.5 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d_6) 136.9, 135.2, 134.8, 134.5, 126.9, 126.5, 126.4, 126.3, 125.7, 113.0, 100.9, 21.7; FT-IR (film) 3421, 2922, 2853, 1661, 1636, 1581, 1518, 1490, 1382, 1079, 940, 822, 787, 738, 561, 466 cm^{-1}; HRMS (ESI) calcd for $C_{14}H_{11}NO$ $[M+H]^+$ 210.0913, found 214.0913; mp 157-161 ^oC

8-chloro-6*H*-pyrido[1,2-*b*]isoquinolin-6-one (**2m**)



Yellow solid, 18 mg, 39% yield, eluent: petroleum ether/ethyl acetate 10/1. ^1H NMR (400 MHz, DMSO-d_6) δ 8.70 (d, *J* = 7.6 Hz, 1H), 8.34 (d, *J* = 2.0 Hz, 1H), 7.88 (d, *J* = 8.4 Hz, 1H), 7.80 (dd, *J* = 8.8, 2.4 Hz, 1H), 7.57 (d, *J* = 9.2 Hz, 1H), 7.22-7.18 (m,1H), 7.14 (s, 1H), 6.86 (t, *J* = 6.8 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) 157.8, 138.0, 135.1, 133.2, 129.7, 128.9, 127.7, 126.6, 126.4, 125.9, 120.2, 113.8, 100.5; FT-IR (film) 3069, 2958, 2922, 2851, 1726, 1666, 1633, 1573, 1510, 1469, 1305, 1157, 1146, 839, 824, 736, 677, 558 cm^{-1}; HRMS (ESI) calcd for $C_{13}H_8ClNO$ $[M+H]^+$ 230.0367, found 230.0368; mp 144-146 ^oC.

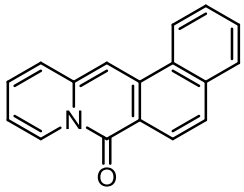
5*H*-[1,3]dioxolo[4,5-*g*]pyrido[1,2-*b*]isoquinolin-5-one (**2n**)



Yellow solid, 30 mg, 63% yield, eluent: petroleum ether/ethyl acetate 10/1. ^1H NMR (400 MHz, DMSO-d_6) δ 8.65 (d, *J* = 7.6 Hz, 1H), 7.66 (s, 1H), 7.47 (d, *J* = 9.2 Hz,

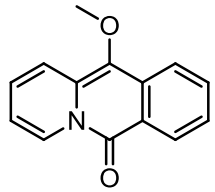
1H), 7.24 (s, 1H), 7.11-7.07 (m, 1H), 6.98 (s, 1H), 6.78 (dd, *J* = 7.2, 6.4 Hz, 1H), 6.20 (s, 2H); ^{13}C NMR (125 MHz, DMSO-d_6) 157.4, 152.7, 147.4, 136.5, 134.6, 126.4, 125.9, 125.6, 115.0, 113.2, 104.0, 103.0, 102.5, 101.0; FT-IR (film) 3423, 3122, 2920, 1660, 1637, 1608, 1571, 1531, 1478, 1242, 1233, 1143, 1030, 935, 816, 649, 543 cm^{-1}; HRMS (ESI) calcd for $C_{14}H_9NO_3$ $[M+H]^+$ 240.0655, found 240.0652; mp 233-235 ^{o}C.

7*H*-benzo[*f*]pyrido[1,2-*b*]isoquinolin-7-one (**2o**)



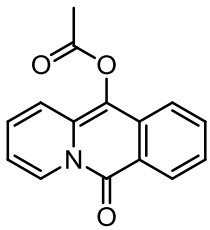
Yellow solid, 30 mg, 61% yield, eluent: petroleum ether/ethyl acetate 15/1. ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.89 (d, *J* = 7.5 Hz, 1H), 8.81 (d, *J* = 7.5 Hz, 1H), 8.33 (d, *J* = 8.5 Hz, 1H), 8.11 (s, 1H), 8.06 (dd, *J* = 7.0, 2.0 Hz, 1H), 7.84 (d, *J* = 9.0 Hz, 1H), 7.80-7.75 (m, 3H), 7.37 (dd, *J* = 8.0, 6.5 Hz, 1H), 7.03 (dd, *J* = 12.5, 6.5 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) 158.2, 138.7, 135.6, 134.8, 129.5, 129.1, 128.4, 128.3, 127.4, 126.7, 126.2, 125.7, 125.0, 124.2, 116.0, 114.5, 97.4; FT-IR (film) 3425, 3055, 2920, 2851, 1651, 1626, 1597, 1567, 1411, 1391, 1159, 1135, 822, 759, 732, 535 cm^{-1}; HRMS (ESI) calcd for $C_{17}H_{11}NO$ $[M+H]^+$ 246.0913, found 246.0914; mp 198-201 ^{o}C.

11-methoxy-6*H*-pyrido[1,2-*b*]isoquinolin-6-one (**2p**)



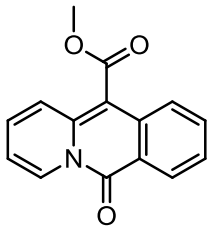
Orange solid, 38 mg, 84% yield, eluent: petroleum ether/ethyl acetate 10/1. ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.68 (d, *J* = 7.5 Hz, 1H), 8.47 (d, *J* = 8.0 Hz, 1H), 7.98 (d, *J* = 8.5 Hz, 1H), 7.92-7.89 (m, 1H), 7.68 (d, *J* = 9.0 Hz, 1H), 7.60 (dd, *J* = 7.5, 7.0 Hz, 1H), 7.19-7.16 (m, 1H), 6.80-6.77 (m, 1H), 3.88 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) 157.8, 132.4, 132.1, 131.9, 130.1, 128.9, 126.3, 125.5, 120.4, 120.0, 111.7, 62.1; FT-IR (film) 3424, 3106, 3064, 2928, 1658, 1632, 1607, 1572, 1549, 1525, 1472, 1443, 1424, 1389, 1343, 1120, 1082, 964, 765, 686, 592 cm^{-1}; HRMS (ESI) calcd for $C_{14}H_{11}NO_2$ $[M+H]^+$ 226.0863, found 226.0862; mp 148-151 ^{o}C.

6-oxo-6*H*-pyrido[1,2-*b*]isoquinolin-11-yl acetate (**2q**)



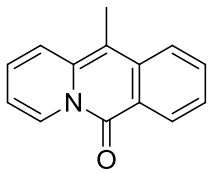
Yellow solid, 34 mg, 67% yield, eluent: petroleum ether/ethyl acetate 10/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.82 (d, *J* = 7.6 Hz, 1H), 8.65 (d, *J* = 8.4 Hz, 1H), 7.78 (dd, *J* = 8.4, 7.2 Hz, 1H), 7.59-7.52 (m, 2H), 7.28-7.26 (m, 1H), 7.03 (dd, *J* = 9.2, 6.4 Hz, 1H), 6.63 (t, 6.4 Hz, 1H), 2.54 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) 169.5, 158.2, 132.9, 131.1, 130.0, 128.8, 127.0, 126.4, 125.6, 122.5, 119.6, 119.5, 119.1, 111.6, 20.5; FT-IR (film) 3421, 3109, 2927, 2852, 1753, 1667, 1635, 1610, 1575, 15828, 1477, 1452, 1427, 1390, 1343, 1279, 1071, 758, 700, 679, 576 cm^{-1}; HRMS (ESI) calcd for $C_{15}H_{11}NO_3$ $[M+H]^+$ 254.0812, found 254.0811; mp 196-199 ^{o}C.

methyl 6-oxo-6*H*-pyrido[1,2-*b*]isoquinoline-11-carboxylate (**2r**)



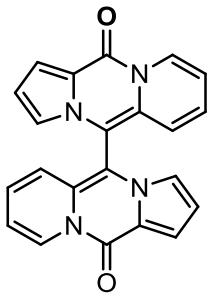
Yellow solid, 28 mg, 55% yield, eluent: petroleum ether/ethyl acetate 10/1. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.87 (d, *J* = 7.6 Hz, 1H), 8.47 (dd, *J* = 8.0, 0.8 Hz, 1H), 7.95 (d, *J* = 8.4 Hz, 1H), 7.88-7.81 (m, 2H), 7.60-7.56 (m, 1H), 7.44-7.39 (m, 1H), 6.99-6.96 (m, 1H), 3.99 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d_6) 167.5, 158.6, 138.0, 133.9, 130.8, 128.3, 126.9, 126.0, 124.3, 123.3, 118.8, 113.7, 103.1, 52.9; FT-IR (film) 3409, 3114, 3038, 2955, 1716, 1677, 1641, 1609, 1525, 1472, 1428, 1385, 1226, 1210, 1114, 1025, 779, 745, 707, 573 cm^{-1}; HRMS (ESI) calcd for $C_{15}H_{11}NO_3$ $[M+H]^+$ 254.0812, found 254.0814; mp 175-177 ^{o}C.

11-methyl-6*H*-pyrido[1,2-*b*]isoquinolin-6-one (**2s**)[2]



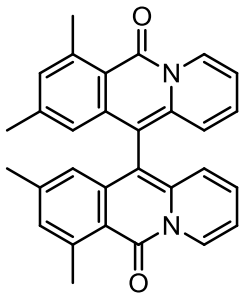
Yellow solid, 34 mg, 82% yield, eluent: petroleum ether/ethyl acetate 10/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.89 (d, *J* = 7.5 Hz, 1H), 8.70 (d, *J* = 8.0 Hz, 1H), 7.92 (d, *J* = 8.5 Hz, 1H), 7.81-7.77 (m, 1H), 7.58 (d, *J* = 9.5 Hz, 1H), 7.53 (dd, *J* = 8.0, 7.0 Hz, 1H), 6.99 (dd, *J* = 9.0, 6.0 Hz, 1H), 6.61 (t, *J* = 6.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) 159.0, 136.2, 133.9, 132.3, 128.7, 126.8, 125.6, 124.9, 122.8, 122.4, 120.3, 111.3, 104.3, 12.2.

11*H*,11'*H*-[5,5'-bipyrido[1,2-*a*]pyrrolo[1,2-*d*]pyrazine]-11,11'-dione (**2t**)



Orange solid, 22 mg, 60% yield, eluent: petroleum ether/ethyl acetate 8/1. ^{1}H NMR (500 MHz, DMSO-d_6) δ 8.366 (d, *J* = 6.0 Hz, 2H), 7.28 (d, *J* = 3.0 Hz, 2H), 7.14 (s, 2H), 6.73-6.67 (m, 6H), 6.41 (t, *J* = 7.0 Hz, 2H); ^{13}C NMR (125 MHz, DMSO-d_6) 152.3, 128.4 127.1, 125.4, 121.1, 121.0, 121.0, 117.5, 113.9, 110.9, 110.3, 101.6; FT-IR (film) 3420, 3104, 2924, 2853, 1668, 1640, 1354, 1316, 1197, 1095, 800, 755, 728, 692, 465 cm^{-1}; HRMS (ESI) calcd for $C_{22}H_{14}N_4O_2$ $[M+H]^+$ 367.1190, found 367.1174; mp 235-238 ^{o}C.

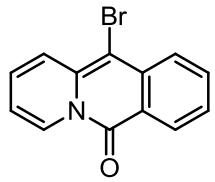
7,7',9,9'-tetramethyl-6*H*,6'*H*-[11,11'-bipyrido[1,2-*b*]isoquinoline]-6,6'-dione (**2u**)



Orange solid, 21 mg, 47% yield, eluent: petroleum ether/ethyl acetate 8/1. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.85 (d, *J* = 7.2 Hz, 2H), 7.11 (s, 2H), 6.95-6.91 (m, 2H),

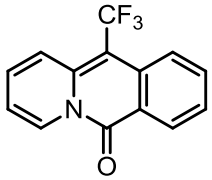
6.77-6.71 (m, 6H), 2.99 (s, 6H), 2.16 (s, 6H); ^{13}C NMR (125 MHz, DMSO-d_6) 159.2, 142.7, 130.3, 128.7, 126.8, 123.6, 121.7, 112.4, 24.8, 21.9; FT-IR (film) 3442, 3108, 2964, 2923, 1656, 1632, 1608, 1577, 1525, 1453, 1316, 1295, 1135, 886, 847, 760, 695, 566 cm^{-1}; HRMS (ESI) calcd for $C_{30}H_{24}N_2O_2$ $[M+H]^+$ 445.1911, found 445.1913; mp 158-161 ^{o}C.

11-bromo-6*H*-pyrido[1,2-*b*]isoquinolin-6-one (**3a**)[3]



Yellow solid, 34 mg, 62% yield, eluent: petroleum ether/ethyl acetate 10/1. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.81(d, *J* = 7.6 Hz, 1H), 8.49 (d, *J* = 8.0 Hz, 1H), 8.09 (d, *J* = 8.4 Hz, 1H), 7.96 (dd, *J* = 8.0, 7.2 Hz, 1H), 7.91 (d, *J* = 9.2 Hz, 1H), 7.63 (dd, *J* =7.6, 7.2 Hz, 1H), 7.41 (dd, *J* = 8.4, 6.4 Hz, 1H), 6.92 (t, *J* = 6.8 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) 158.2, 135.1, 135.1, 134.5, 130.8, 128.6, 127.2, 126.4, 125.7, 124.6, 120.0, 113.2, 93.5; FT-IR (film) 3424, 1674, 1661, 1632, 1603, 1561, 1519, 1462, 1446, 1343, 1167, 918, 750, 675, 562 cm^{-1}; HRMS (ESI) calcd for $C_{13}H_8BrNO$ $[M+H]^+$ 273.9862, found 273.9862; mp 199-202 ^{o}C.

11-(trifluoromethyl)-6*H*-pyrido[1,2-*b*]isoquinolin-6-one (**3b**)[4]



Yellow solid, 30 mg, 57% yield, eluent: petroleum ether/ethyl acetate 10/1. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.03 (d, *J* = 7.6 Hz, 1H), 8.65 (dd, *J* = 8.0, 0.8 Hz, 1H), 8.12 (d, *J* = 8.4 Hz, 1H), 7.94 (d, *J* = 10.0 Hz, 1H), 7.82-7.77 (m, 1H), 7.54 (dd, *J* = 7.6, 7.2 Hz, 1H), 7.29-7.25 (m, 1H), 6.81-6.78 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) 159.4, 138.1, 133.5, 133.3, 130.2, 128.8, 127.6, 125.6, 123.5 (q, *J* = 6.1 Hz), 118.8 (q, *J* = 6.7 Hz), 119.4; FT-IR (film) 3086, 2959, 2923, 2851, 1691, 1639, 1607, 1571, 1474, 1430, 1333, 1320, 1242, 1216, 1083, 946, 769, 687 cm^{-1}; HRMS (ESI) calcd for $C_{14}H_9F_3NO$ $[M+H]^+$ 264.0631, found 264.0635; mp 133-137 ^{o}C.

IV References

(1) (a) Shang, S.; Yang, Z. W.; Wang, Y.; Zhang, S. L.; Liu, L. *J. Am. Chem. Soc.* **2010**, *132*, 14391; (b) Desai, L. V.; Stowers, K. J.; Sanford, M. S. *J. Am. Chem. Soc.* **2008**, *130*, 13285; (c) Xu, Z.; Zhang, C.; Jiao, N. *Angew. Chem., Int. Ed.* **2012**, *51*, 11367;

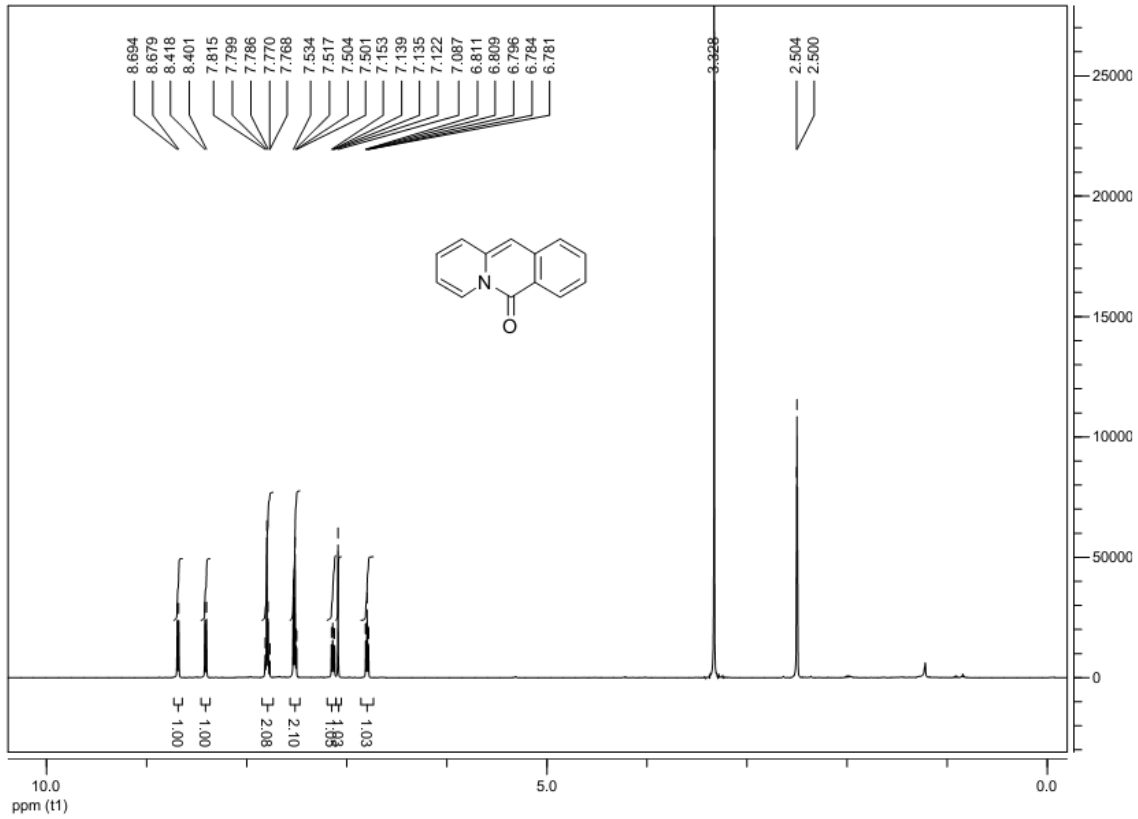
(2) M. A. Cinellu, A. Zucca, S. Stoccoro, G. Minghetti, M. Manassero, M. Sansoni, *J. Chem. Soc. Dalton. Trans.* **1995**, 2865.

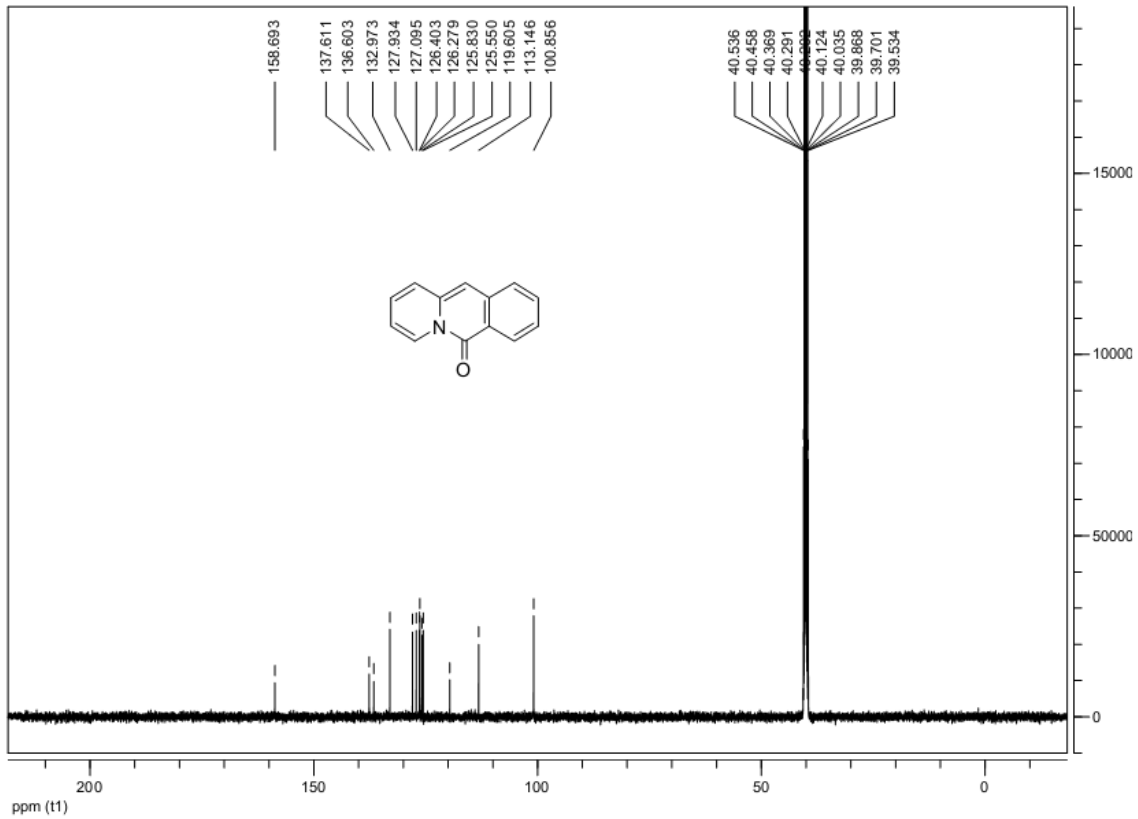
(3) S. Song, X. Sun, X. Li, Y. Yuan, N. Jiao, *Org. Lett.*, **2015**, *17*, 2886.

(4) B. Zhang, C. M. Lichtenfeld, C. G. Daniliuc, A. Studer, *Angew Chem., Ed. Int.*, **2013**, *52*, 10792.

V. Copies of NMR Spectra

2a





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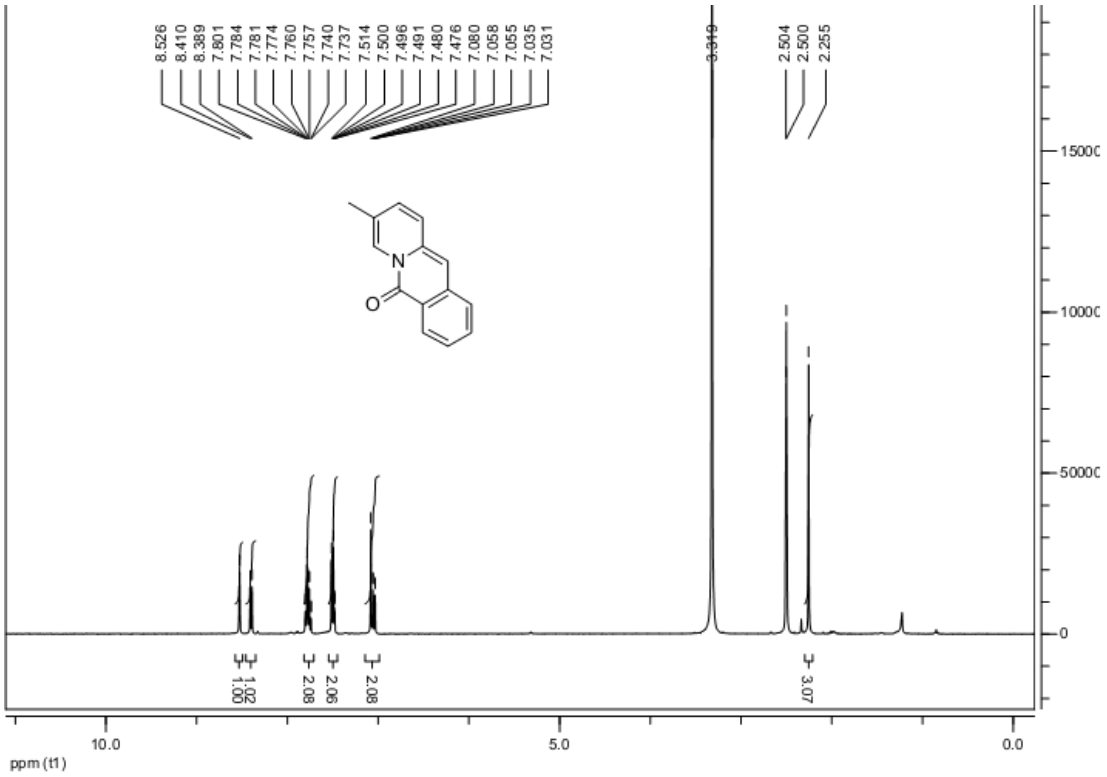
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7.491
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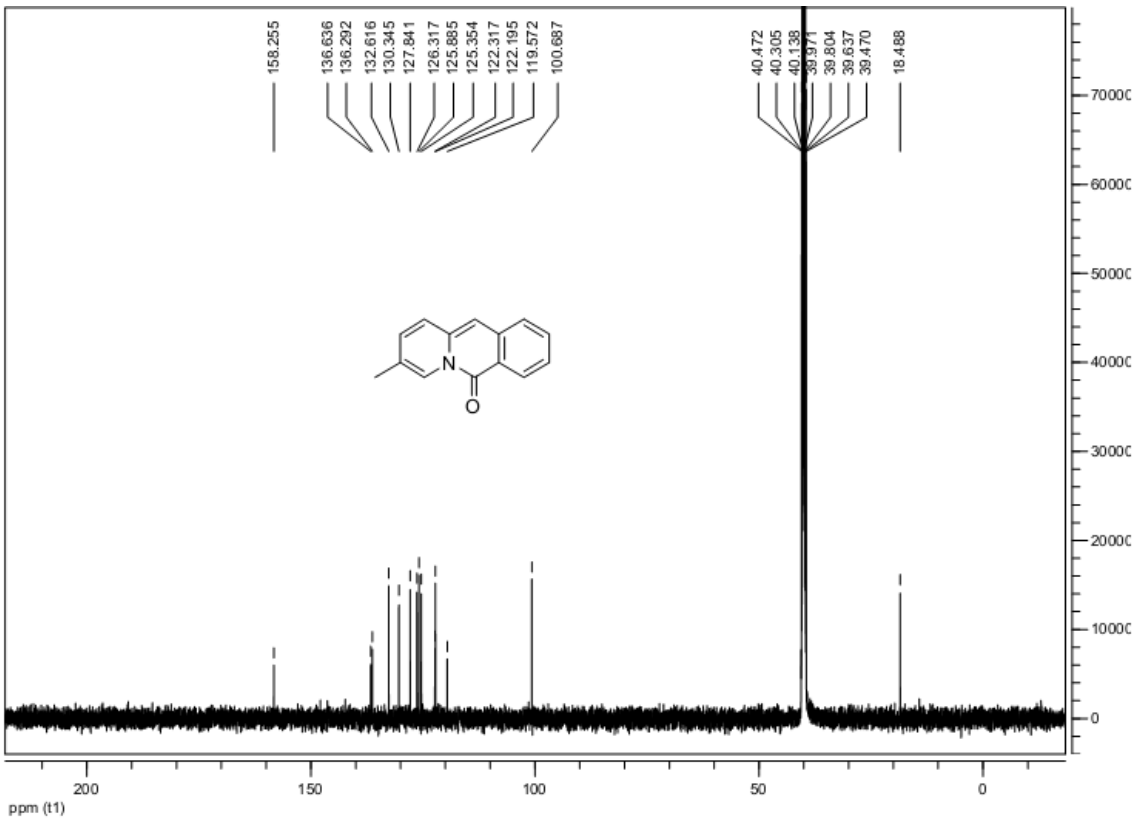
7.476
7.080
7.058
7.055
7.035
7.031
3.349
2.504
2.500
2.255
1.00
1.02
2.08
2.06
2.08
3.07
10.0
5.0
0.0
ppm (t1)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40.472
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39.470
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200
150
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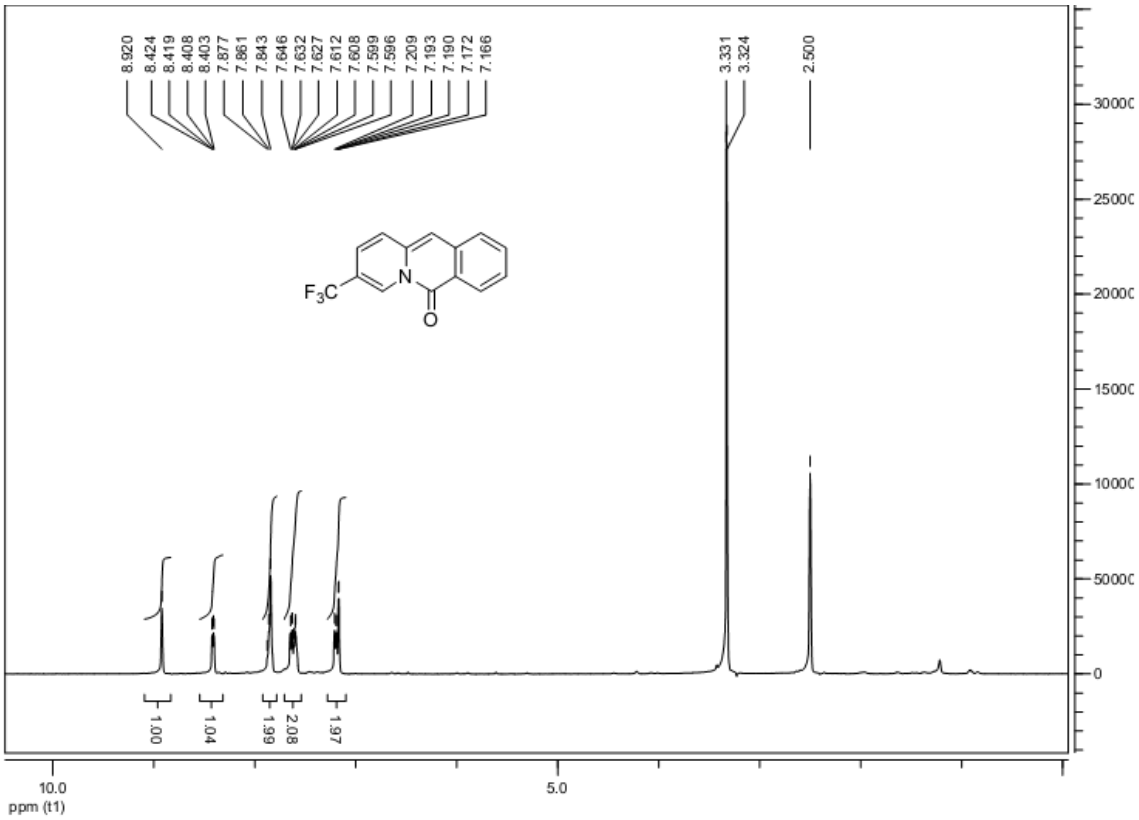
50
0
ppm (t1)

2c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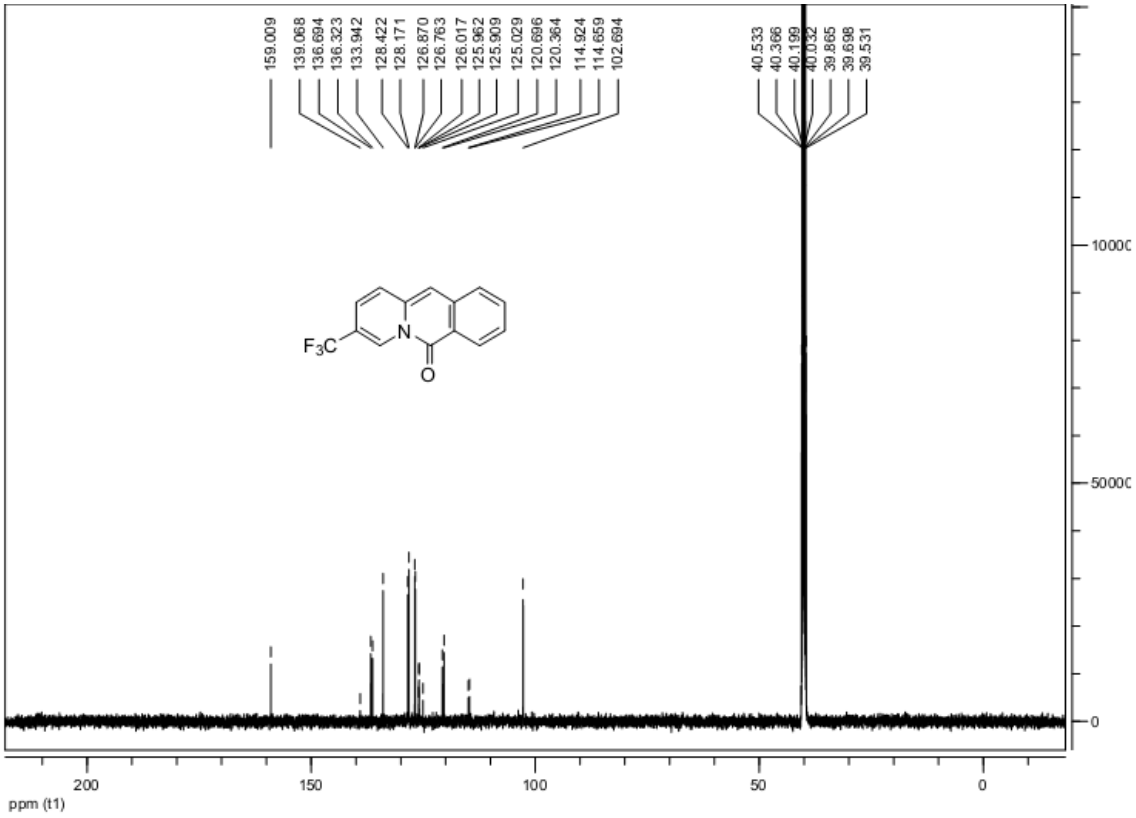
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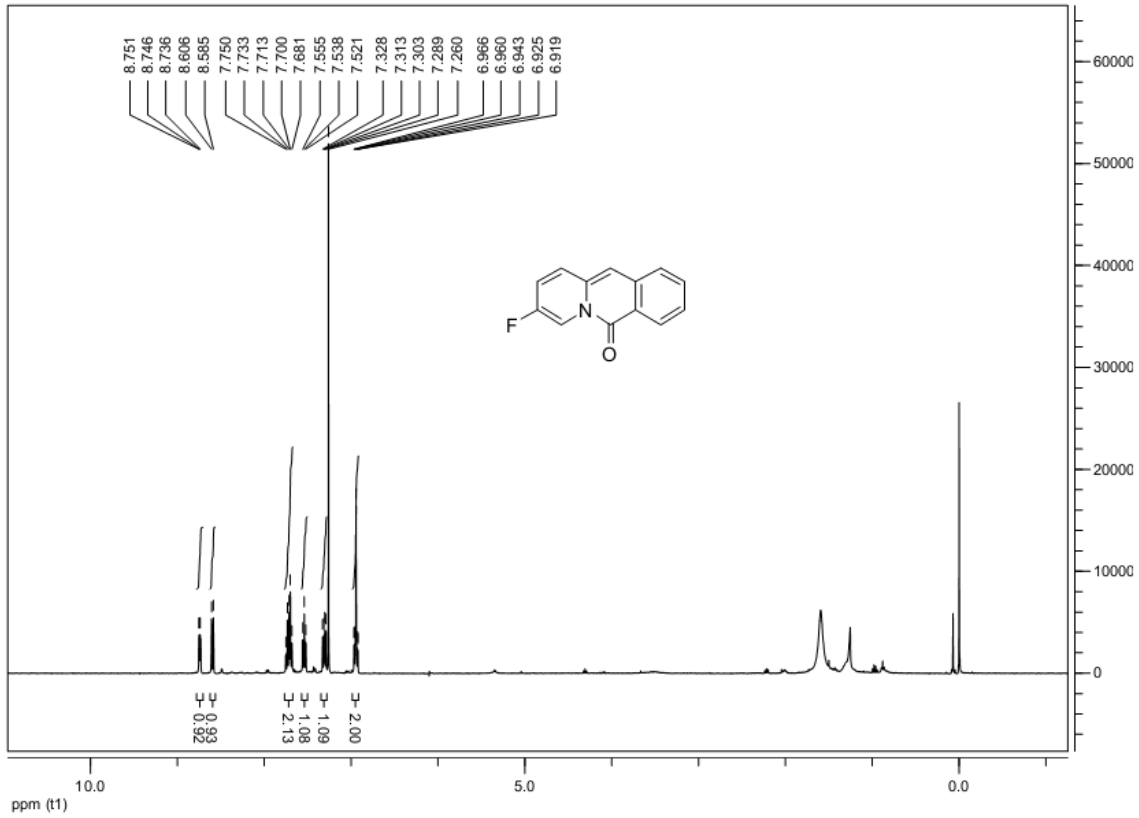
ppm (t1)

2d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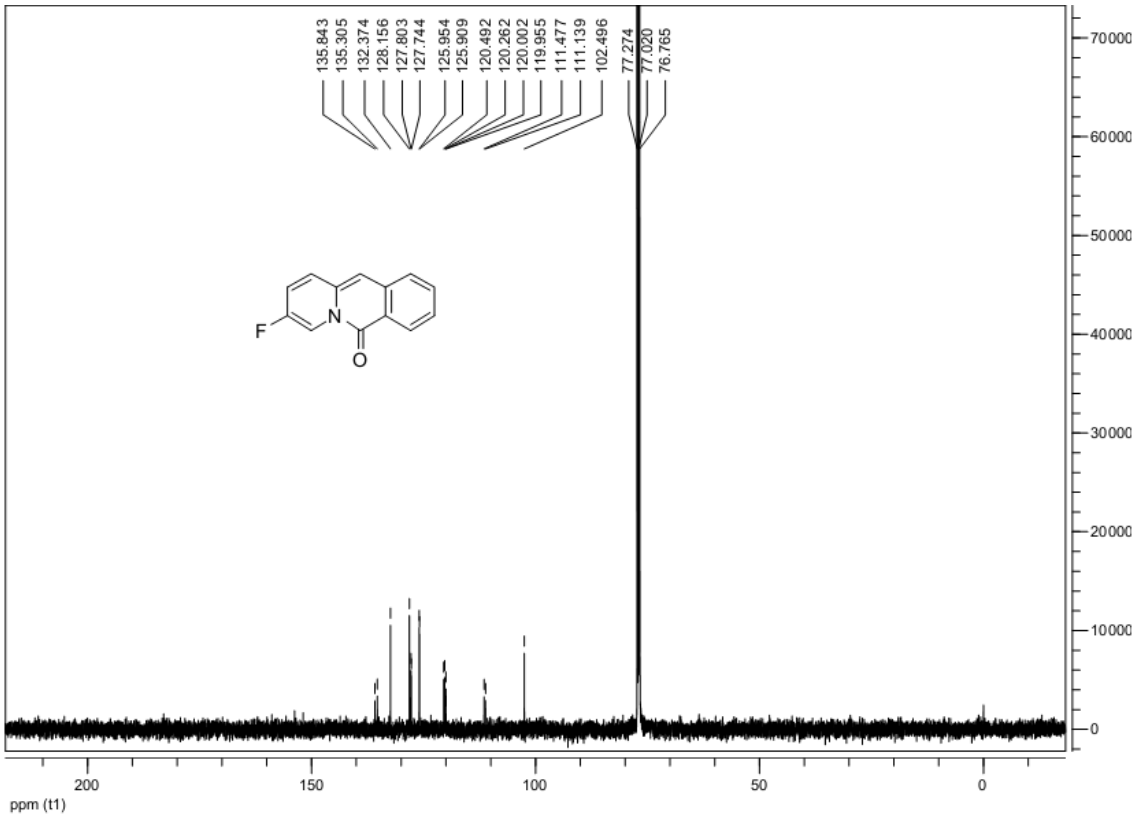
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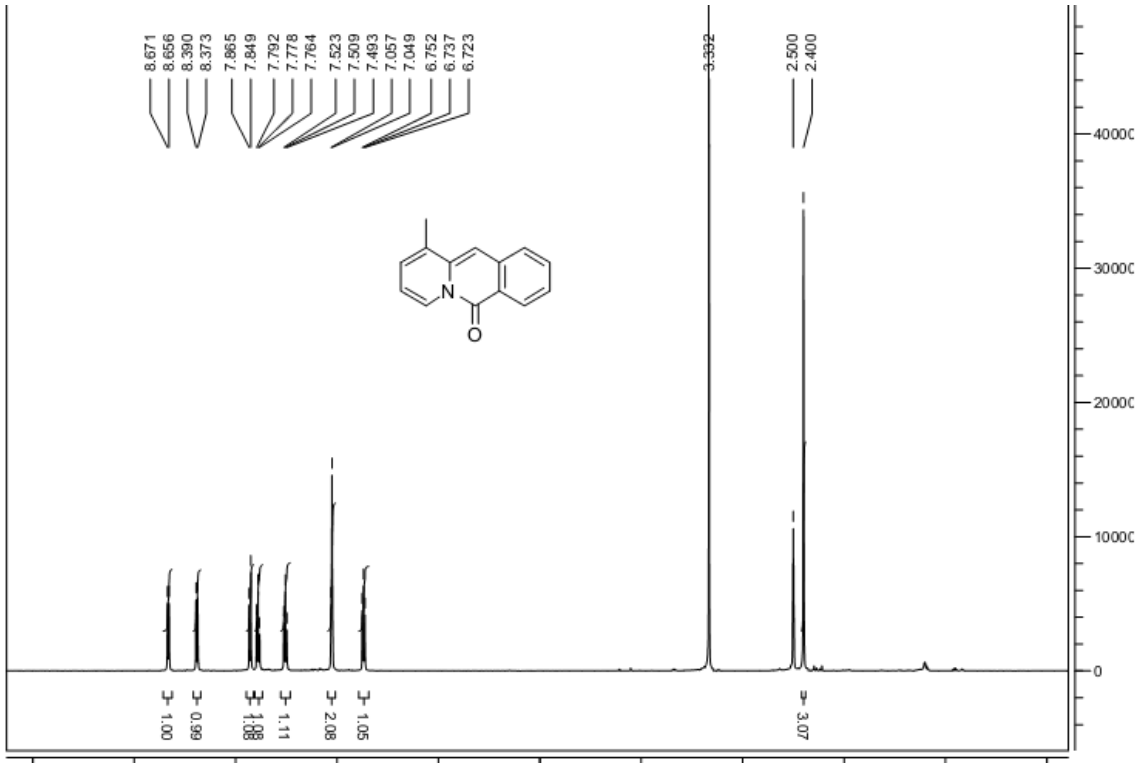
ppm (t1)

2e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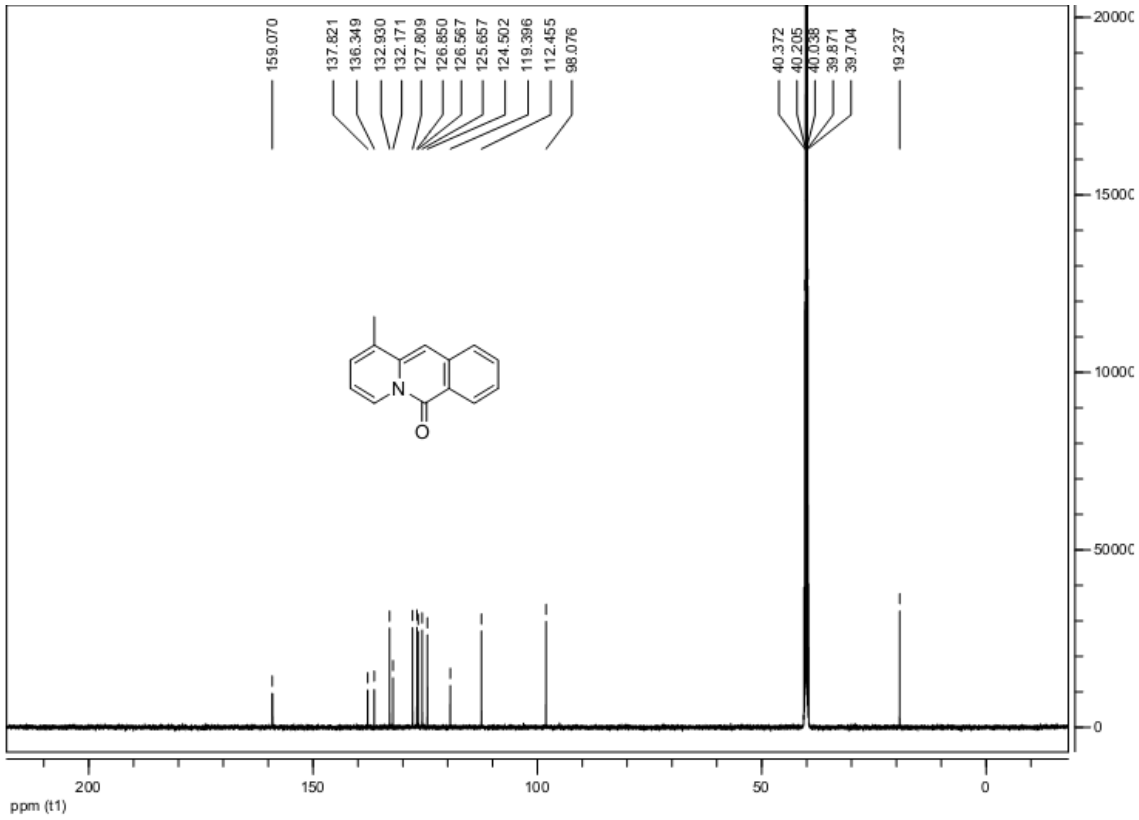
1.05
3.07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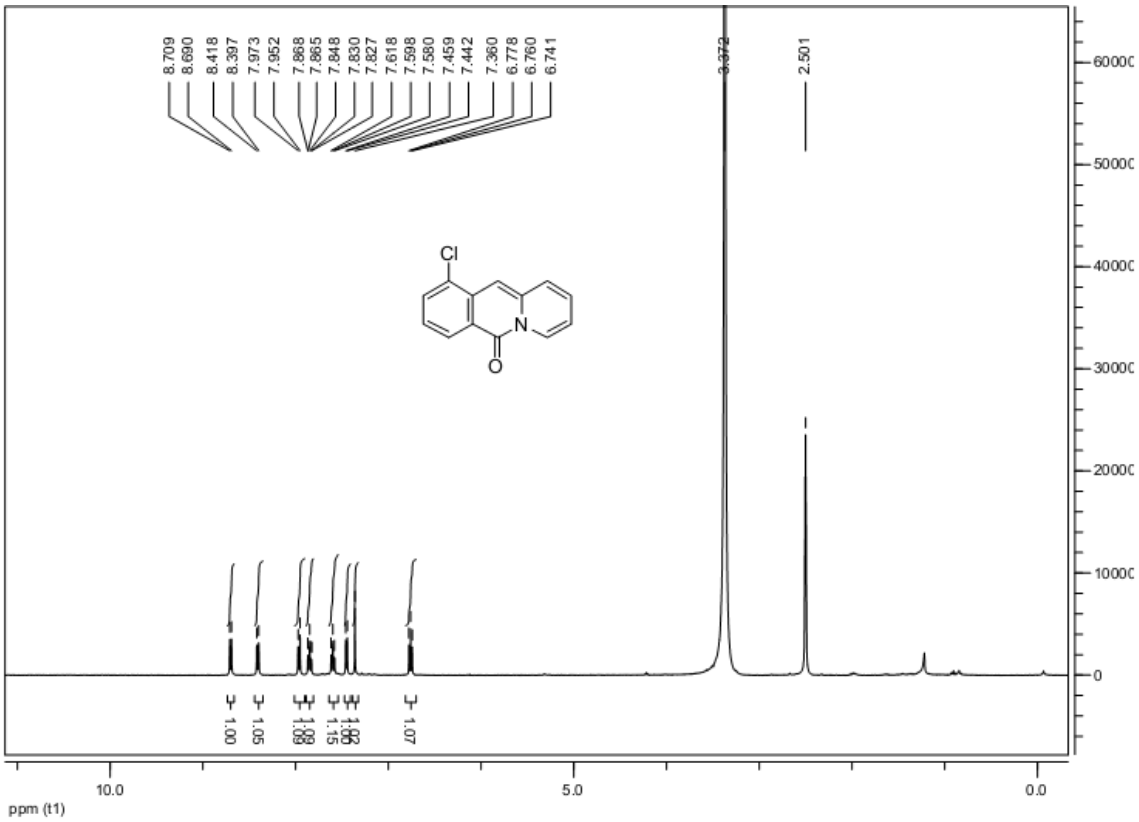
137.821
136.349
132.930
132.171
127.809
126.850
126.567
125.657
124.502
119.396
112.455
98.076
40.372
40.205
40.038
39.871
39.704
19.237
ppm (t1)

2f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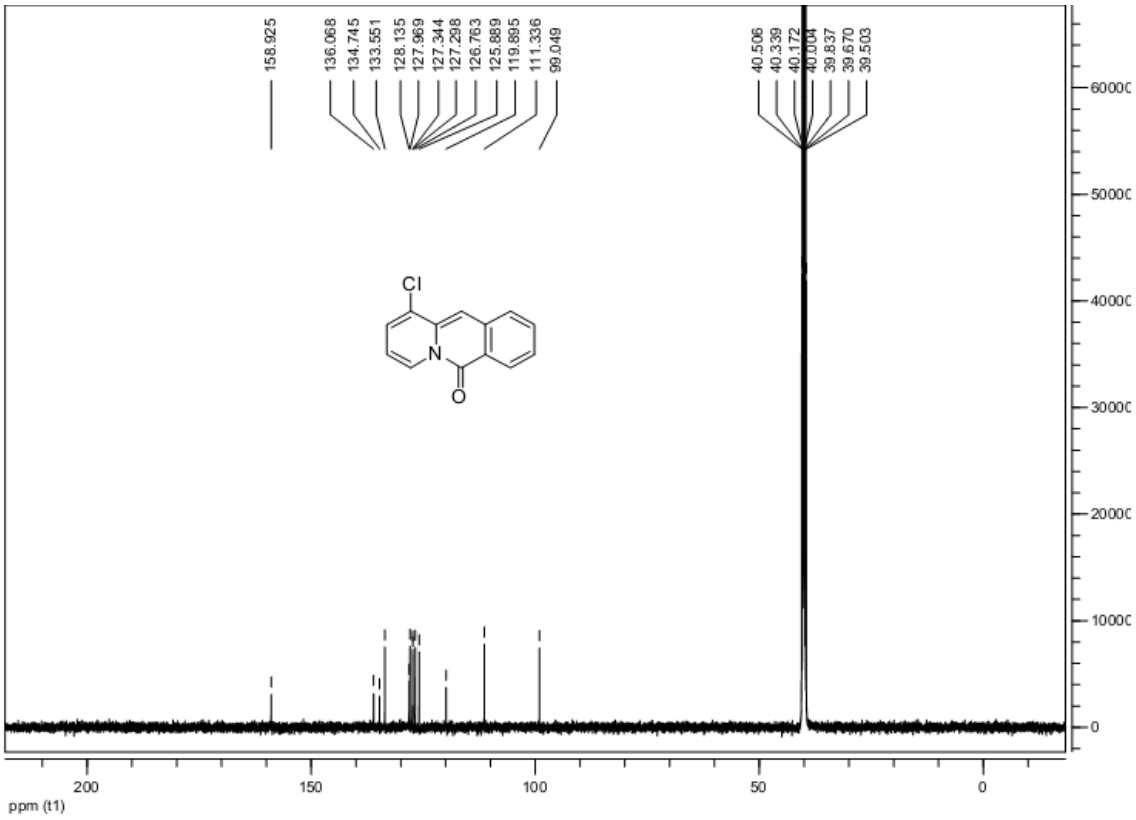
8.690
8.418
8.397
7.973
7.952
7.868
7.865
7.848
7.830
7.827
7.618
7.598
7.580
7.459
7.442
7.360
6.778
6.760
6.741
3.372
2.501
Cl
N
O
1.00
1.05
1.08
1.08
1.15
1.08
1.08
1.07
10.0
5.0
0.0
ppm (t1)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134.745
133.551
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127.344
127.298
126.763
125.889
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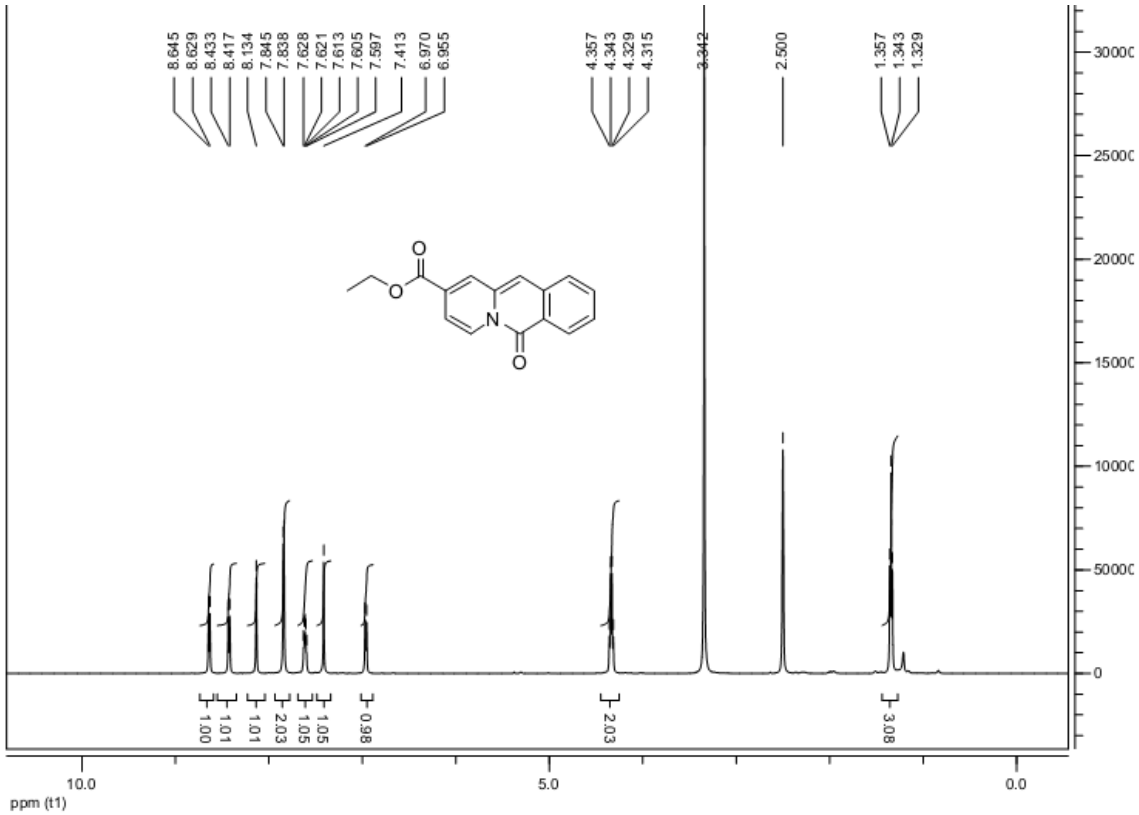
111.336
99.049
40.506
40.339
40.172
40.004
39.837
39.670
39.503
Cl
N
O
200
150
100
50
0
ppm (t1)

2g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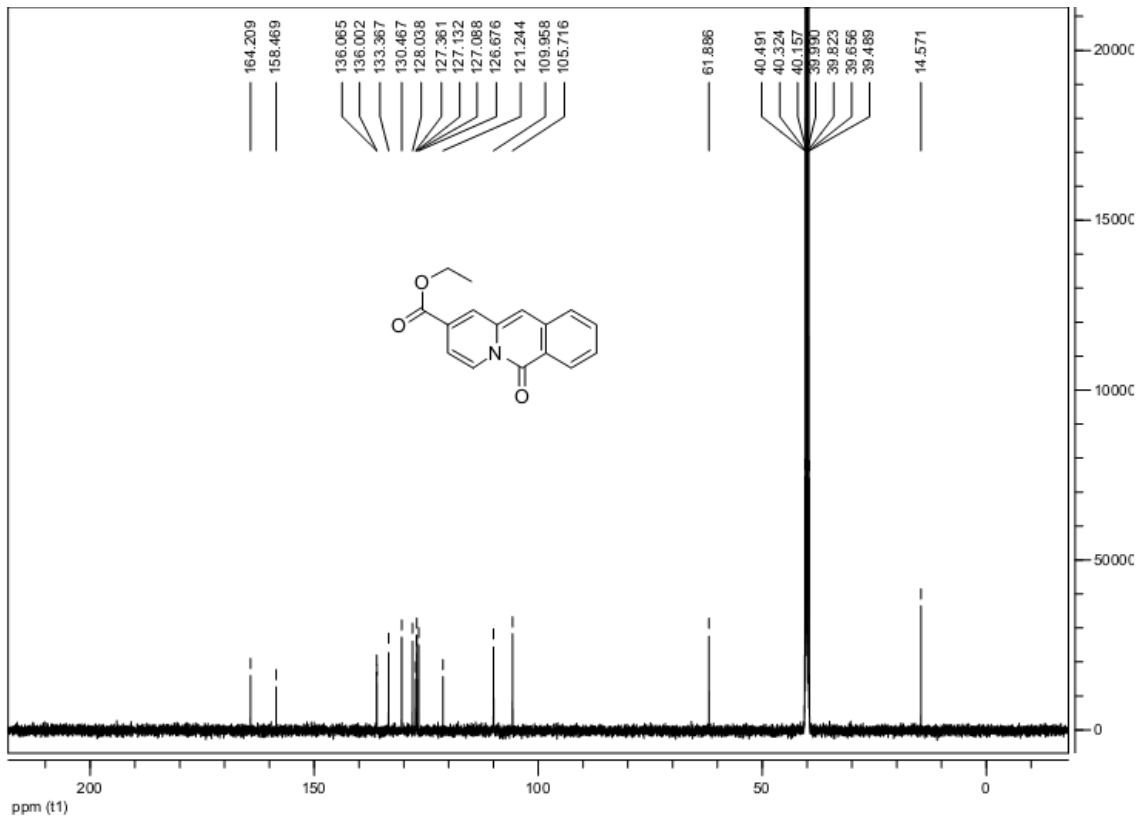
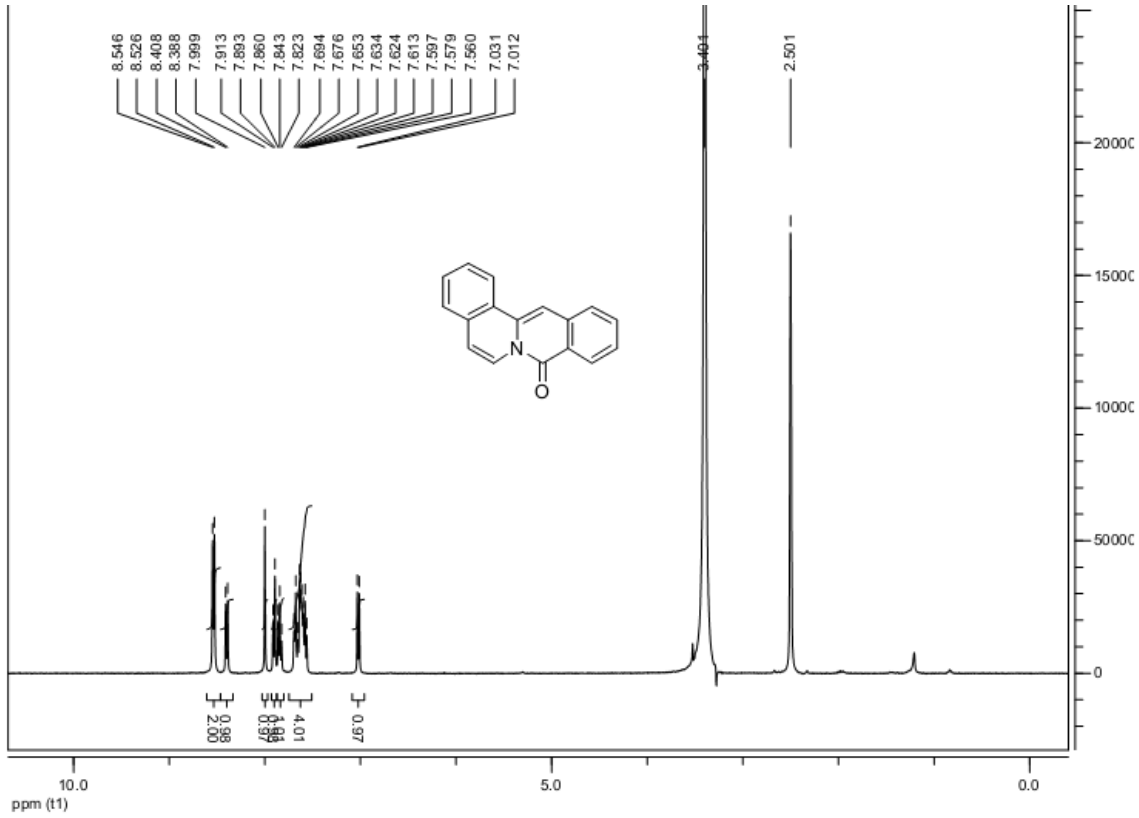
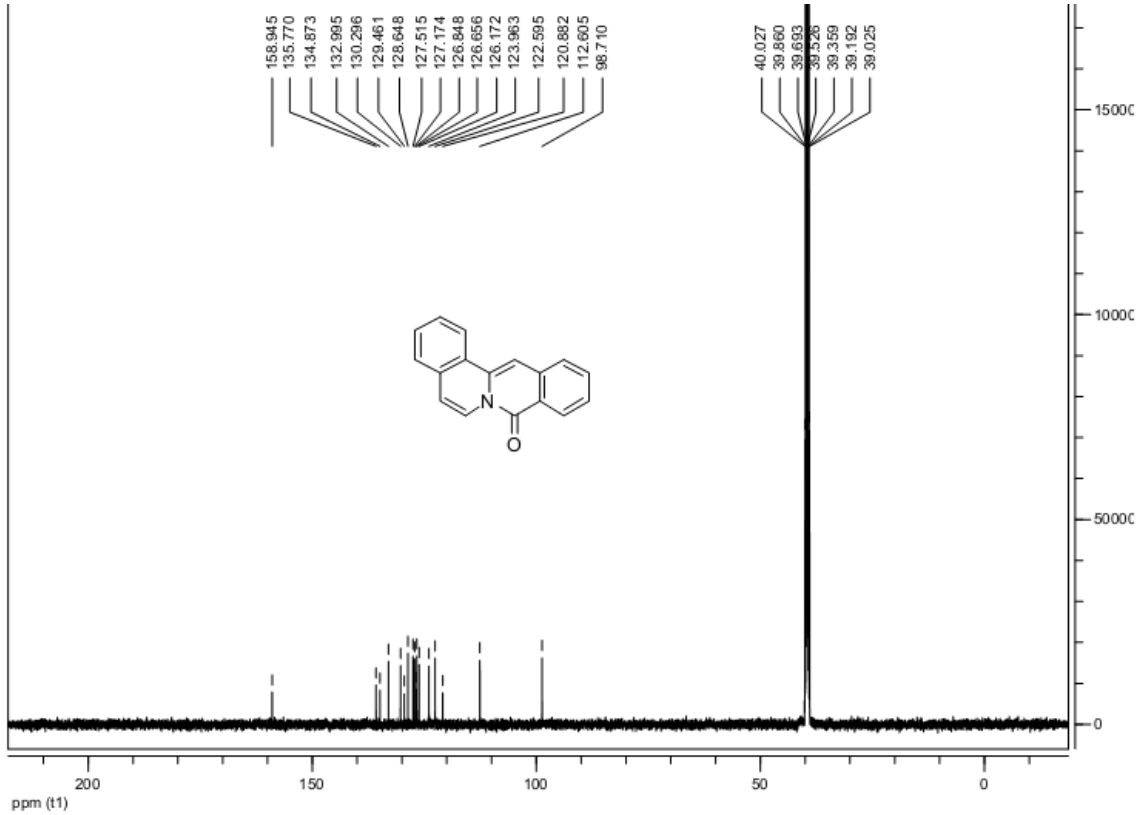
8.629
8.433
8.417
8.134
7.845
7.838
7.628
7.621
7.613
7.605
7.597
7.413
6.970
6.955
4.357
4.343
4.329
4.315
3.342
2.500
1.357
1.343
1.329
1.00
1.01
1.01
2.03
1.05
1.05
0.98
2.03
3.08
10.0
5.0
0.0
ppm (t1)


164.209
158.469
136.065
136.002
133.367
130.467
128.038
127.361
127.132
127.088
126.676
121.244
109.958
105.716
61.886
40.491
40.324
40.157
39.990
39.823
39.656
39.489
14.571
200
150
100
50
0
ppm (t1)

2h


8.546
8.526
8.408
8.388
7.999
7.913
7.893
7.860
7.843
7.823
7.694
7.676
7.653
7.634
7.624
7.613
7.597
7.579
7.560
7.031
7.012
3.401
2.501
2.00
0.98
0.97
0.97
4.01
0.97
10.0
5.0
0.0
ppm (t1)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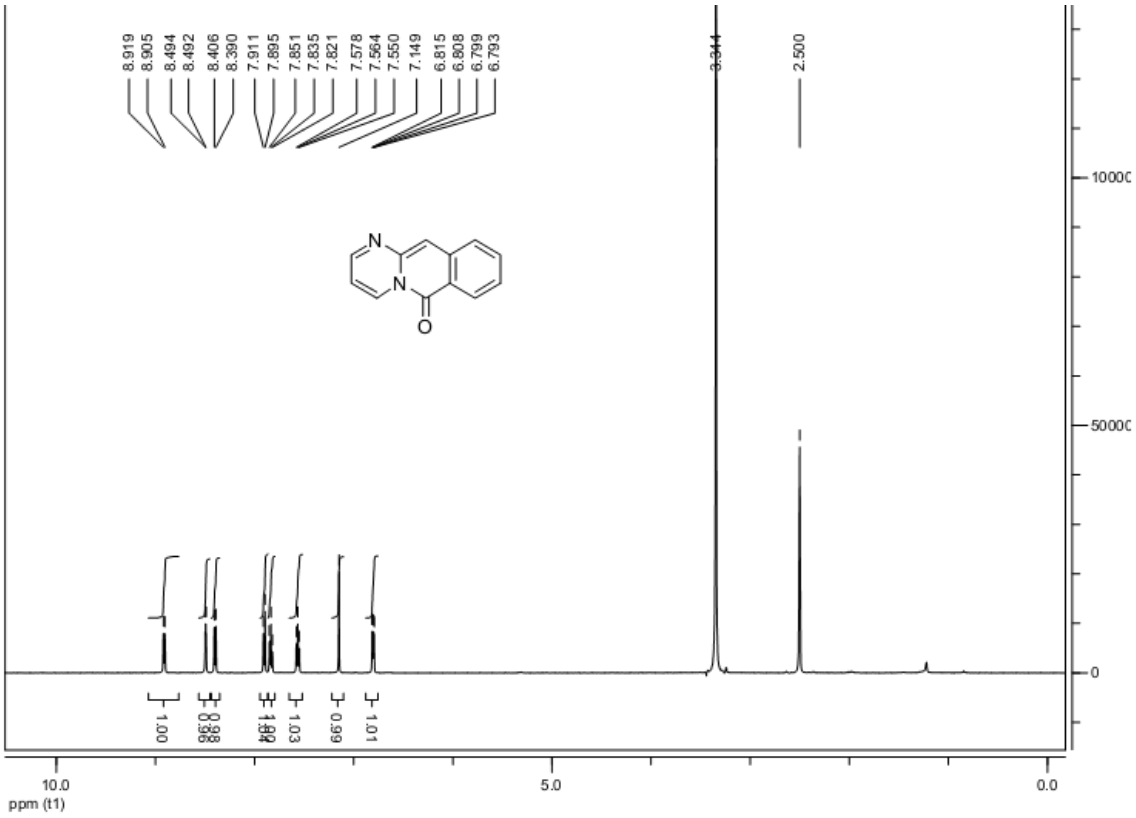
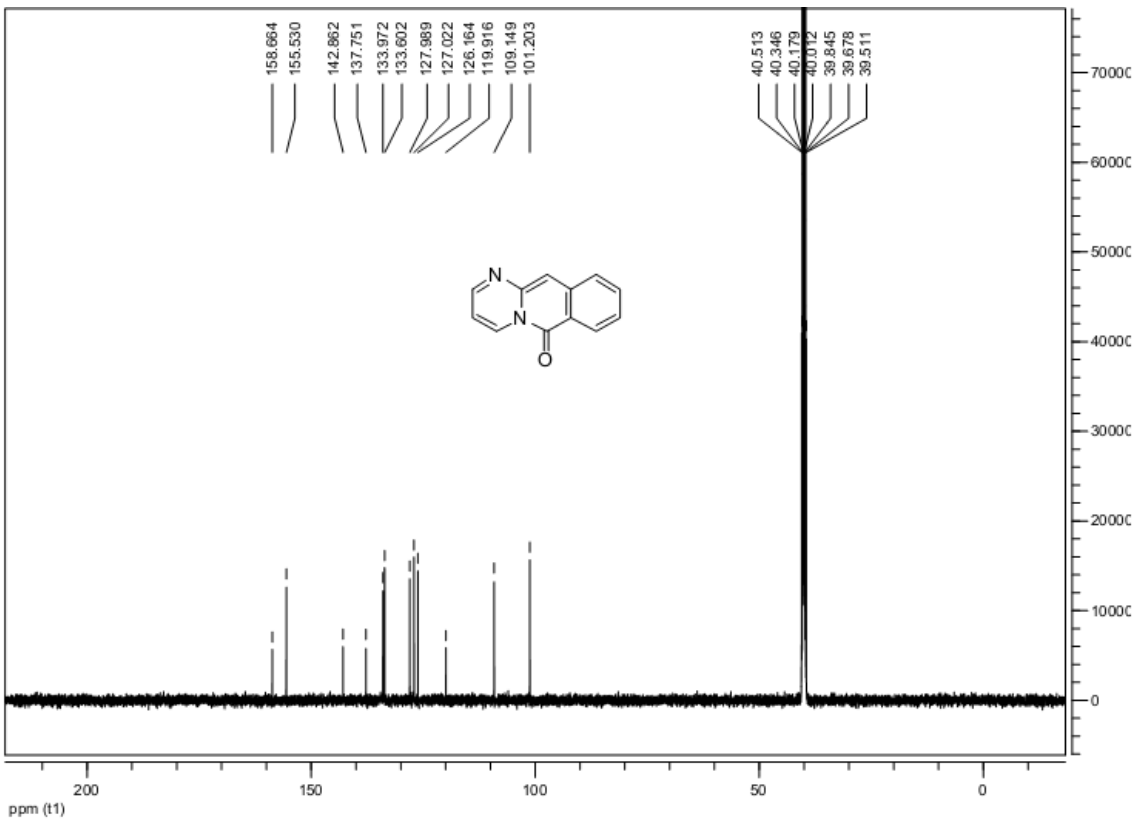
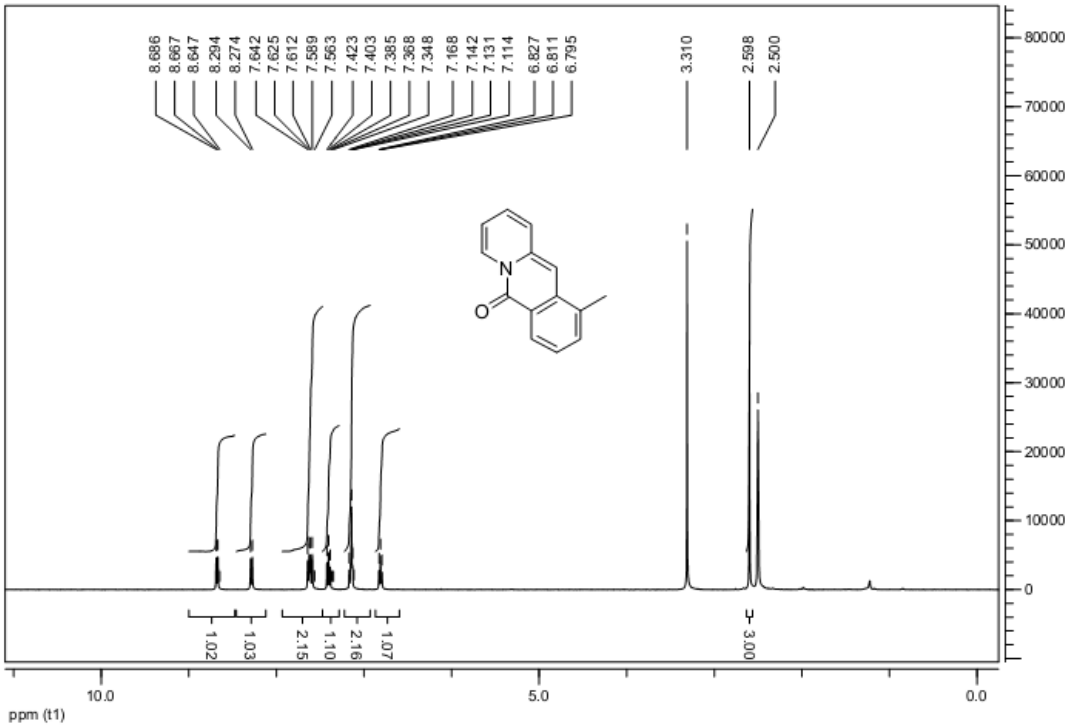
134.873
132.995
130.296
129.461
128.648
127.515
127.174
126.848
126.656
126.172
123.963
122.595
120.882
112.605
98.710
40.027
39.860
39.693
39.526
39.359
39.192
39.025
200
150
100
50
0
ppm (t1)

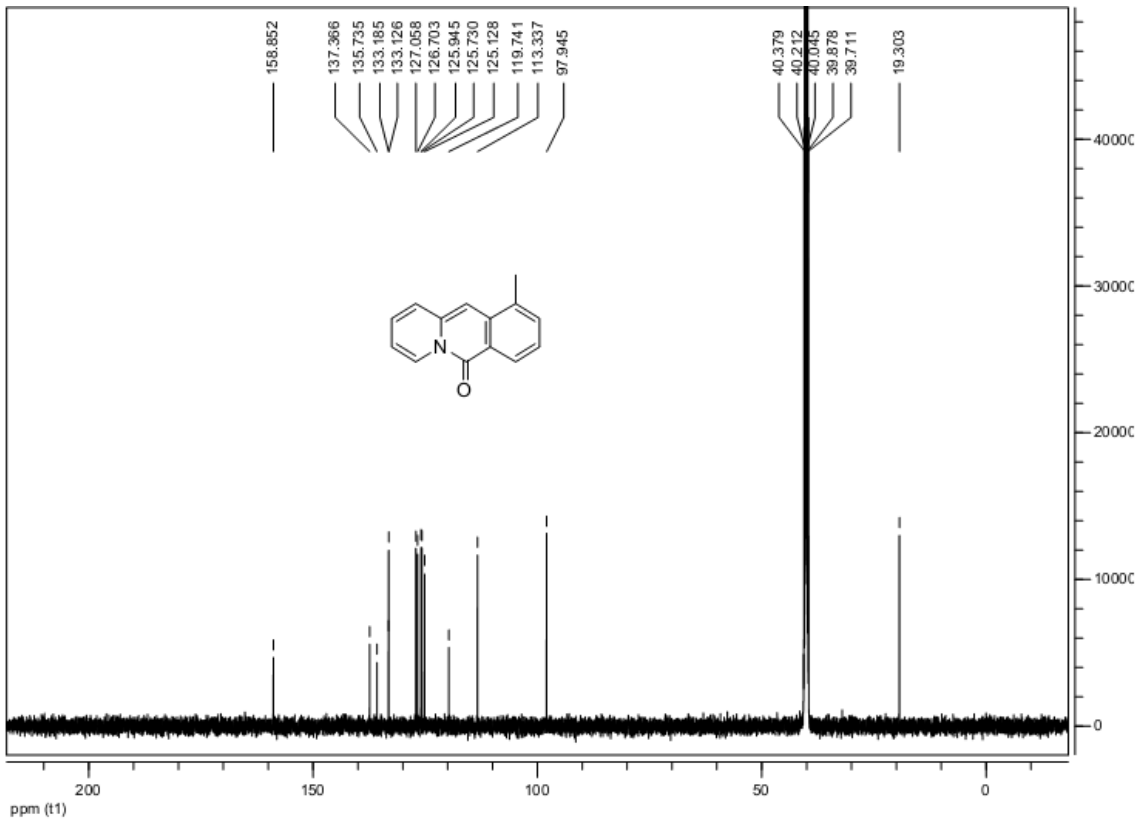
2i


8.919
8.905
8.494
8.492
8.406
8.390
7.911
7.895
7.851
7.835
7.821
7.578
7.564
7.550
7.149
6.815
6.808
6.799
6.793
3.344
2.500
1.00
0.96
0.98
1.00
1.03
0.99
1.01
10.0
5.0
0.0
ppm (t1)


158.664
155.530
142.862
137.751
133.972
133.602
127.989
127.022
126.164
119.916
109.149
101.203
40.513
40.346
40.179
40.012
39.845
39.678
39.511
200
150
100
50
0
ppm (t1)

2j


8.686
8.667
8.647
8.294
8.274
7.642
7.625
7.612
7.589
7.563
7.423
7.403
7.385
7.368
7.348
7.168
7.142
7.131
7.114
6.827
6.811
6.795
3.310
2.598
2.500
1.02
1.03
2.15
1.10
2.16
1.07
3.00
ppm (t1)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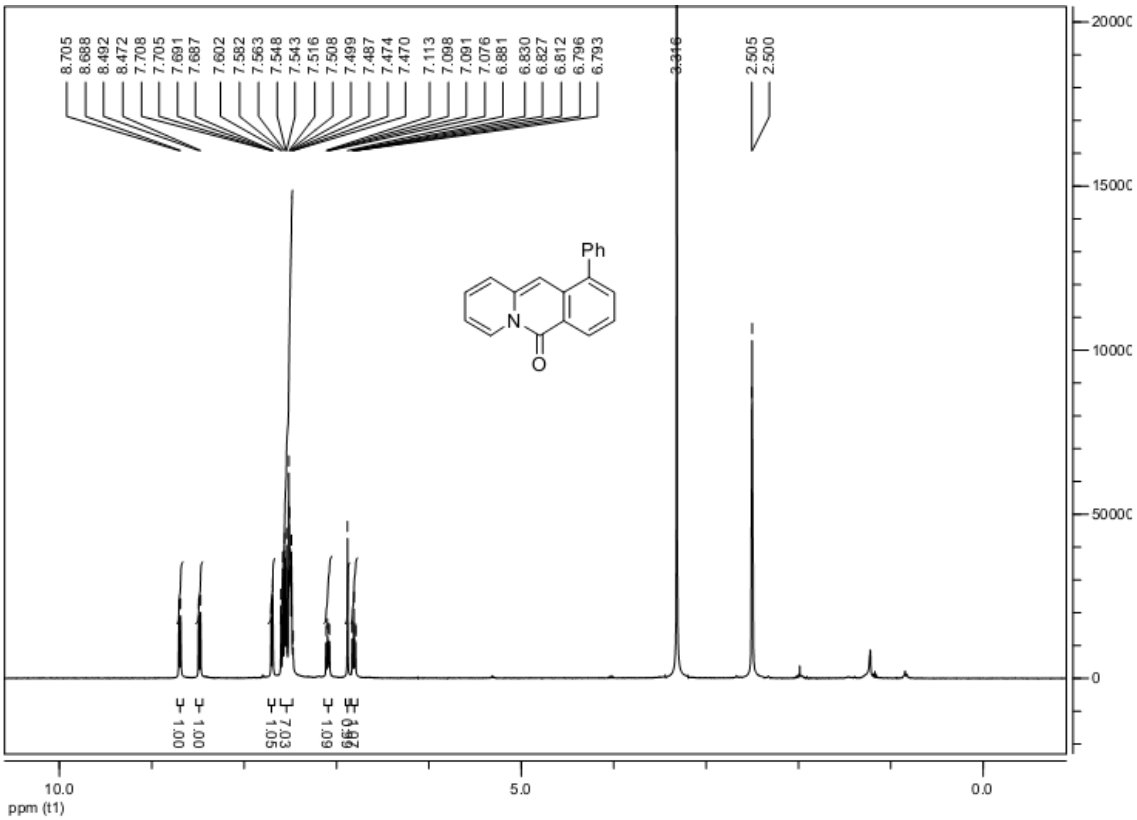
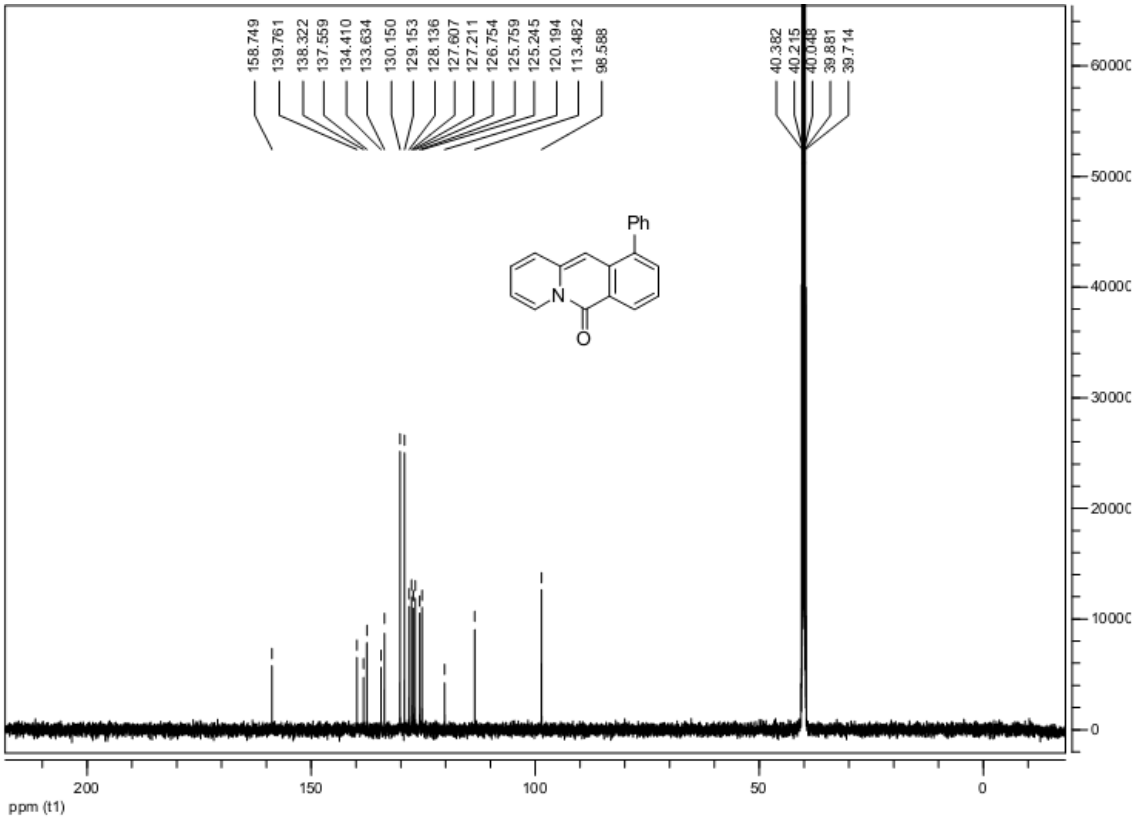
137.366
135.735
133.185
133.126
127.058
126.703
125.945
125.730
125.128
119.741
113.337
97.945
40.379
40.212
40.045
39.878
39.711
19.303
ppm (t1)

2k


8.705
8.688
8.492
8.472
8.708
7.705
7.691
7.687
7.602
7.582
7.563
7.548
7.543
7.516
7.508
7.499
7.487
7.474
7.470
7.113
7.098
7.091
7.076
6.881
6.830
6.827
6.812
6.796
6.793
3.346
2.505
2.500
Ph
N
O
1.00
1.00
7.03
1.05
1.09
0.99
0.99
10.0
5.0
0.0
ppm (t1)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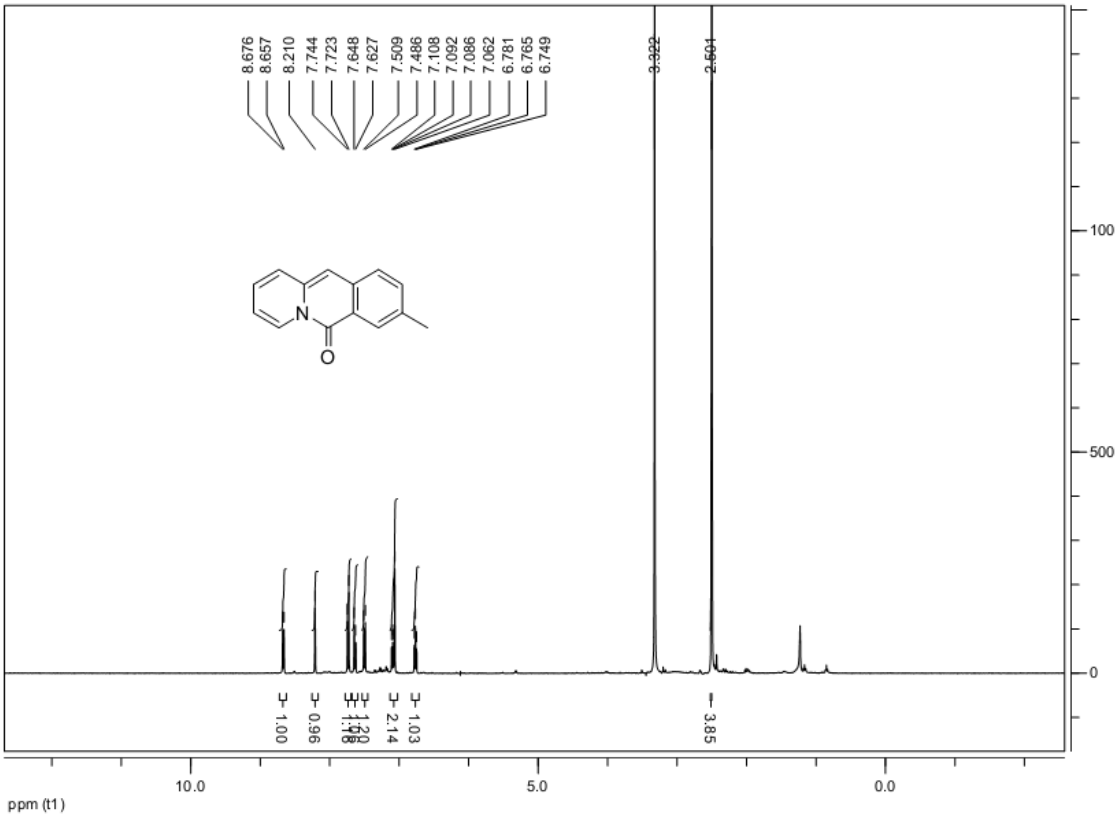
138.322
137.559
134.410
133.634
130.150
129.153
128.136
127.607
127.211
126.754
125.759
125.245
120.194
113.482
98.588
40.382
40.215
40.048
39.881
39.714
Ph
N
O
200
150
100
50
0
ppm (t1)

2l


8.676
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8.210
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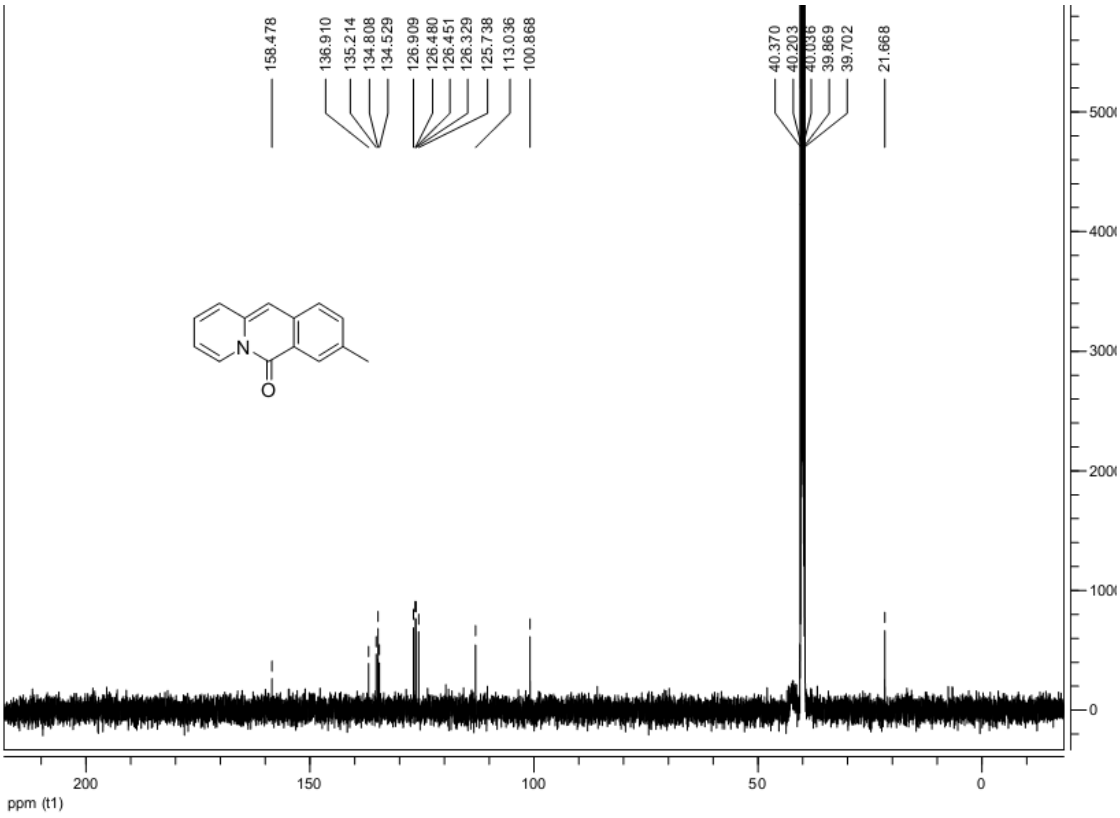
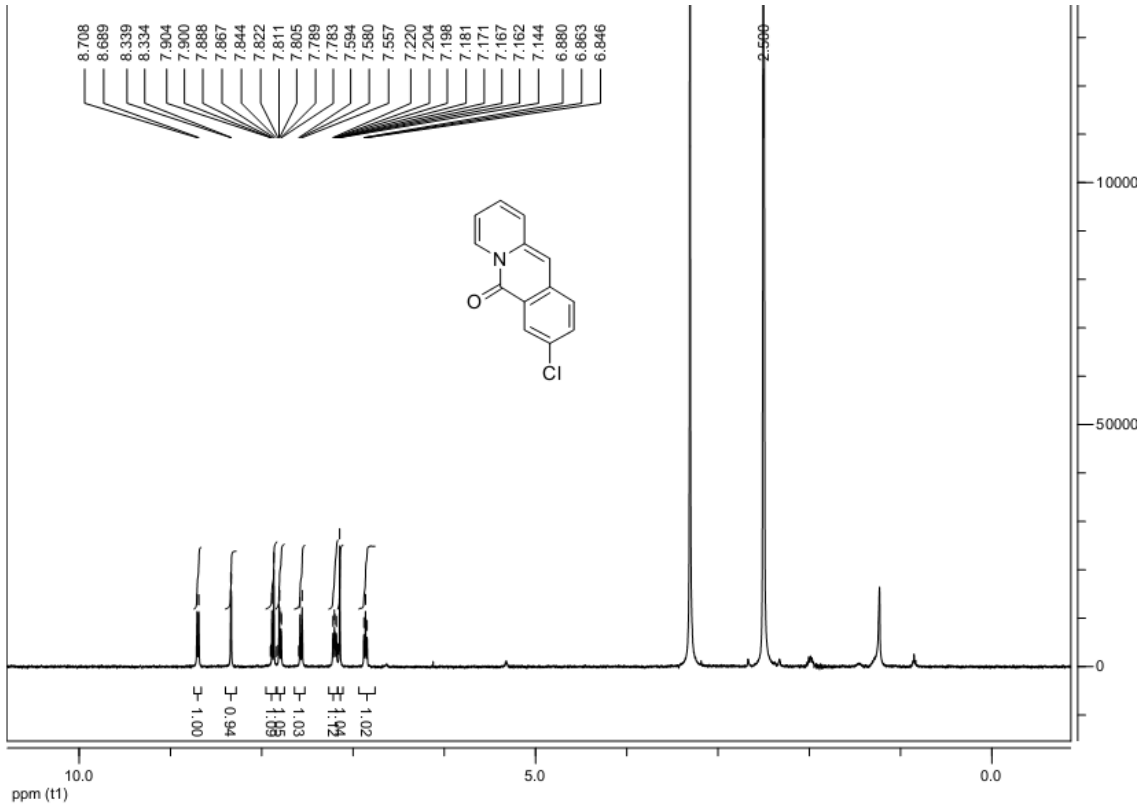
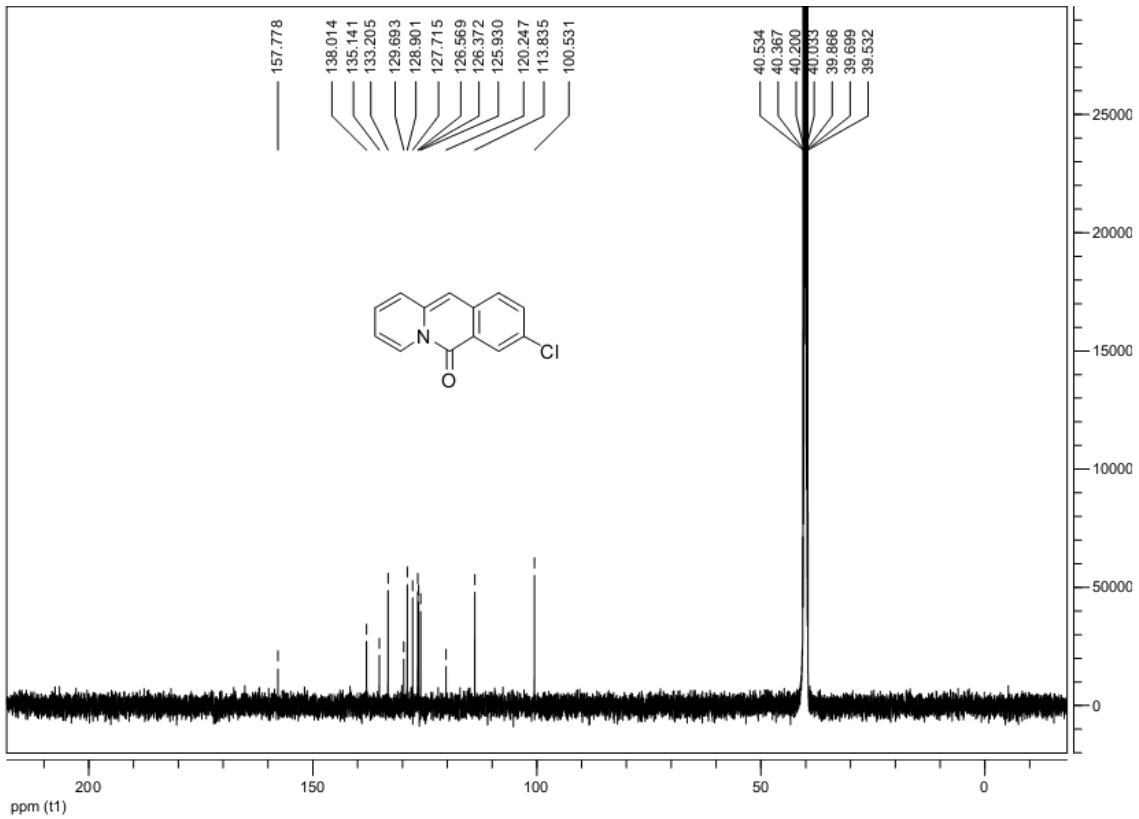
7.723
7.648
7.627
7.509
7.486
7.108
7.092
7.086
7.062
6.781
6.765
6.749
3.322
2.501
1.00
0.99
2.14
1.03
3.85
10.0
5.0
0.0
ppm (t1)


158.478
136.910
135.214
134.808
134.529
126.909
126.480
126.451
126.329
125.738
113.036
100.868
40.370
40.203
40.038
39.869
39.702
21.668
200
150
100
50
0
ppm (t1)

2m


8.708
8.689
8.339
8.334
7.904
7.900
7.888
7.867
7.844
7.822
7.811
7.805
7.789
7.783
7.594
7.580
7.557
7.220
7.204
7.198
7.181
7.171
7.167
7.162
7.144
6.880
6.863
6.846
2.500
N
O
Cl
1.00
0.94
1.05
1.03
1.04
1.02
10.0
5.0
0.0
ppm (t1)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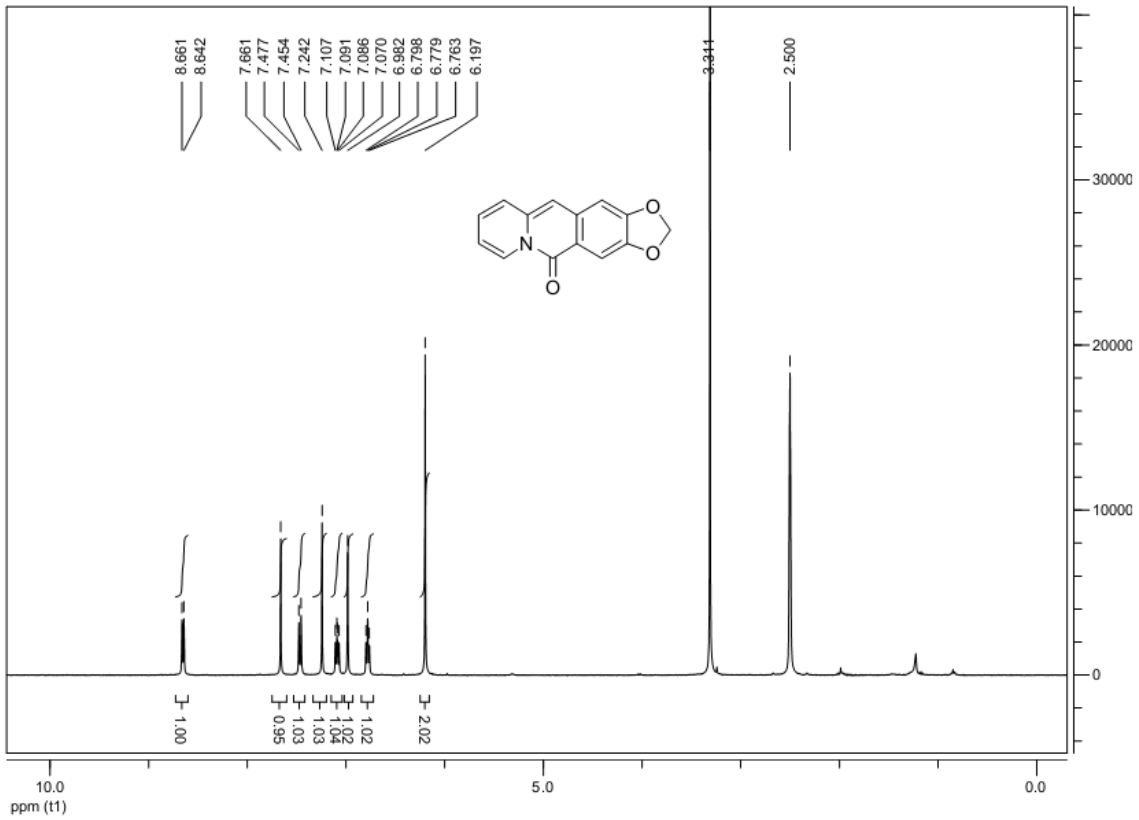
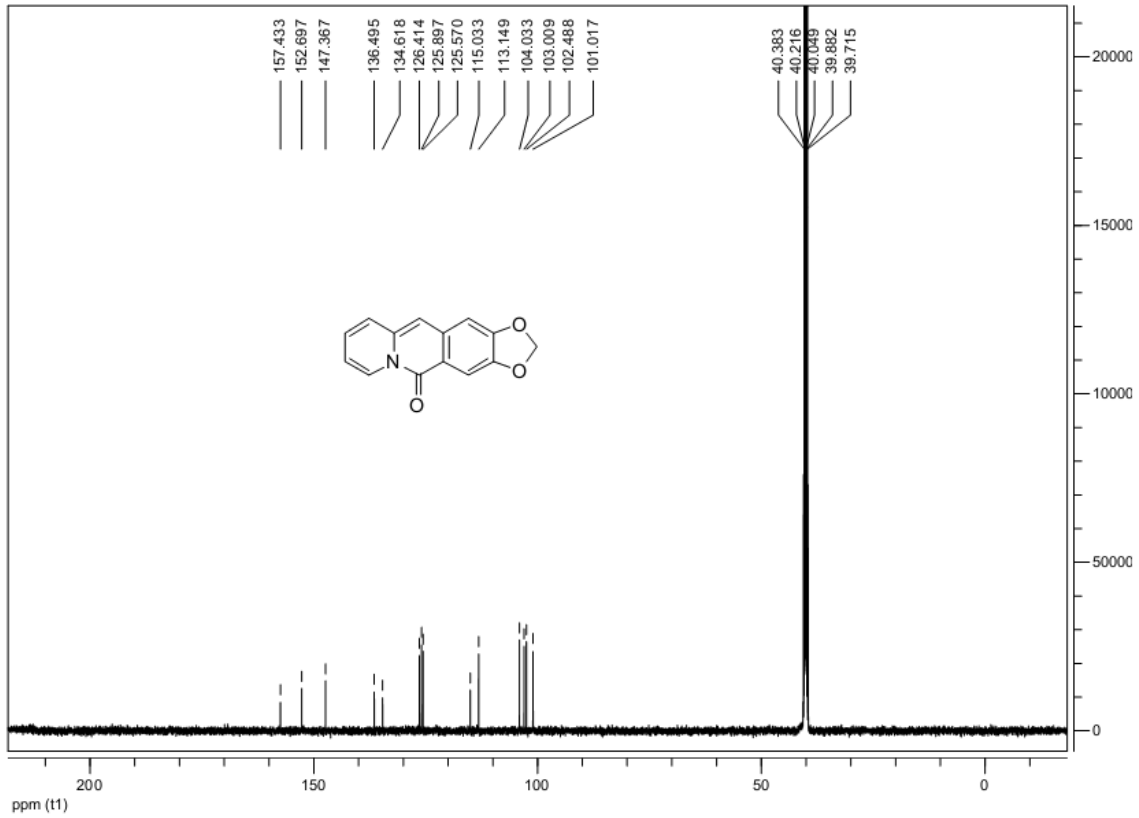
138.014
135.141
133.205
129.693
128.901
127.715
126.569
126.372
125.930
120.247
113.835
100.531
40.534
40.367
40.200
40.033
39.866
39.699
39.532
N
O
Cl
200
150
100
50
0
ppm (t1)

2n


8.661
8.642
7.661
7.477
7.454
7.242
7.107
7.091
7.086
7.070
6.982
6.798
6.779
6.763
6.197
3.311
2.500
1.00
0.95
1.03
1.03
1.04
1.02
2.02
30000
20000
10000
0
10.0
5.0
0.0
ppm (t1)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147.367
136.495
134.618
126.414
125.897
125.570
115.033
113.149
104.033
103.009
102.488
101.017
40.383
40.216
40.049
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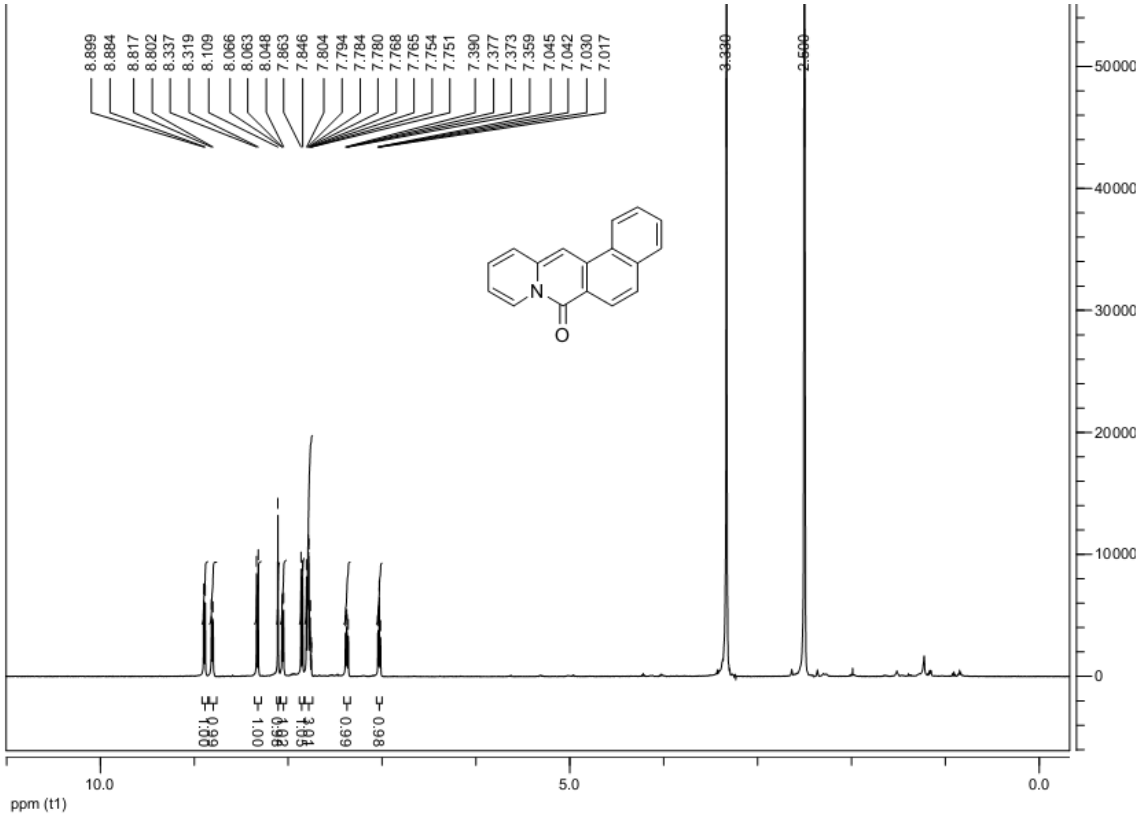
39.715
20000
15000
10000
50000
0
200
150
100
50
0
ppm (t1)

2o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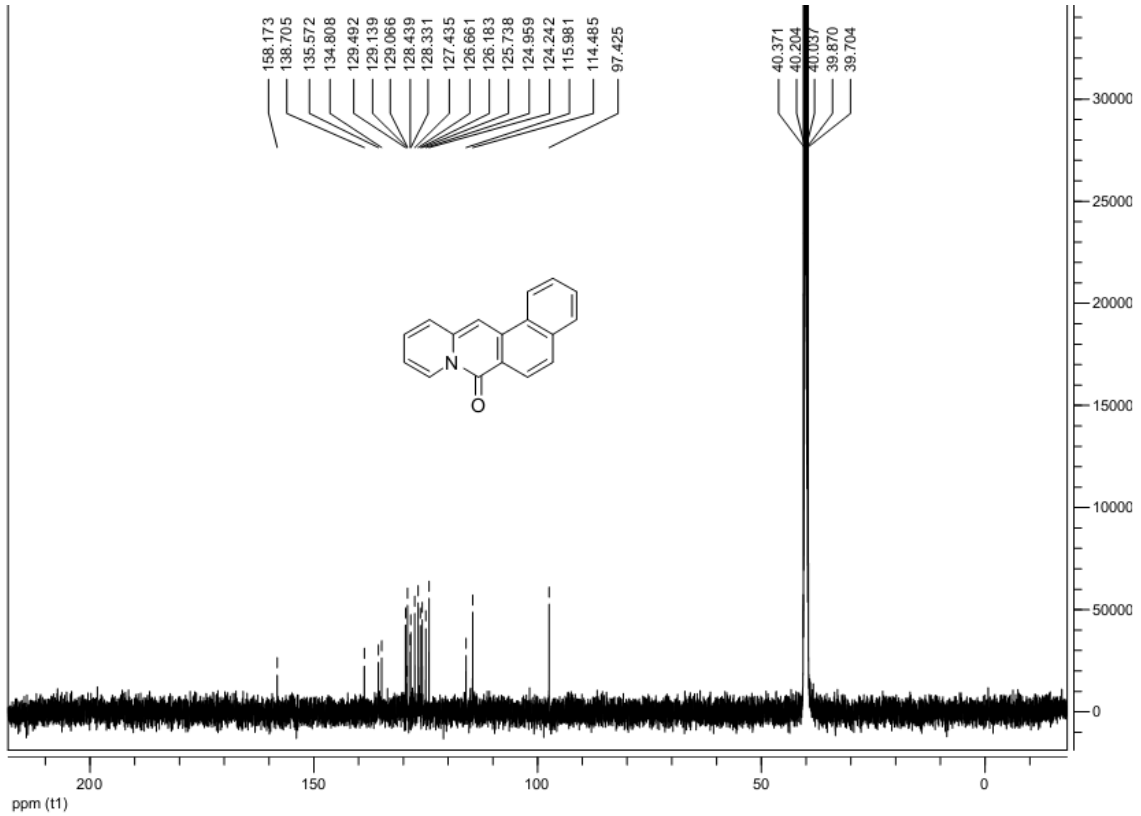
8.802
8.337
8.319
8.109
8.066
8.063
8.048
7.863
7.846
7.804
7.794
7.784
7.780
7.768
7.765
7.754
7.751
7.390
7.377
7.373
7.359
7.045
7.042
7.030
7.017
3.330
2.500
1.00
1.00
0.98
1.05
0.99
0.98
N
O
50000
40000
30000
20000
10000
0
10.0
5.0
0.0
ppm (t1)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135.572
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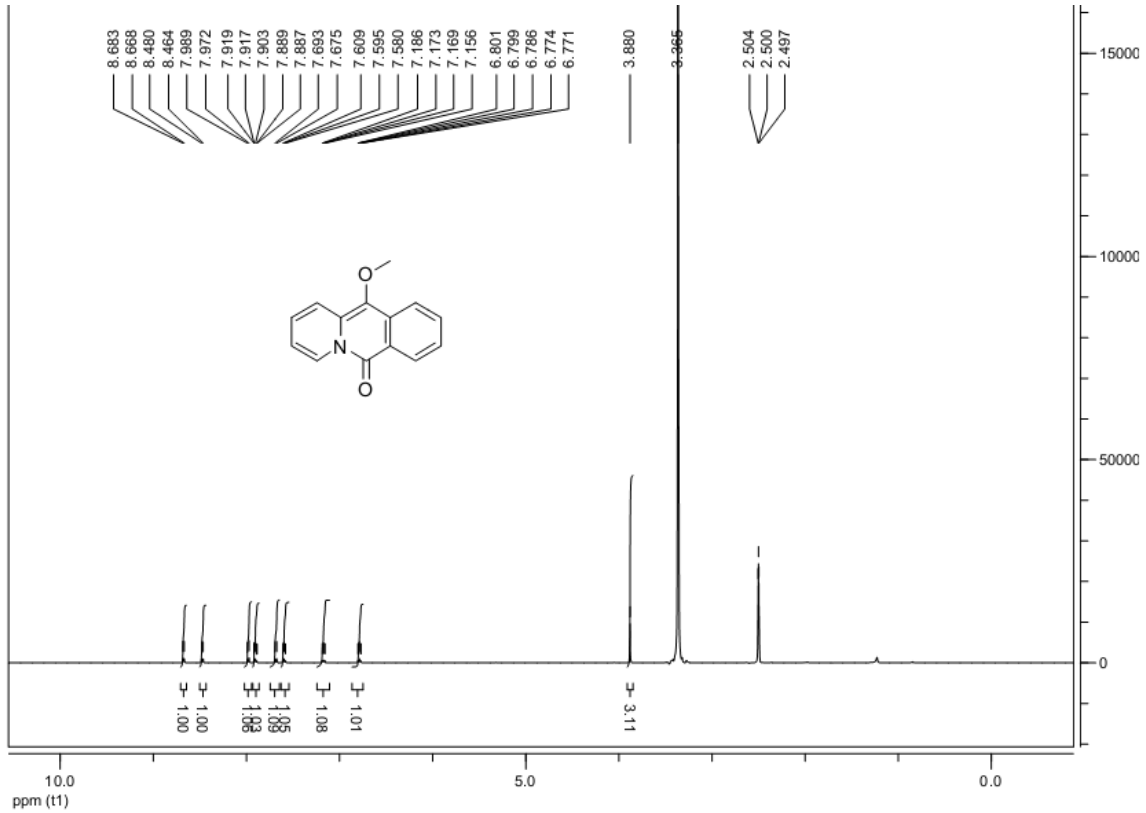
129.492
129.139
129.066
128.439
128.331
127.435
126.661
126.183
125.738
124.959
124.242
115.981
114.485
97.425
40.371
40.204
40.037
39.870
39.704
N
O
30000
25000
20000
15000
10000
50000
0
200
150
100
50
0
ppm (t1)

2p


8.683
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7.972
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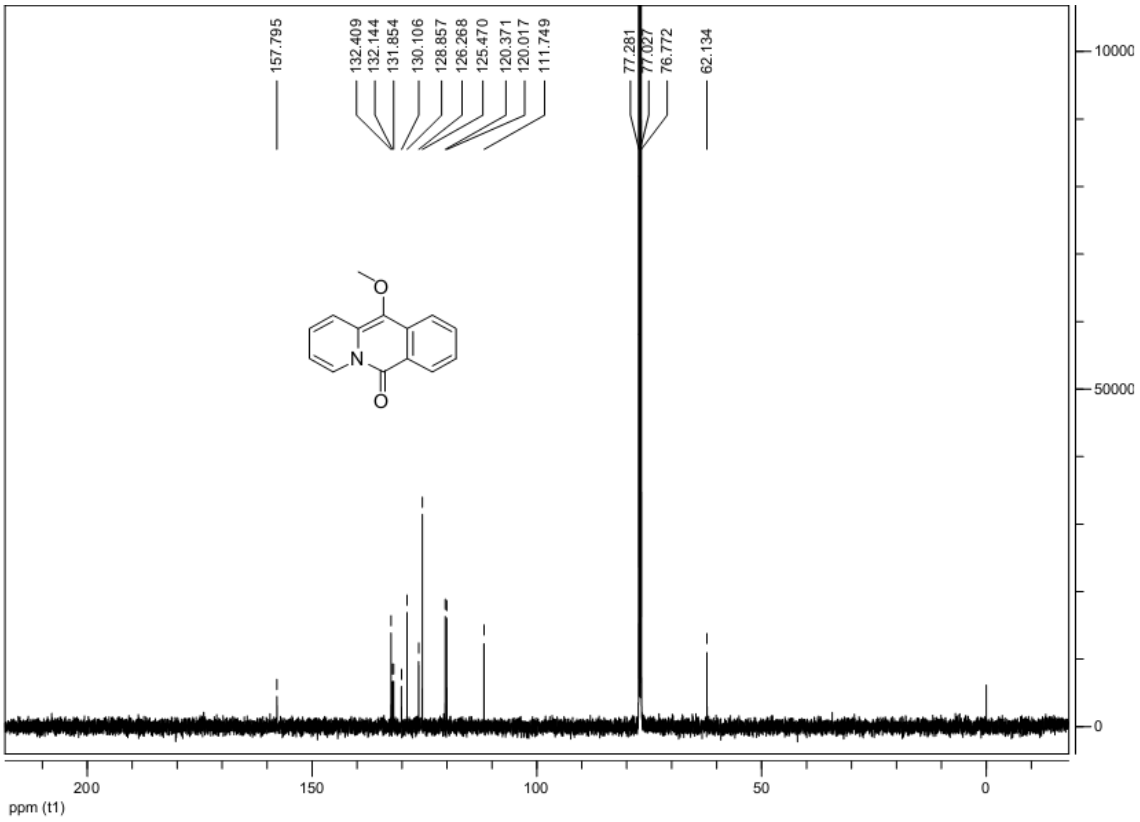
7.917
7.903
7.889
7.887
7.693
7.675
7.609
7.595
7.580
7.186
7.173
7.169
7.156
6.801
6.799
6.786
6.774
6.771
3.880
3.365
2.504
2.500
2.497
1.00
1.00
1.08
1.01
3.11
15000
10000
50000
0
10.0
5.0
0.0
ppm (t1)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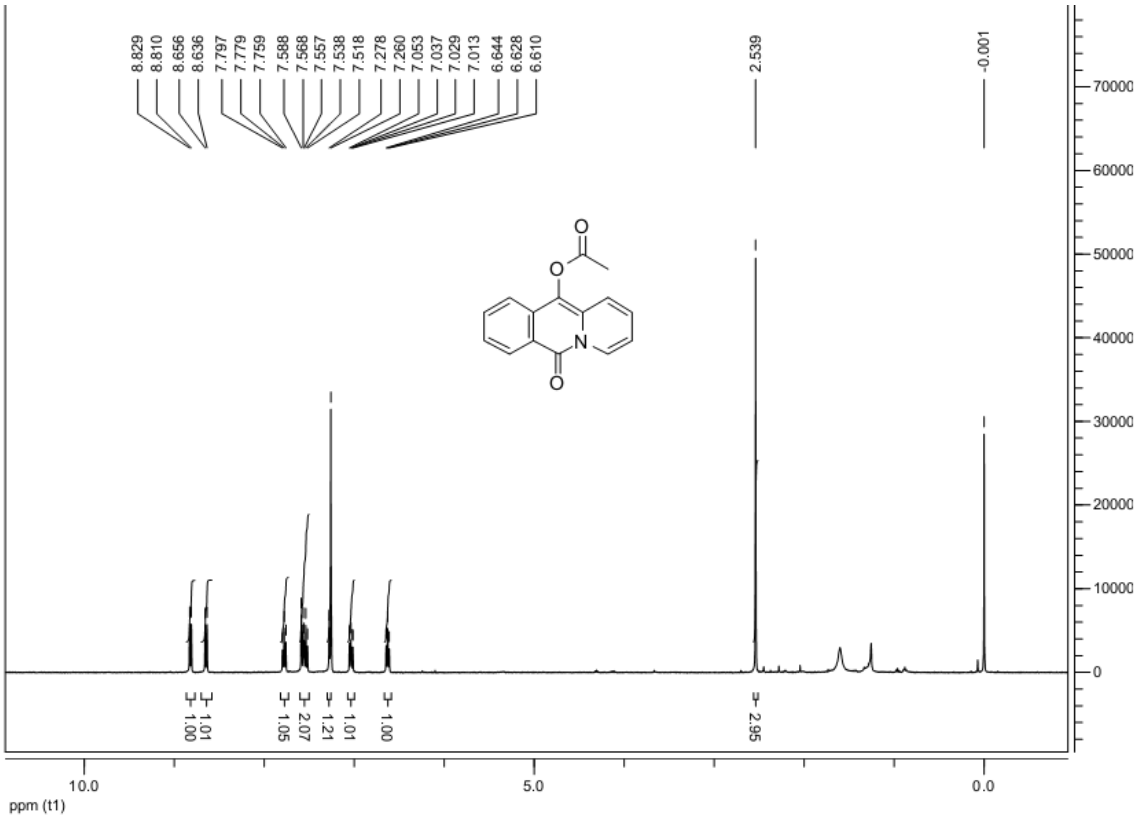
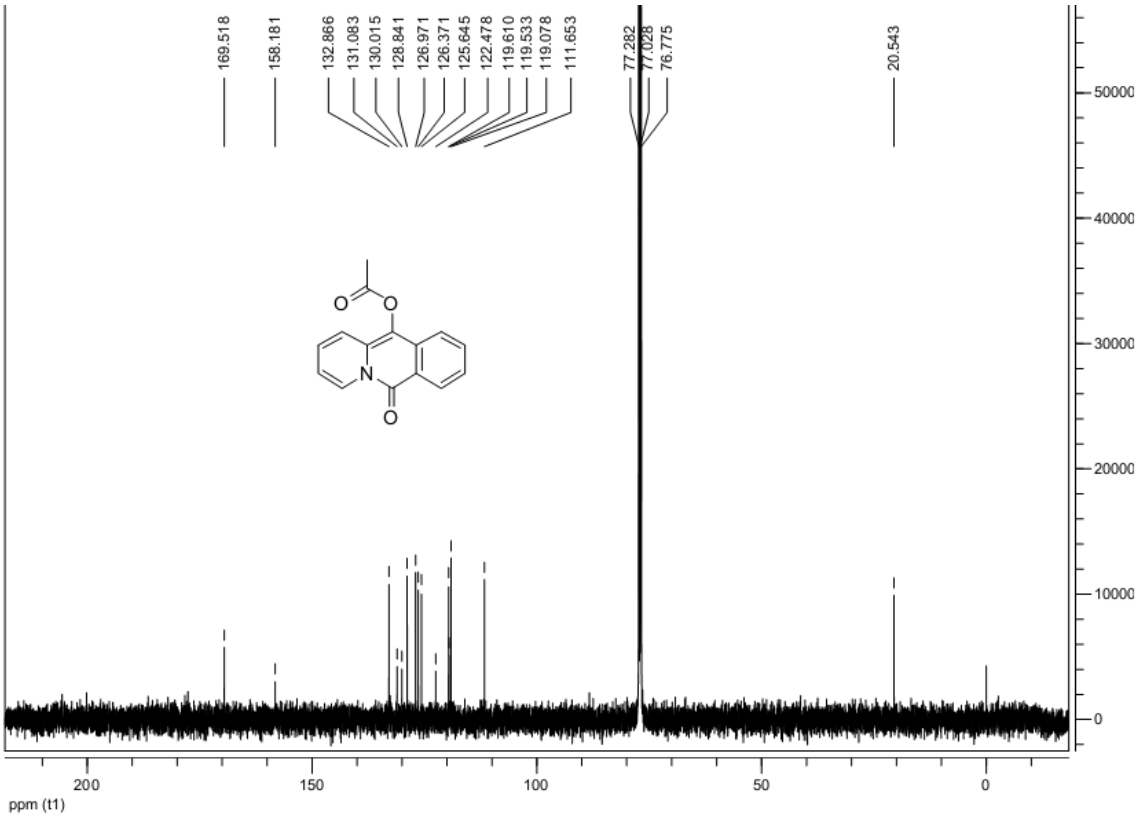
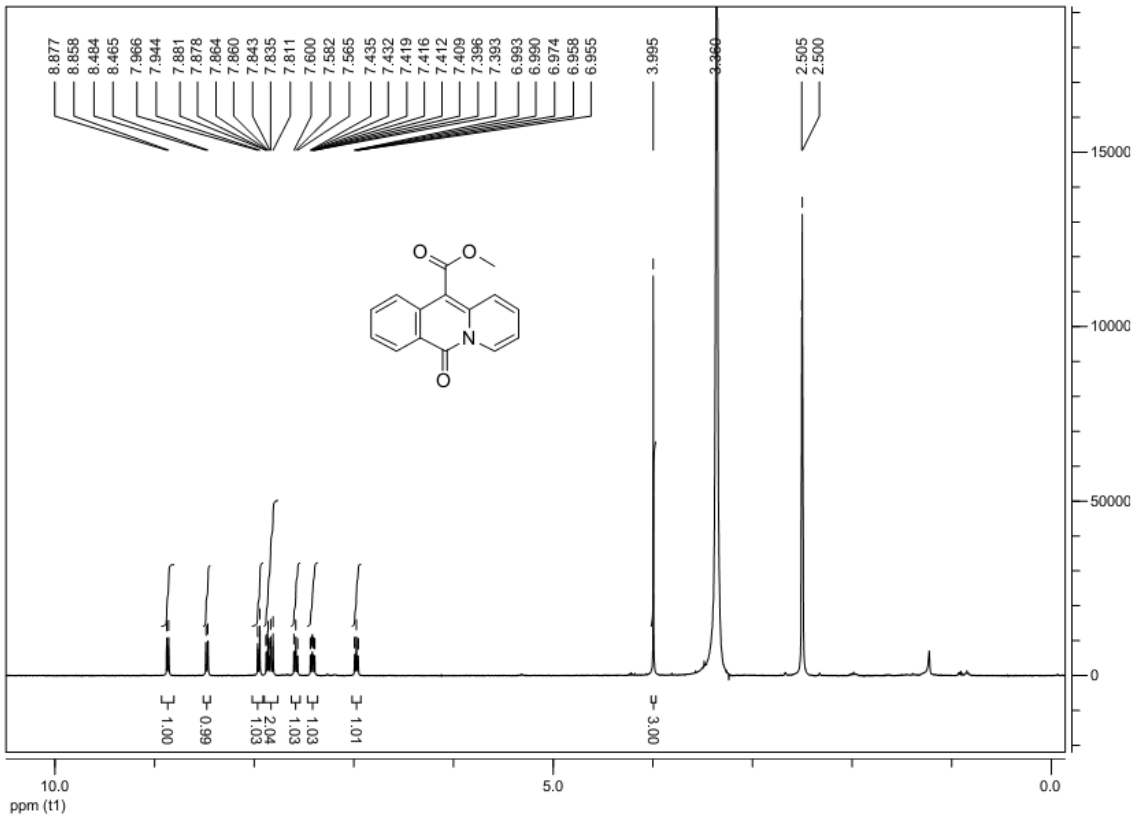
132.144
131.854
130.106
128.857
126.268
125.470
120.371
120.017
111.749
77.281
77.027
76.772
62.134
10000
50000
0
200
150
100
50
0
ppm (t1)

2q


8.829
8.810
8.656
8.636
7.797
7.779
7.759
7.588
7.568
7.557
7.538
7.518
7.278
7.260
7.053
7.037
7.029
7.013
6.644
6.628
6.610
2.539
-0.001
1.00
1.01
1.05
2.07
3.21
1.01
1.00
2.96
ppm (t1)


169.518
158.181
132.866
131.083
130.015
128.841
126.971
126.371
125.645
122.478
119.610
119.533
119.078
111.653
77.282
77.028
76.775
20.543
ppm (t1)

2r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8.465
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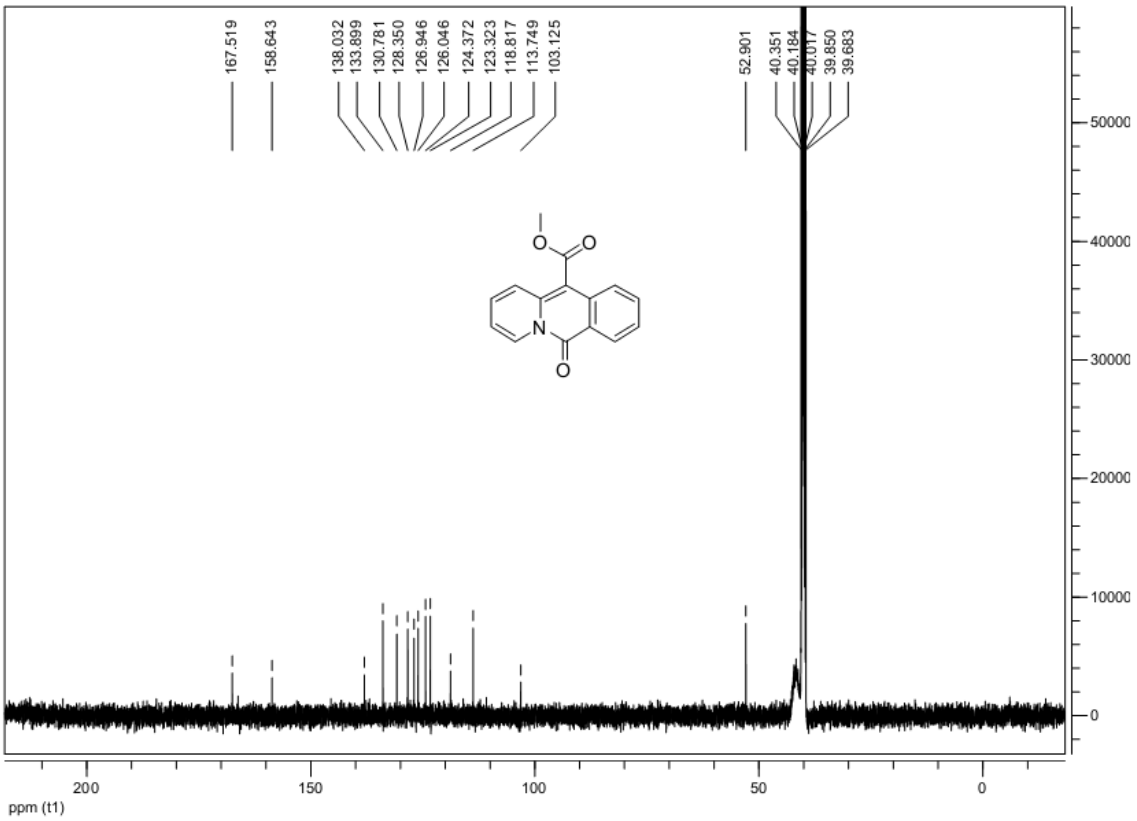
7.944
7.881
7.878
7.864
7.860
7.843
7.835
7.811
7.600
7.582
7.565
7.435
7.432
7.419
7.416
7.412
7.409
7.396
7.393
6.993
6.990
6.974
6.958
6.955
3.995
3.360
2.505
2.500
1.00
0.99
2.04
1.03
1.03
1.03
1.01
3.00
15000
10000
50000
0
10.0
5.0
0.0
ppm (t1)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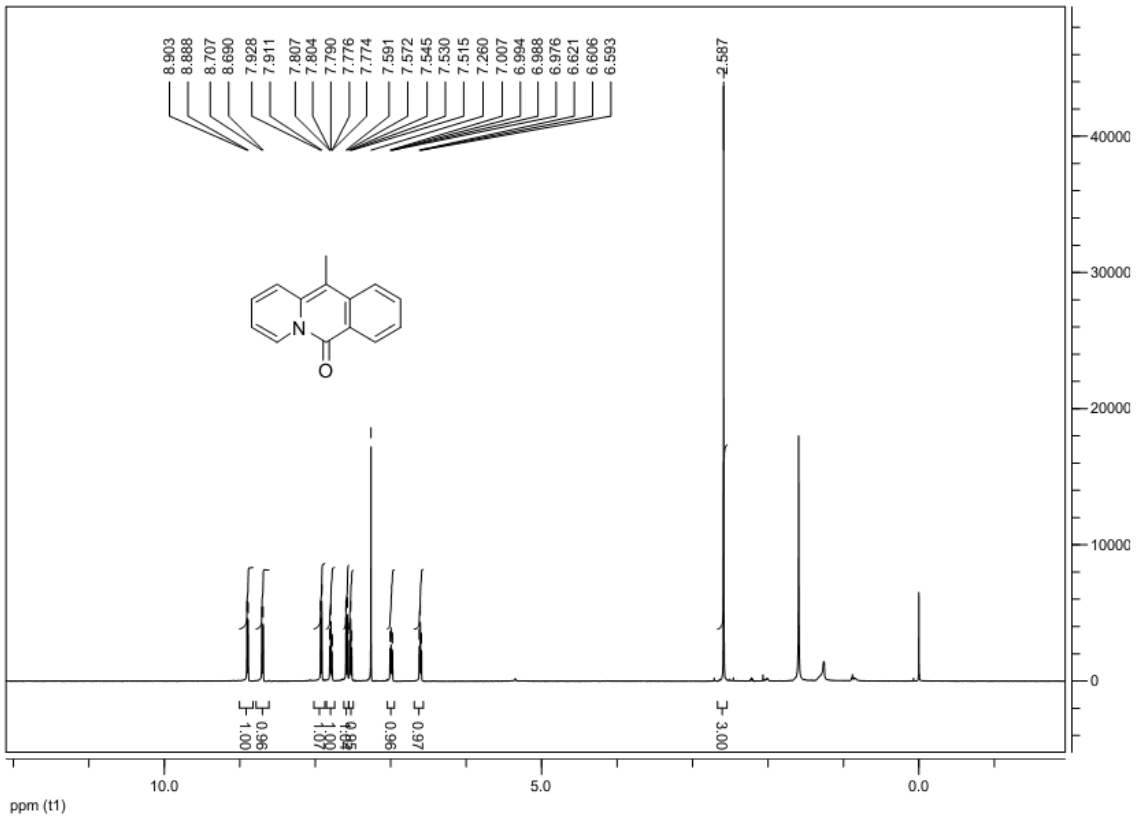
133.899
130.781
128.350
126.946
126.046
124.372
123.323
118.817
113.749
103.125
52.901
40.351
40.184
40.017
39.850
39.683
50000
40000
30000
20000
10000
0
200
150
100
50
0
ppm (t1)

2s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8.690
7.928
7.911
7.807
7.804
7.790
7.776
7.774
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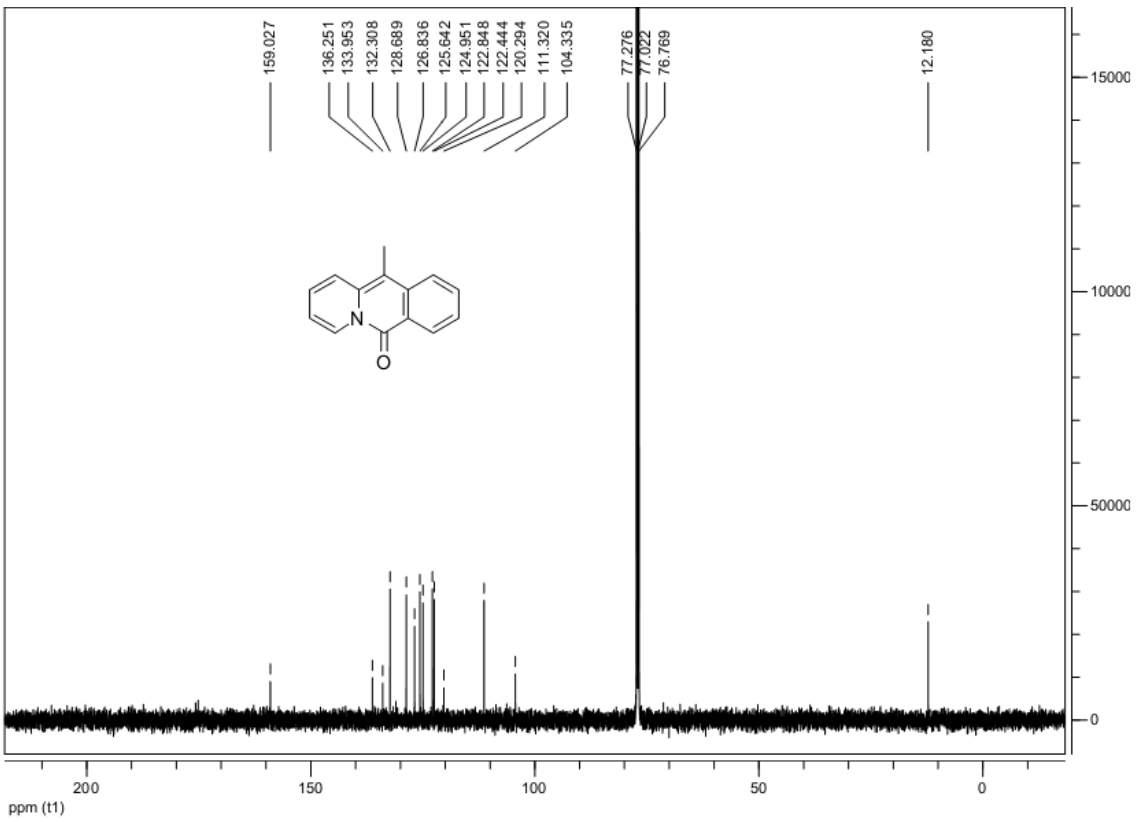
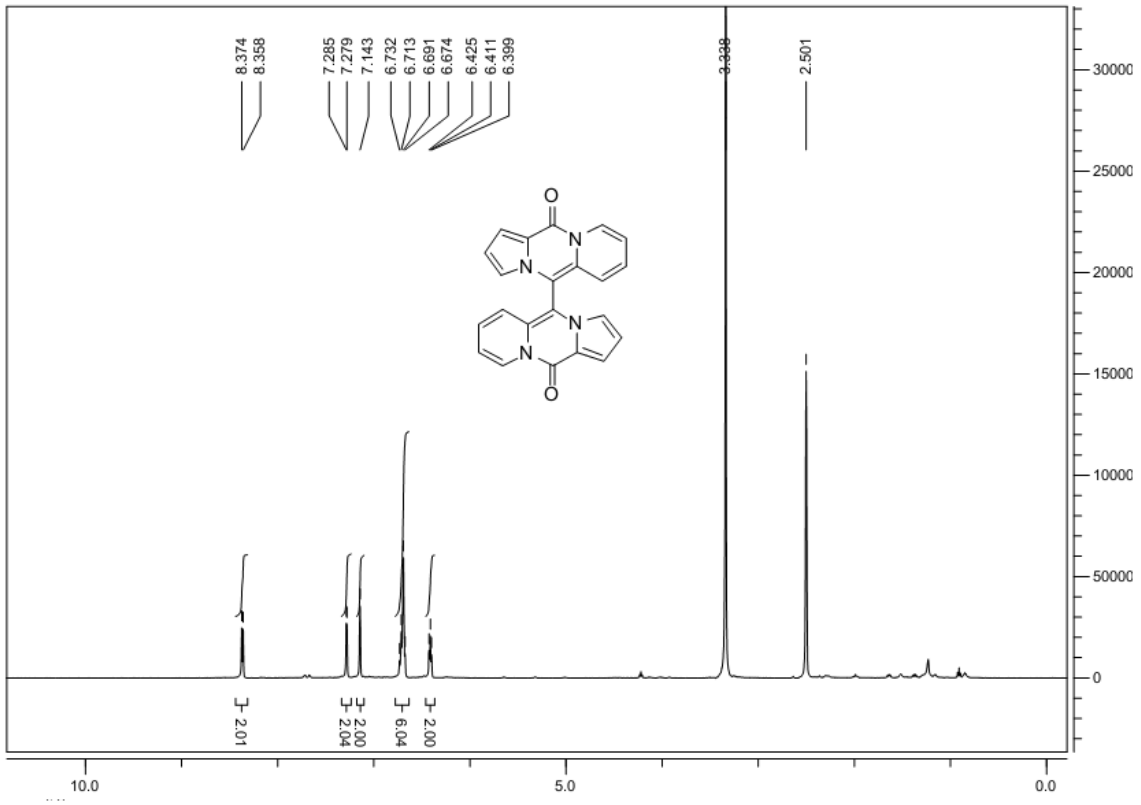
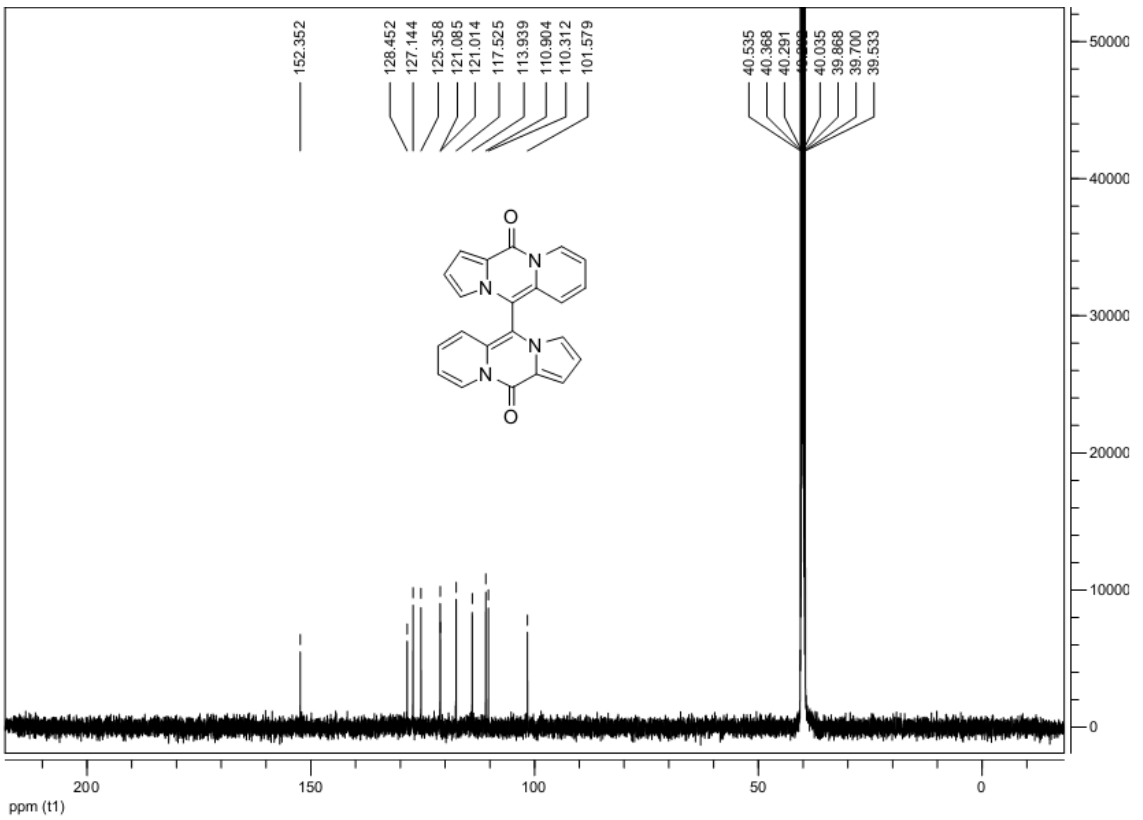
7.572
7.545
7.530
7.515
7.260
7.007
6.994
6.988
6.976
6.621
6.606
6.593
2.587
0.96
1.00
0.96
0.97
3.00
40000
30000
20000
10000
0
10.0
5.0
0.0
ppm (t1)

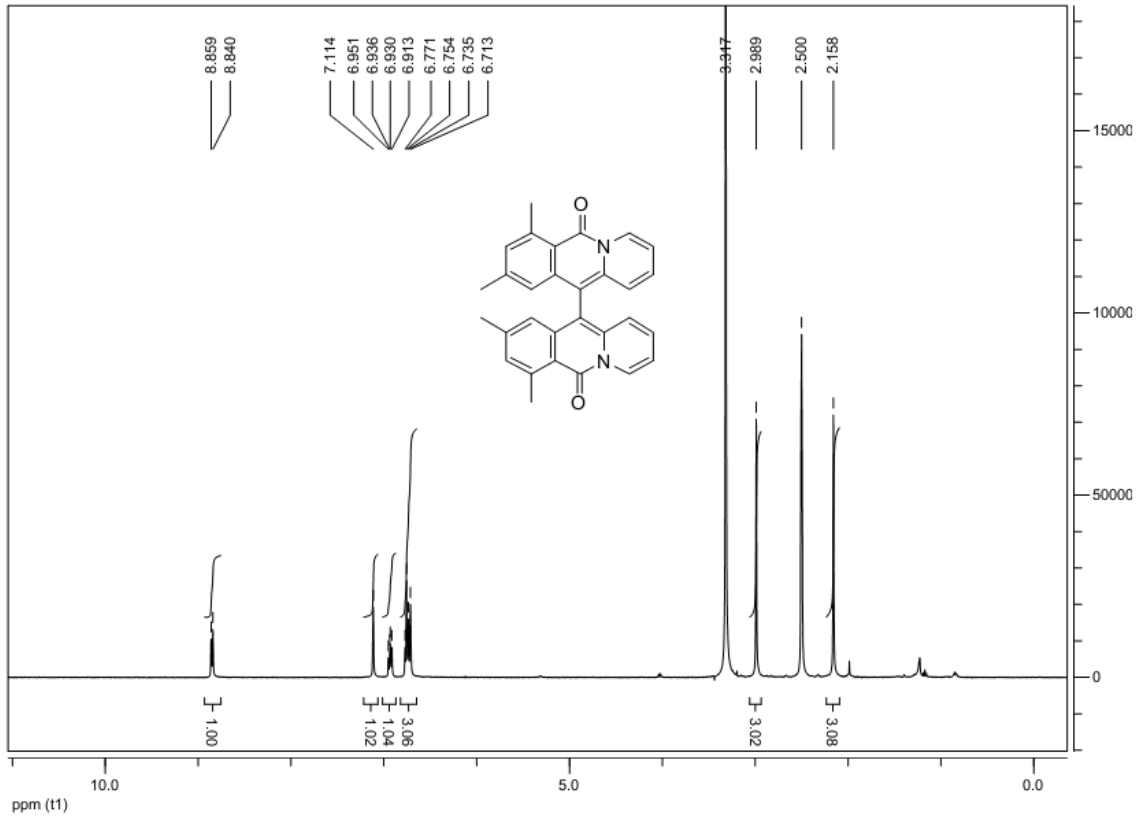

159.027
136.251
133.953
132.308
128.689
126.836
125.642
124.951
122.848
122.444
120.294
111.320
104.335
77.276
77.022
76.769
12.180
15000
10000
50000
0
200
150
100
50
0
ppm (t1)

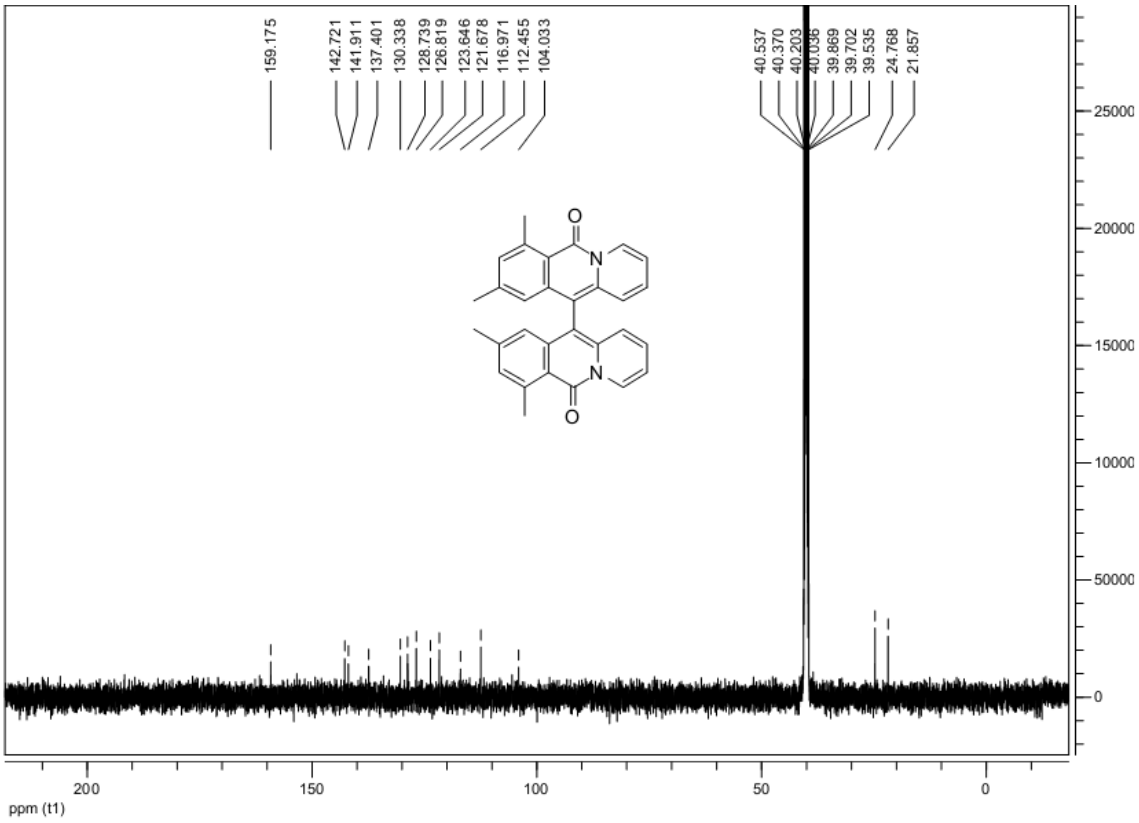
2t


8.374
8.358
7.285
7.279
7.143
6.732
6.713
6.691
6.674
6.425
6.411
6.399
3.338
2.501
2.01
2.00
2.04
6.04
2.00

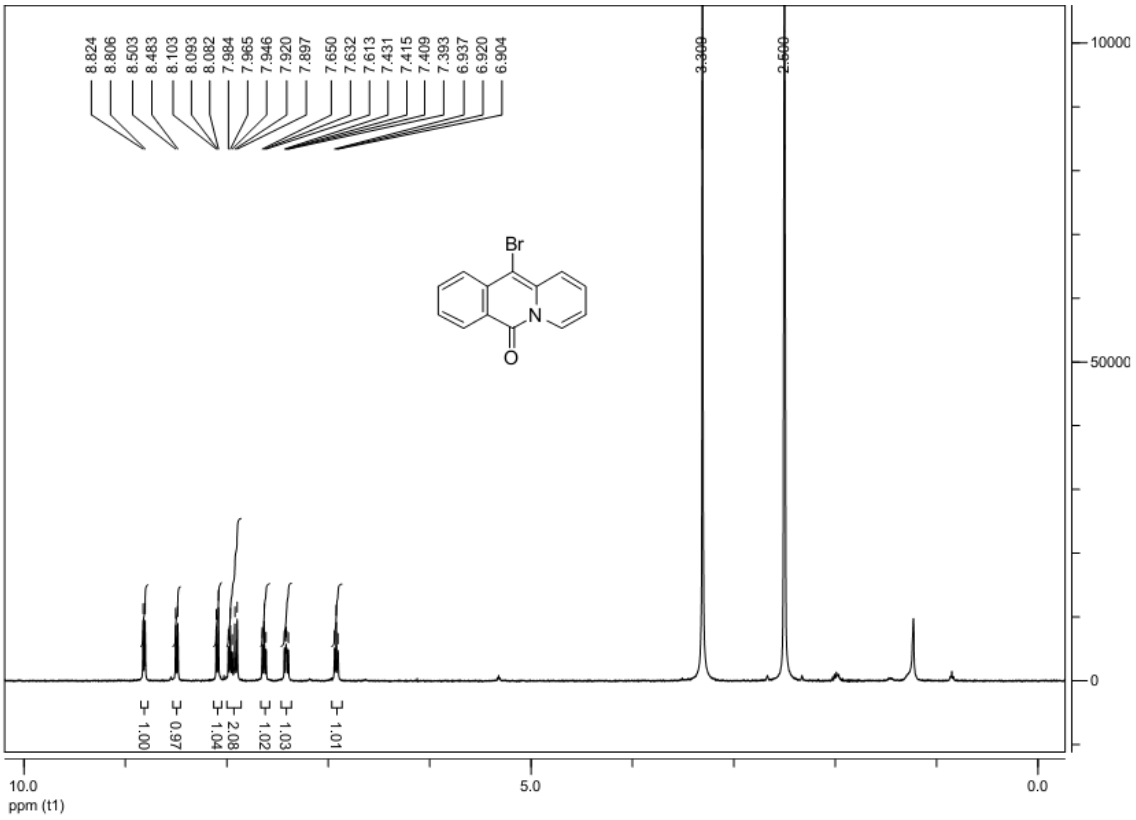

152.352
128.452
127.144
125.358
121.085
121.014
117.525
113.939
110.904
110.312
101.579
40.535
40.368
40.291
40.035
39.868
39.700
39.533
ppm (t1)

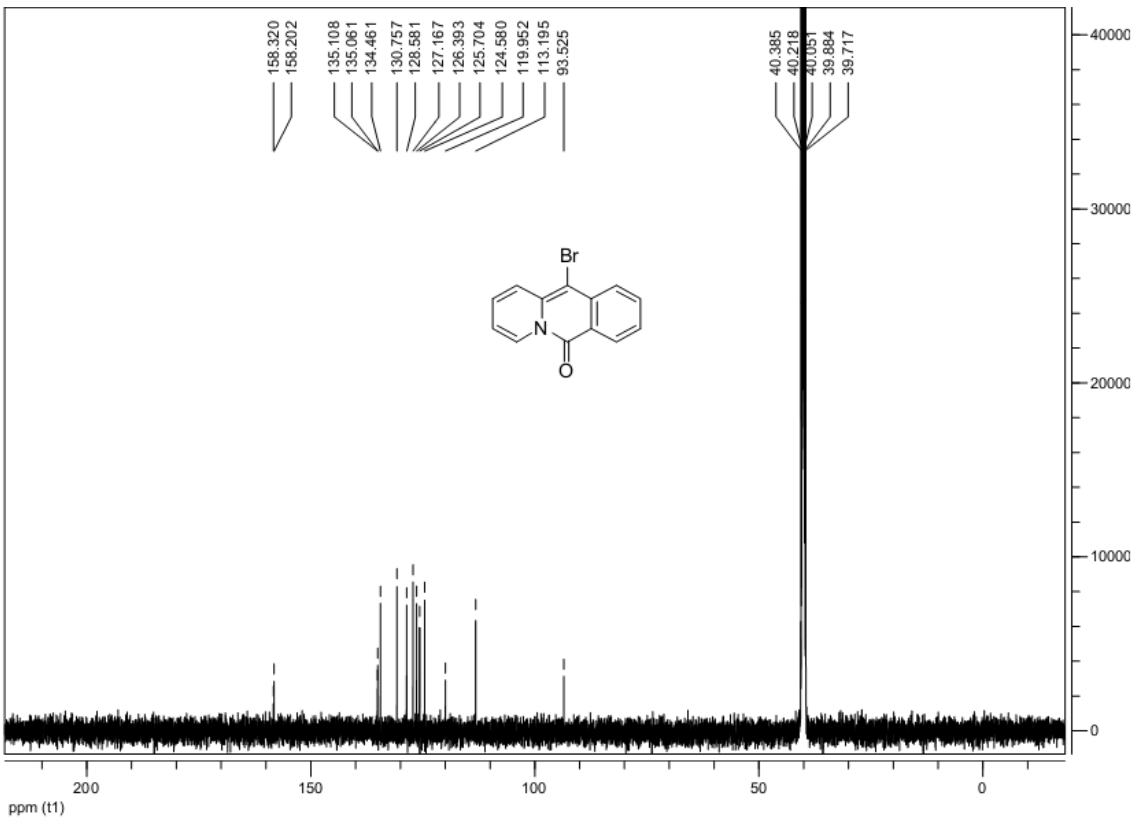
2u


8.859
8.840
7.114
6.951
6.936
6.930
6.913
6.771
6.754
6.735
6.713
3.347
2.989
2.500
2.158
1.00
1.02
1.04
3.06
3.02
3.08
10.0
5.0
0.0
ppm (t1)

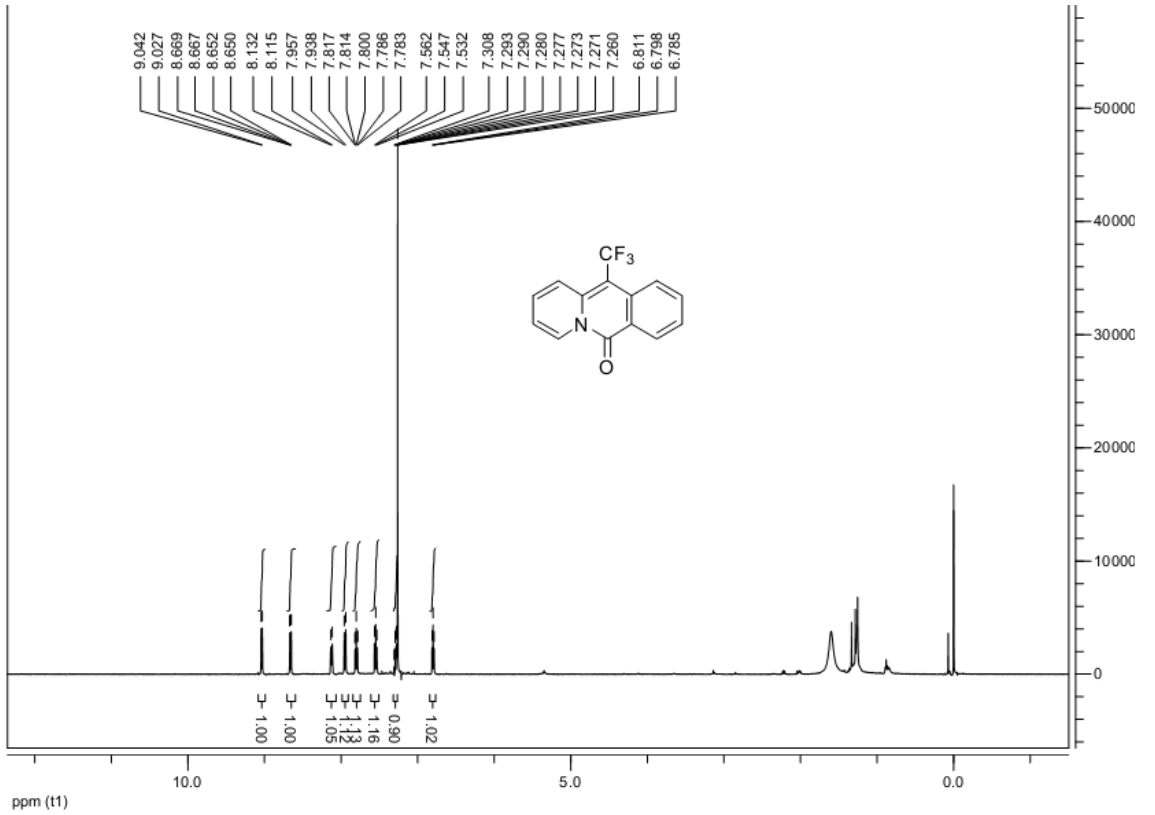

159.175
142.721
141.911
137.401
130.338
128.739
126.819
123.646
121.678
116.971
112.455
104.033
40.537
40.370
40.203
40.036
39.869
39.702
39.535
24.768
21.857
200
150
100
50
0
ppm (t1)

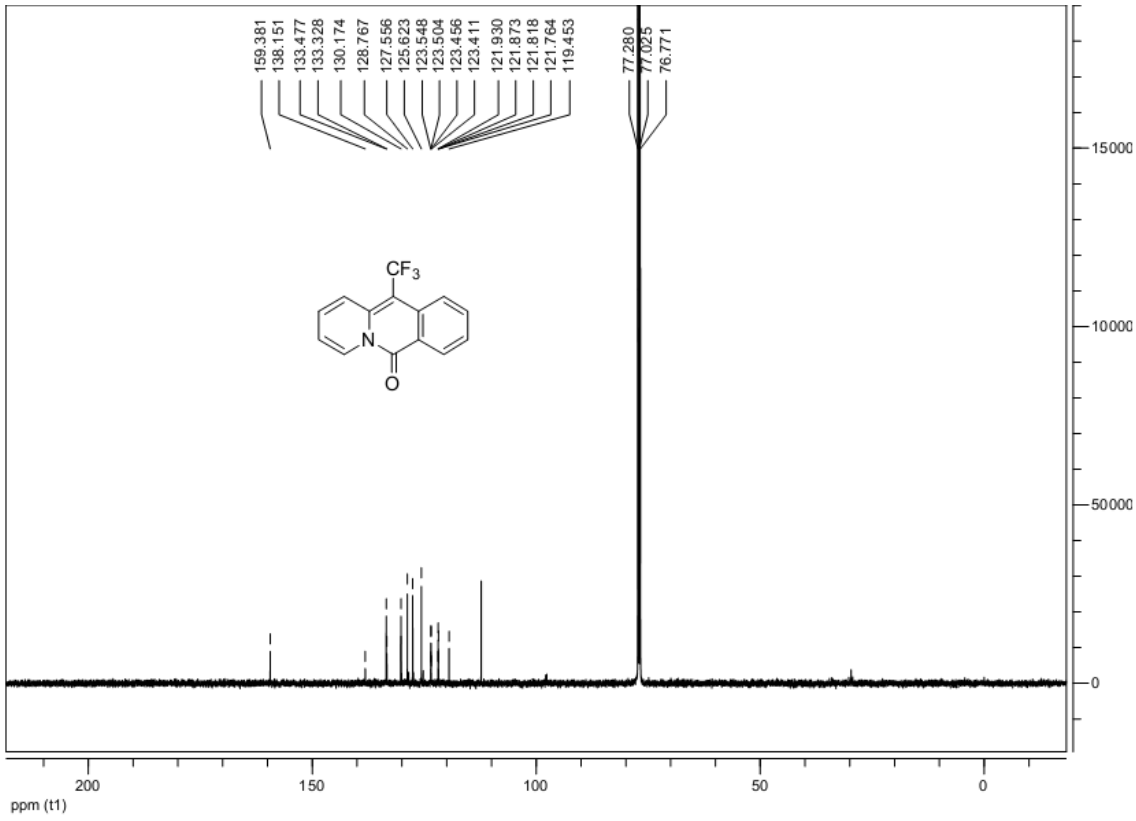
3a

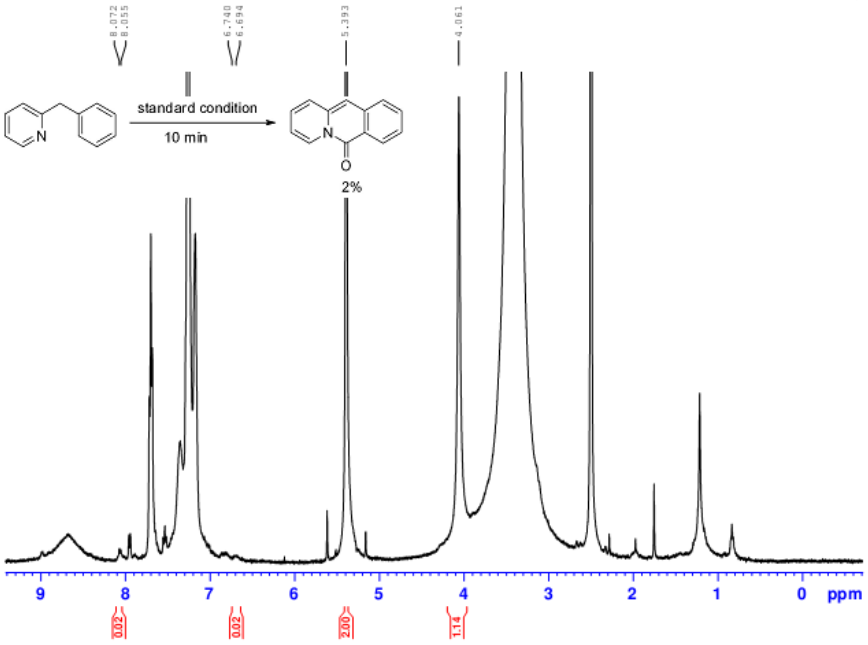

8.824
8.806
8.503
8.483
8.103
8.093
8.082
7.984
7.965
7.946
7.920
7.897
7.650
7.632
7.613
7.431
7.415
7.409
7.393
6.937
6.920
6.904
3.300
2.560
Br
N
O
1.00
0.97
1.04
2.08
1.02
1.03
1.01
10.0
5.0
0.0
ppm (t1)

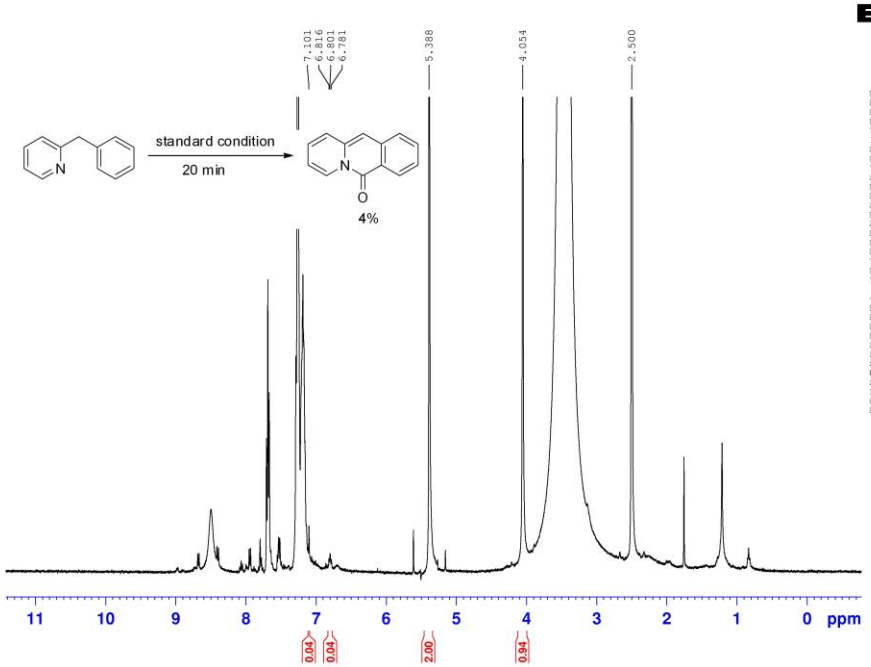

158.320
158.202
135.108
135.061
134.461
130.757
128.581
127.167
126.393
125.704
124.580
119.952
113.195
93.525
40.385
40.218
40.051
39.884
39.717
Br
N
O
200
150
100
50
0
ppm (t1)

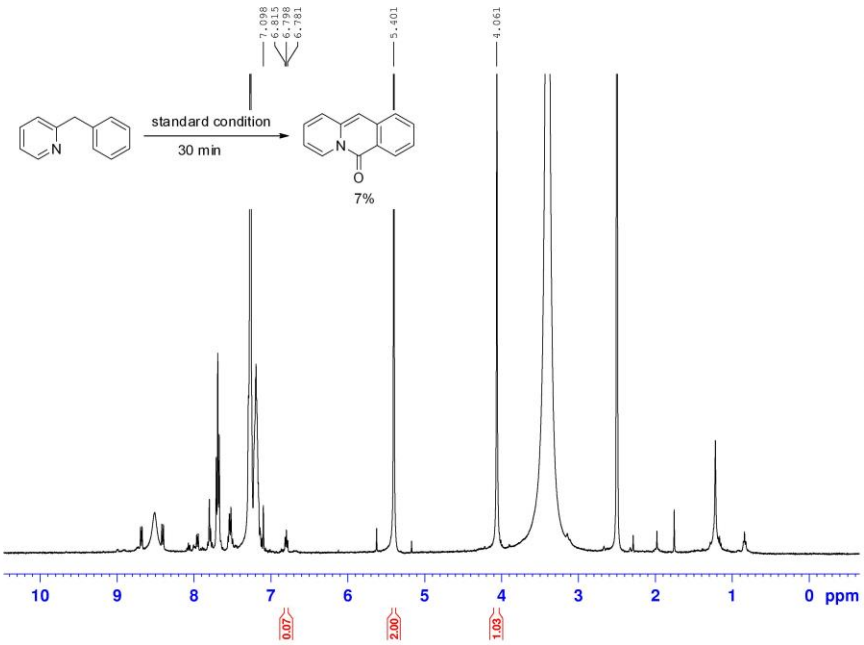
3b


9.042
9.027
8.669
8.667
8.652
8.650
8.132
8.115
7.957
7.938
7.817
7.814
7.800
7.786
7.783
7.562
7.547
7.532
7.308
7.293
7.290
7.280
7.277
7.273
7.271
7.260
6.811
6.798
6.785
CF3
N
O
1.00
1.00
1.05
1.12
1.13
1.16
0.90
1.02
10.0
5.0
0.0
ppm (t1)

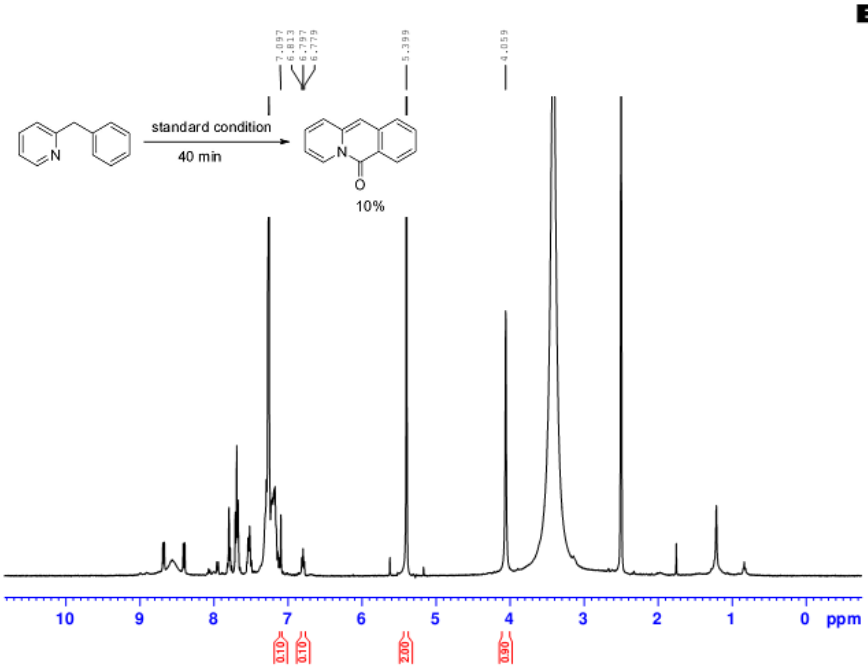

159.381
138.151
133.477
133.328
130.174
128.767
127.556
125.623
123.548
123.504
123.456
123.411
121.930
121.873
121.818
121.764
119.453
77.280
77.025
76.771
CF3
N
O
200
150
100
50
0
ppm (t1)

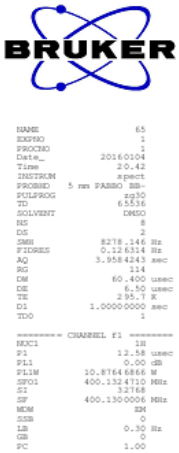

8.072
8.055
6.740
6.694
5.393
4.061
standard condition
10 min
2%
9
8
7
6
5
4
3
2
1
0
ppm
0.02
0.02
2.00
1.14

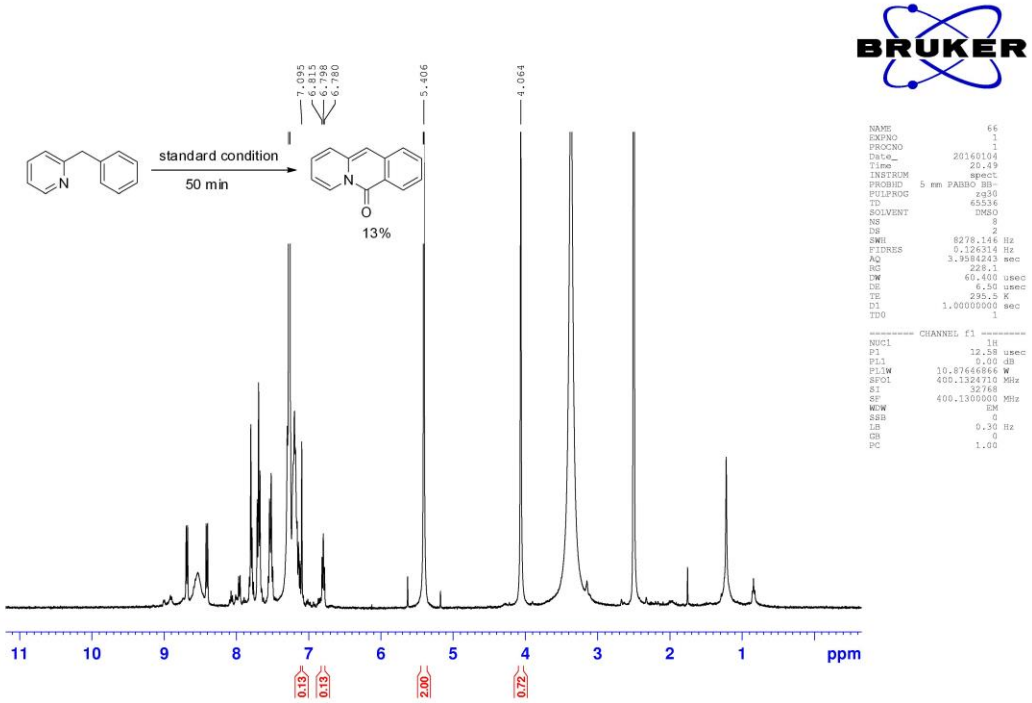

7.101
6.816
6.801
6.781
5.388
4.054
2.500
standard condition
20 min
4%
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.04
0.04
2.00
0.94

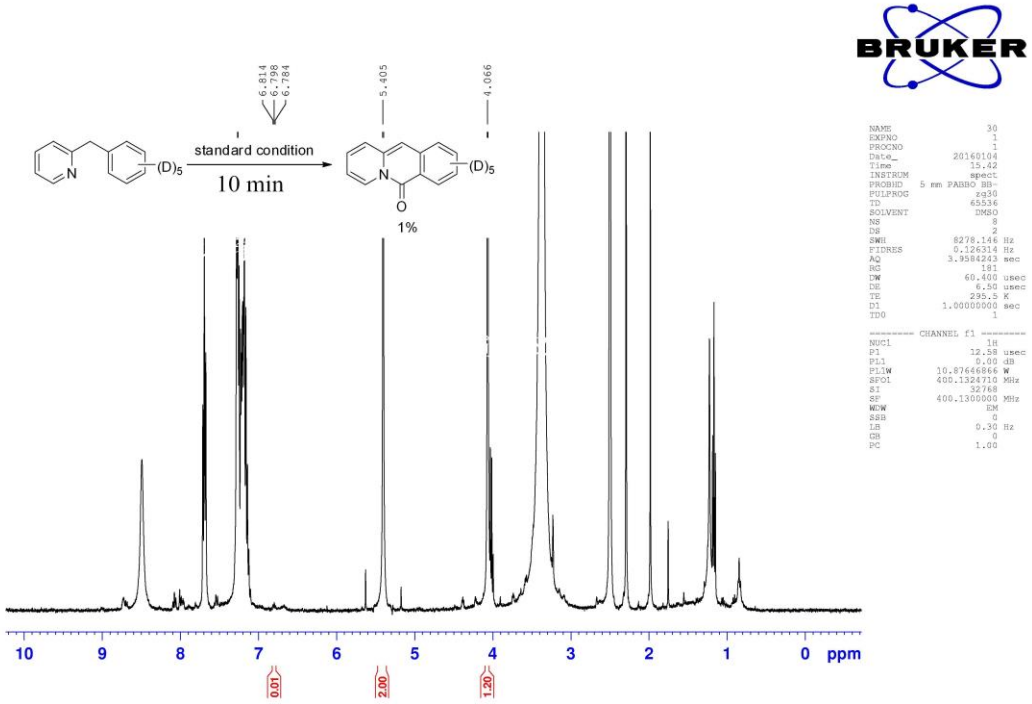

7.098
6.815
6.798
6.781
5.401
4.061
standard condition
30 min
7%
10
9
8
7
6
5
4
3
2
1
0 ppm
0.07
2.00
1.03

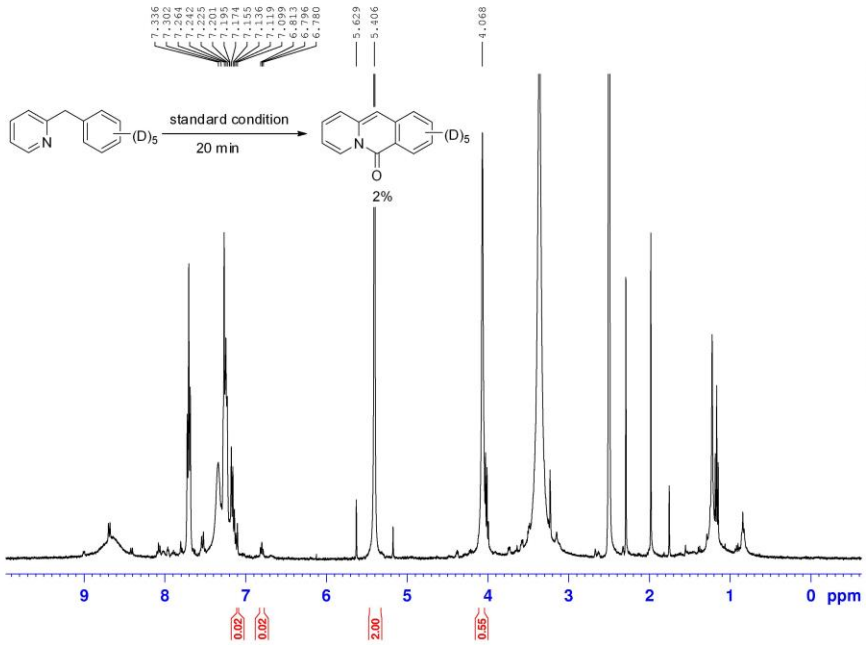

NAME 64
EXPNO 1
PROCNO 1
Date_ 20160104
Time 20.36
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 161.2
DW 60.400 usec
DE 6.50 usec
TE 295.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300007 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


7.097
6.813
6.797
6.779
5.399
4.059
standard condition
40 min
10%
10
9
8
7
6
5
4
3
2
1
0 ppm
0.10
0.10
2.00
0.90

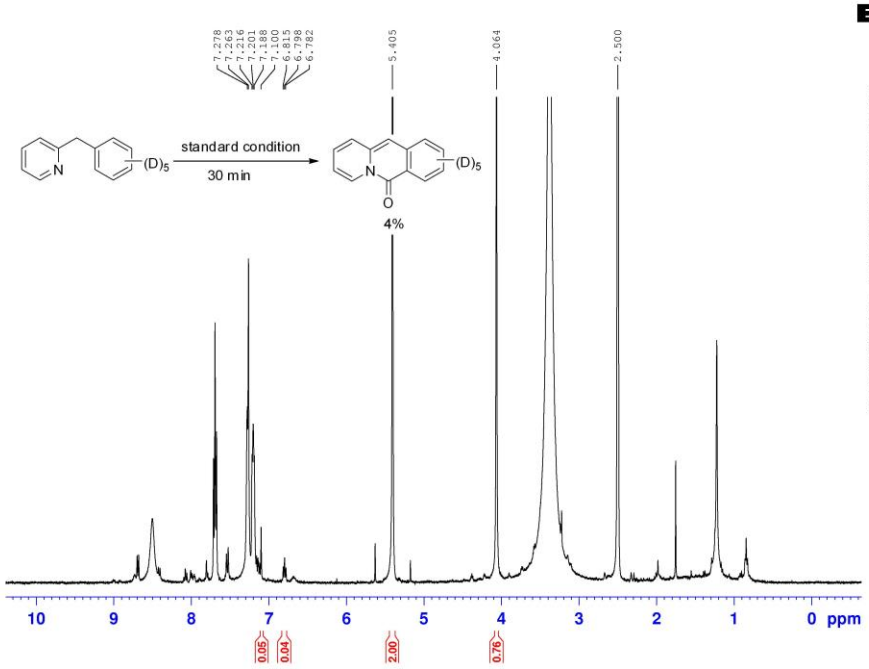

NAME 65
EXPNO 1
PROCNO 1
Date_ 20160104
Time 20.42
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 114
DW 60.400 usec
DE 6.50 usec
TE 295.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300006 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


7.095
6.815
6.798
6.780
5.406
4.064
standard condition
50 min
13%
11 10 9 8 7 6 5 4 3 2 1 ppm
0.13
0.13
2.00
0.72

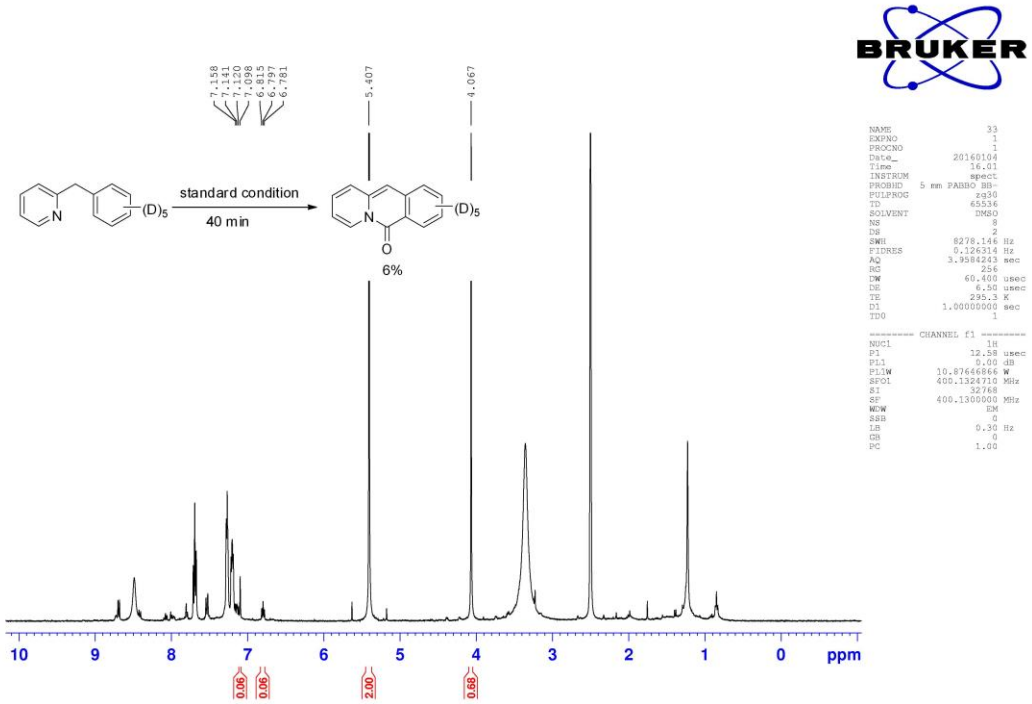

6.814
6.798
6.784
5.405
4.066
standard condition
10 min
(D)5
(D)5
1%
10 9 8 7 6 5 4 3 2 1 0 ppm
0.01
2.00
1.20


7.336
7.302
7.264
7.242
7.225
7.201
7.195
7.174
7.155
7.136
7.119
7.099
6.813
6.796
6.780
5.629
5.406
4.068
standard condition
20 min
(D)5
(D)5
2%
9
8
7
6
5
4
3
2
1
0 ppm
0.02
0.02
2.00
0.55


NAME 31
EXPNO 1
PROCNO 1
Date_ 20160104
Time 15.49
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPNOG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DW 60.400 usec
DE 6.50 usec
TE 295.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

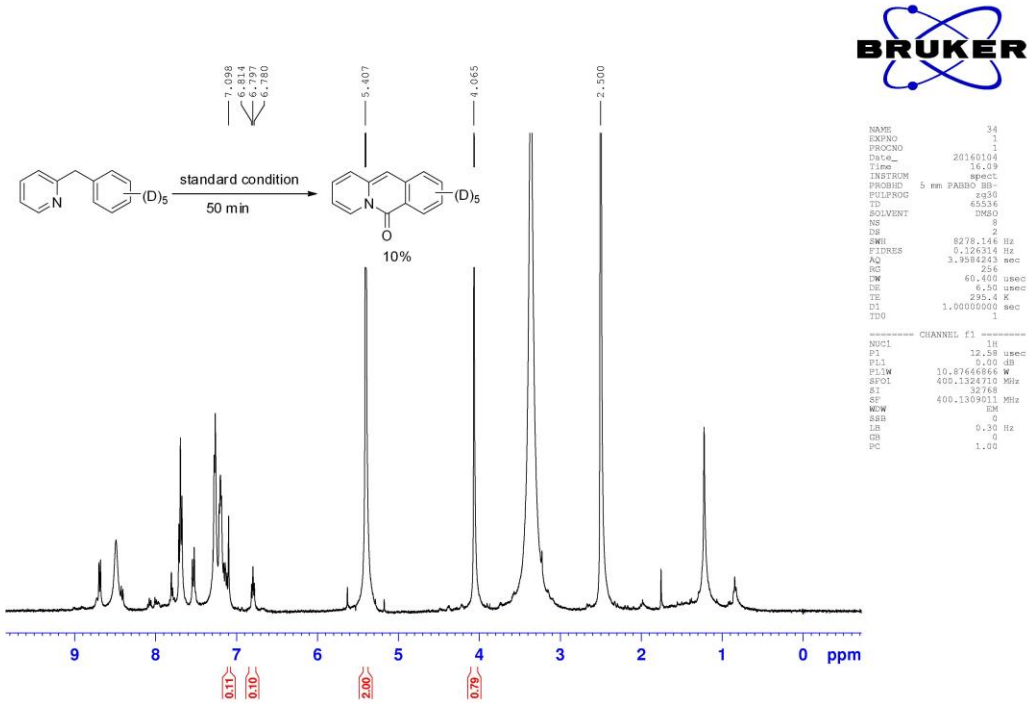

7.278
7.263
7.216
7.201
7.188
7.100
6.815
6.798
6.782
5.405
4.064
2.500
standard condition
30 min
(D)5
(D)5
4%
10
9
8
7
6
5
4
3
2
1
0 ppm
0.05
0.04
2.00
0.76


NAME 32
EXPNO 1
PROCNO 1
Date_ 20160104
Time 15.55
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPNOG zg30
TD 65536
SOLVENT DMSO
NS 8
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DW 60.400 usec
DE 6.50 usec
TE 295.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.58 usec
PL1 0.00 dB
PL1W 10.87646866 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1299999 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00




```
NAME                33
EXPNO                1
PROCNO               1
Date_         20160104
Time             16.01
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG          zg30
TD              65536
SOLVENT          DMSO
NS                   8
DS                   2
SWH          8278.146 Hz
FIDRES       0.126314 Hz
AQ           3.9584243 sec
RG                 256
DW              60.400 usec
DE               6.50 usec
TE              295.3 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1               12.58 usec
PL1               0.00 dB
PL1W        10.87646966 W
SFO1       400.1324710 MHz
SI                32768
SF         400.1300000 MHz
WDW                  EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```




```
NAME                34
EXPNO                1
PROCNO               1
Date_         20160104
Time             16.09
INSTRUM          spect
PROBHD   5 mm PABBO BB-
PULPROG          zg30
TD              65536
SOLVENT          DMSO
NS                   8
DS                   2
SWH          8278.146 Hz
FIDRES       0.126314 Hz
AQ           3.9584243 sec
RG                 256
DW              60.400 usec
DE               6.50 usec
TE              295.4 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1               12.58 usec
PL1               0.00 dB
PL1W        10.87646866 W
SFO1       400.1324710 MHz
SI                32768
SF         400.1309011 MHz
WDW                  EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```